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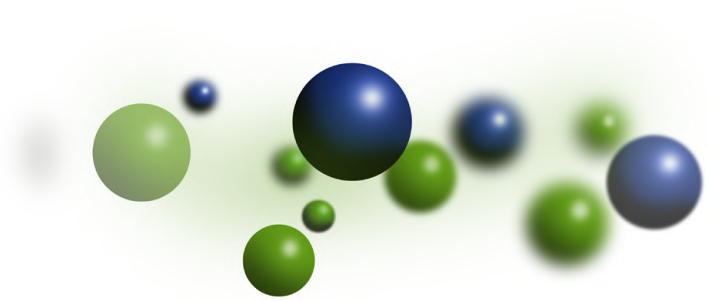
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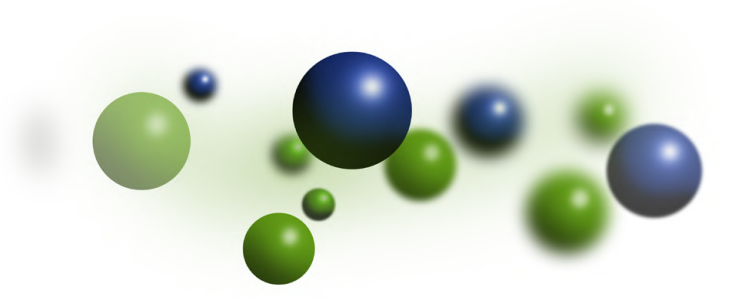
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PLENARY SPEAKERS



Challenges and opportunities in particle processing: an industrial perspective

Hendrickson, Willie

As attendees to this conference, we are all aware that solids handling and processing can be challenging. Consistent outcomes can appear to be transient from day to day or from one piece of equipment to another. Great strides in understanding the fundamentals of particle processing have been made over the past 50 years, but all if us, academic and industrial practitioners, will have moments when we struggle. While getting consistent, publishable results in the lab can be a challenge, the problems of consistency, scale-up, and reproducibility in large scale processing with variations in ambient conditions (e.g. humidity), raw materials, and seemingly small process changes are vexing and require constant control and analysis.

This talk will present how industry approaches solids handling, uses engineering solutions to help with consistency, and what are some of the gaps in our understanding of the fundamentals of particle processing that would be helpful in full scale industrial processing. Examples and opportunities in blending, size separation, drying, and particle formation using polymer, food, and ceramic materials will be provided to illustrate these challenges. A discussion of Technology Readiness Levels (TRL) as it relates to the difficulty of transitioning lab discoveries into products will be made along with the criticality of knowing how to overcome the barriers to full-scale, consistent production. Finally, time will be spent during this presentation on the needs of industry in education and research and how academia and industry can best interact to both parties' advantage.

Nanoparticles as therapeutic agents. Expectations, reality and perspectives

Santamaría, Jesús

Nanotechnology has opened up new perspectives in all aspects of life. In health, a new discipline, Nanomedicine, was created to exploit the exciting new properties of nanomaterials: they were able to cross biological membranes, target specific cells, transport drugs, or even do drug-free therapy by being able to heat up or to generate reactive oxygen species when activated by the suitable electromagnetic radiation. A myriad of nanostructured systems was designed and built in the laboratory to channel these functionalities, and different families of nanoparticles emerged with previously unseen capabilities. The obvious question is: Why of all of these promising nanoparticle systems only a handful have reached clinical trials and even fewer have succeeded?

In my view, the success of Nanomedicine has been restricted due to three main challenges: First, nanomaterials must be able to perform effectively under the conditions prevailing in the target environment, which may be very different from those used in the laboratory as a proof of concept. Second, the nanoparticle must be delivered selectively (or at least preferentially) to the target. This is also a formidable challenge. For instance, in oncology, current methods that rely on the EPR effect or even targeted delivery, present very low delivery efficiency, and this has given rise to a variety of alternative delivery methods. Third, ideally the nanoparticle should be activated only at the target site, to avoid side effects in healthy cells. For this, a variety of possibilities exist such as electromagnetic field-based activation, environment-related activation, and so on.

The talk will present an overview of the area, identifying roadblocks and some possible solutions. Recent advances regarding each of the above areas will be critically discussed.

Suds, soup and shampoo - unilever: my world of powders

Bonsall, Judith

The talk will cover some of the aspects of dealing with particles in a FMCG company such as the pitfalls of only considering mean particle size and neglecting to factor in the spread of the distribution or talking about fine particles without establishing the actual size meant by fine!

I will also give some examples of how being a member of IFPRI has given valuable insights when dealing with the everyday problems that occur in powders handling and manufacture.

Advanced functional particles: scalable formation, property classification and multidimensional characterization

Peukert, Wolfgang (1)
(1) University Erlangen-Nuremberg , Institue of Particle Technology

A long-term vision in particle science and technology is the targeted design of particulate products by rigorous optimization based on predictive structure-property and process-structure functions. Particulate products consist in the simplest case of dispersed single particles and in more complex cases of hierarchically organized assemblies of particles, e.g. in the form of functional thin films or other formulated products. We address progress in the scale-up of continuous formation processes, in property classification of nanoparticles with respect to size, shape or composition and in their multi-dimensional characterization. Recent breakthroughs and current needs will be discussed for different material systems and applications.

Nanostructured materials to design innovative nanomedicines

Alonso, María José

The world of particles we emit: how well do we understand and control it?

Morawska, Lidia (1)
(1) Queensland University of Technology

Advances in solids processing research and modeling to adress industrial challenges

Dahlhaus, Jürgen

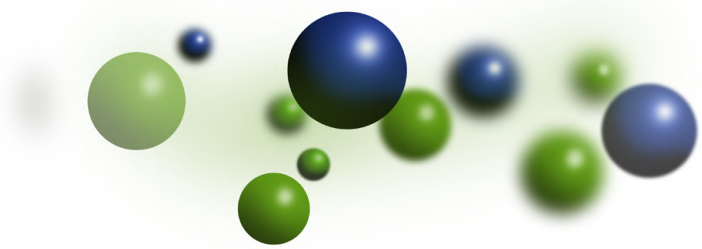
Simulation and modelling of particulate systems

Yu, Aibing (1)
(1) Monash University

Particle science and technology is a rapidly developing interdisciplinary research area with its core being the understanding of the relationships between micro- and macro-scopic properties of particulate/granular matter – a state of matter that is widely encountered but poorly understood. The macroscopic behaviour of particulate matter is controlled by the interactions between individual particles as well as interactions with surrounding gas or liquid and wall. Understanding the microscopic mechanisms in terms of these interaction forces is therefore key to leading to truly interdisciplinary research into particulate matter and producing results that can be generally used. This aim can be effectively achieved via particle scale research based on detailed microdynamic information such as the forces acting on and trajectories of individual particles in a considered system. In the past two decades or so, such research has been rapidly developed worldwide, mainly as a result of the rapid development of discrete particle simulation technique and computer technology.

This talk presents a brief overview of the theoretical developments in discrete element modelling. It covers three important aspects: models for the calculation of particle-particle and particle-fluid interaction forces under different conditions, coupling of discrete element method with computational fluid dynamics to describe particle-fluid flow, heat and mass transfer, and the theories for linking discrete to continuum modelling. Focus is however given to those developed in my laboratory “SIMPAS”. It is also demonstrated through examples that the study of small particles is well linked to many challenging problems in big science. The examples also demonstrate that particle scale approach has gradually emerged to be a powerful tool not only for fundamental research but also for engineering application. Needs for future development are also discussed.

ORAL PRESENTATIONS



1 PARTICULATE SOLIDS HANDLING

KEYNOTE LECTURES

Acoustic behavior of inner ear canaliths

Klinzing, George (1); Dumm, Christopher M. (1); Vipperman, Jeffrey S. (1); Balaban, Carey (1); Ozarkar, Shailesh (2)
(1) University of Pittsburgh, (2) Ansys Corporation

Motion of otoconia particles (predominantly calcium carbonate) in the inner ear is well-known clinically to be a potential source of vertigo and other symptoms indicating dysfunction of the vestibular system. Benign paroxysmal positional vertigo (BPPV) is associated with otoconia moving into the semicircular canals of the inner ear, causing improper sensation of angular acceleration. Procedures to alleviate BPPV symptoms involve maneuvering of the head to reorient the particles under gravitational influence. Another type of disorder, historically termed the "Tullio phenomenon," is characterized by the triggering of vertigo in response to loud noises, sound, or pressure changes, and has occasionally been linked to thinning of bone (dehiscence) adjacent to the superior semicircular canal. Prior to the advent of computed tomography imaging and the ability to nonsurgically assess structural defects, various historical attempts to treat vestibular dysfunction reported some improvement in symptoms after applying ultrasound stimuli to the inner ear labyrinth at empirically-selected levels. Recent work indicates that acoustic stimuli may be mechanically amplified in non-obvious ways by the structure of the human temporal bone and its inner ear labyrinth. Environmental acoustic stimuli might accordingly be associated with inducing motion of otoconia, enabling alleviation of symptoms of vestibular dysfunction. The present exploratory study computationally assesses how acoustic stimuli affect behavior of the otoconia particles naturally present in the inner ear.

Characteristic particle size is on the order of 20 microns, and the inner ear fluids are primarily water at an atmospheric working pressure. Various geometric approximations for the inner ear are considered. Various levels of acoustic stimuli and excitation schemes are considered.

Particle shape-induced axial segregation of binary mixtures of spheres and ellipsoids in a rotating drum

Zhou, Zongyan (1); He, Siyuan (2); Yu, Aibing (2); Pinson, David (3)
(1) Jiangxi Key Laboratory for Modelling and Simulation of Particulate Systems, Jiangxi University of Science and Technology, (2) Department of Chemical and Biological Engineering, Monash University, (3) Bluescope Steel Research

Rotating drums are widely used in industry for mixing, milling, coating and drying processes. Particle mixtures with different physical properties in the rotating drums could segregate in the both radial and axial directions from an initially well-mixed state. In this presentation, we report the segregation phenomenon induced by particle shape. Particle shape used in our work is spheroidal which can be described by the parameter of aspect ratio (AR) defined as the ratio of both principal diameters of spheroids. Aspect ratio varies within a large range representing from oblate (AR<1) and spherical (AR=1) and problate shapes. Binary mixtures of spheres and spheroids are used in the discrete element method (DEM) simulation, and the segregations in both radial and axial directions are investigated. The results show that in a short drum, particle shape difference can cause radial segregation, but the segregation patters such as the locations of spheres and spheroids vary with the magnitude of the shape difference. With particle shape difference increasing, the segregation patterns could totally be reversed which can be explained by the reduced diffusion mechanism. For a long drum, the results show that for spheroid-sphere mixtures, spheres tend to segregate axially to the middle section of the drum, particularly for spheroids at aspect ratios = 0.67 and 2.0. The analysis reveals that the frictional end walls and the segregation developed in the radial direction induce the axial flow of particles, and are essential for the development of axial segregation.

Industry Centric Research Challenges in Australia – case studies from the Centre for Bulk Solids and Particulate Technologies

Williams, Kenneth (1)
(1) University of Newcastle

Keywords | Dust, Wet and Sticky Ores, Transportable moisture Limits, Biomass

With the boom in mining in recent times driven by iron ore production, and the recent expansion of mining for critical minerals and rare earth elements, materials handling challenges are still prevalent. In addition, the increase in biomass-type resource collection from waste streams has new challenges requiring investigation. Of interest is the recent work conducted by the Centre for Bulk Solids and Particulate Technologies in Australia on a number of research projects that are having a direct industry impact. Discussion on the water effect on a range of operational aspects in the iron ore and coal sector are presented where handleability indices and ship stability are presented. In addition, recent work on dust liberation and electrostatic adhesion, dust surveillance and health impacts are presented. Finally, biomass storage and associated off- gassing of CO, CO2, CH4 and associated o2 depletion volumes are discussed for wheat straw, wood chips, wood pellets and a native Australian plant mixture.

Optimisation of Material Handling Equipment for Biomass – How to optimise orange peel grab design for wood chips

Katterfeld, Andre (1); Otto, Hendrik (2)
(1) University of Magdeburg, ILM, (2) IBAF GmbH, Magdeburg

Keywords | grab, design, optimisation, wood chips, DEM

The switch from conventional power plants to renewable energy sources through the use of alternative fuels presents crane operators with new challenges. For the increasing mass turnover of wood chips, new grabs have to be developed, which ensures an economical and spillage-free unloading of ships.

This article presents an optimisation strategy of an orange peel grab for wood chips. The shape of the grab shells is modified with simulations in such a way that the closing forces on the grab shells are reduced in order to grasp the long-fibre wood chips safely. With conventional grabs, this leads to a cyclic consolidation of the wood chips and their jamming within the grab. Arch-shaped consolidations in the upper part of the grab create loose areas in the lower part. Due to the consolidation, it may happen that the shells of the grab would not close completely. The material which is falling out represents a major source for water-pollution.

The consolidation / arching can be simulated with the DEM, while at the same time the machine is considered by a multi- body model. Hence, it is possible to evaluate direct interactions and to derive geometry changes. In order to carry out the investigations, a grab optimised for wood chips was developed, which can unload large quantities of wood chips without any losses.

ORAL COMMUNICATIONS

Influence of moisture content on the bulk density of three granular materials usually stored in agricultural silos

Moya, Manuel (1); Sánchez, David (1); Villar, José Ramón (1)
(1) University of Extremadura

Keywords | bulk density, moisture content, wood pellets, wheat, corn, silo

Classical theories for silo design considers several mechanical properties for the determination of the loads generated within them. Since many years ago, it is known that these parameters are strongly dependent on the moisture content of the samples. Despite of that, not

too many studies can be found in the literature to estimate the influence of moisture content on them. In this study, a work is conducted to determine the variation of bulk density when moisture content is varied. For this purpose, a climatic chamber is used and different moisture contents are applied to samples of wheat, corn and wood pellets. The tests are conducted using the automatic oedometer devices available at the geotechnical laboratory of the University Center of Plasencia. Loads of 8, 16, 32, 64, 128 and 256 kPa are applied to the samples tested. When a load is applied to the sample, as soon as it reaches the primary consolidation stage the load is doubled. Once the maximum load (256 kPa) is applied to the samples tested, it is reduced to the half value, thus creating steps of 256, 128, 64, 32, 16 and 8 kPa. The velocity of these tests is key in order to maintain the moisture content of the samples tested. The real moisture content of the samples is determined by drying the samples tested at an oven at a temperature of 105-to-110 ºC. Moisture contents of 13 %, 14.3 % and 17.2 % are selected for corn, whereas moisture contents of 14.2 %, 15.2 % and 19 % are applied to the samples of wheat. Wood pellets will also be tested although at present it is not possible to determine the moisture content values that will be used considering the narrow range of moisture contents allowed by the European Pellet Council. The results obtained from these tests will be exposed in order to know the trend of each sample from the bulk density point of view as moisture content is varied. If possible, an approach to the variation of the Young's modulus as influenced by the moisture content of the samples will be determined, too.

Effects of stainless disk release conditions on overtaking behavior in the disks falling in an expanded polystyrene particle bed

Yoshida, Mikio (1); Kawabata, Daichi (1); Yamada, Hinano (1); Shimosaka, Atsuko (1); Shirakawa, Yoshiyuki (1)
(1) Doshisha University

Keywords | Falling behavior, Particle bed, Displacement volume due to falling, Packing fraction

Cooperative behavior (the multi-object formation of upward and downward convex configurations, which resembles the flying pattern of a flock of bird) was obtained when five stainless disks fell simultaneously in a low-density particle bed (Pacheco-Vázquez and Ruiz-Suárez, 2010). As one reason for generating this behavior, we focused on height difference and horizontal separation distance in disks generated by impacting between the disks and the bed surface. In this study, in order to change these parameters systematically, we released stainless disks in a low-density bed for various differences in the disk release time and initial disk separation distances, and investigated effects of the release conditions on the disk falling behavior.

We used expanded polystyrene (EPS) particles (diameter 5.08 mm, mass 1.45 mg) as the bed particle, and we put the particles in a transparent container (350 × 500 × 5.5 mm, width × height × thickness). One to five steel disks (diameter 25.4 mm, thickness 5.22 mm, mass 20.2 g) were used as the falling object. When we conducted a two-disk falling experiment, we changed disk release time differences (Δt = 0.022–0.154 s) and initial separation distances (Δl = 10–100 mm). We recorded the disk falling behavior in the particle bed by a high-speed video camera and analyzed the behavior by an image analysis. As a result, in the five-disk falling experiment, we obtained a cooperative behavior similar to that reported by Pacheco-Vázquez and Ruiz-Suárez (2010). In the two-disk falling experiment, overtaking behavior occurred for Δl of 10 mm and Δt less than 0.076 s, and for Δl less than 60 mm and Δt of 0.02–0.03 s. From the comparison of the one-disk and two-disk falling experiments, we confirmed that the overtaking behaviors was caused by decreasing falling velocity of the first released disk. This decreasing falling velocity of the first-disk arose from suppressing the displacement behavior of EPS particle around the first disk owing to the second disk falling.

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Tensile strength, the missing link to understand powder flowability

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Keywords | Tensile strength; flowability; powder handling; formulation

Flowability problems in process industries are quite common and can cause disruption to business. These problems can range from material handling issues to segregation or poor formulation; therefore, the process industries can face severe detrimental effects. Some of these pitfalls can lead to substantial differences in the scale-up and start-up performance of plants processing powders and significant reductions in designed throughputs or not achieving the desired formulated products.

During handling and storage, the flowability may be affected as the formulated blend or product can be subjected to different consolidation pressures, due to its own weight when stored at rest or ejected forces that provoke different stress conditions and deformation due to compression or shear during handling. These problems should be identified and addressed early in the development process however, due to the lack of sufficient knowledge or skilled personnel, it is common to see these pitfalls in industrial plants.

In this research, the tensile strength of a powder material is seen as a key parameter to predict powder flowability together with other particle properties such as particle size and density as well as derived properties such as the Bond number. The implementation of the measurement of tensile strength in order to predict the powder flowability is thoroughly reviewed with especial attention to cohesive powders and experimental methods focussing on preliminary stages of formulation and material handling predictions.

Model-based scale-up of the die filling process from lab-scale to production-scale rotary presses

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Keywords | die filling, rotary press, scale-up

For the industrial production of pharmaceutical tablets, large-scale rotary tablet presses are used. Here, the die filling is a critical process step as it determines the dose and uniformity of both mass and active ingredient. In addition, variations in tablet weight affect the porosity and tensile strength due to the variation of applied compression stress. Therefore, the die filling is the quality-determining process step. Depending on the material properties of the formulation, certain process parameter combinations have to be set to ensure complete and consistent die filling. During the scale-up to production scale tablet presses, the majority of conducted studies focused on the transferability of the compression process. However, the comparability of the die filling efficiency is only rarely taken into account. In order to enable a model-based description of the scale-up of die filling and thus to reduce the time required for cost-intensive process development, an in-depth understanding of the impact of process and material properties is necessary.

The focus of this study was to investigate the die filling efficiency of different pharmaceutical excipients on differently scaled rotary tablet presses (lab-scale: XL 100, production-scale: XL 400, X 3 (all three: Korsch AG)). In order to compare the different rotary presses, the filling time, tablet weight and bulk density were used to derive the volume flow rate which was evaluated for different paddle speeds. It could be shown that the determined volume flow rate is a precise and reliable descriptor of the filling process and thus, the volume flow necessary for complete die filling can be calculated across scales. Subsequently, the required paddle speed can be determined for a given formulation. Despite the reference to the bulk volume, influences of the different materials were observable and were attributed to the differences in flow-related particle properties (particle size, solid density, permeability, cohesiveness, ffc-value, dynamic angle of repose). As a consequence, the results of this study enable a model-based scale-up of filling results between different rotary tablet presses which differ significantly in size. It represents a novel and promising approach for the time-efficient development of new tablet formulations and reasonable process boundaries.

Bending Stresses in Elongated Crystals under Shear Flow

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Keywords | Crystal, DEM, breakage, pharmaceutical, dryer

Crystals with a ‘needle like’, acicular, habit and high aspect-ratios are commonly produced in the pharmaceutical and fine chemicals industries. These crystals are often broken during subsequent process steps, such as drying and mixing, affecting their bulk properties and having an

impact on both process and final product performance.

To understand, and predict breakage behaviour in such systems a discrete element method (DEM)study of elongated particles in a simple shear cell was undertaken. Rigid particles, with aspect ratios of 2 – 6, are modelled with clumped spheres. A Hertz-Midlin contact model with no cohesion is used to simulate a dry system. Mechanical properties of Beta- Glutamic Acid (β-LGA) were used. A fixed strain rate is used giving a quasi-static flow. Particle internal stress is calculated using Euler equation of motion for each particle.

A detailed study of the development of the bed structure as the particles become aligned during shear is reported. The order parameter of the crystals in the bed, the number of particle contacts and the porosity in the bed are investigated. The time taken to achieve steady state is shown to be dependent on aspect ratio but independent of normal stress.

Under steady-steady state conditions the contact force number and positions are investigated, as these are important to bending stress and breakage. The contact force concentration is shown to reduce with aspect ratio, and consequently the mean contact force, which is inversely proportionate to concentration of contacts, reduces with aspect ratio.

The particle bending stress dominates the internal stress in these elongated particles and the mean bending stress rapidly reaches steady state. However, the width of the distribution takes longer to reach steady state especially with the higher aspect ratio particles. Under steady state conditions the mean bending stress is shown to increase significantly with aspect ratio, indicating that under a given normal stress larger particles have a higher chance of breakage even under these steady state conditions where particles are aligned.

These models give useful insights into the behaviour of elongated crystals and help interpret breakage behaviours seen experimentally.

Simulation of the Gas Flow in a Fixed Bed Operated Degassing Silo with Analysis of the Gas Distribution and the Heat Transfer Zone

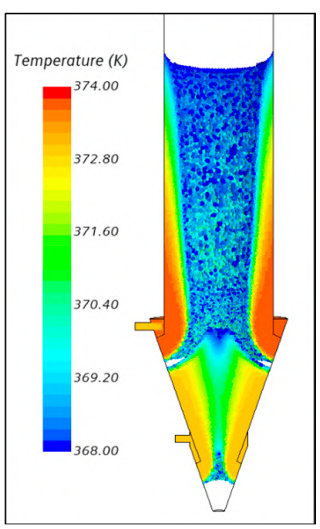
Schneider, Hans (1); Rist, Pia (1)
(1) Zeppelin Systems GmbH

Keywords | Silo, Fixed Bed Flow, CFD Simulation, Thermal Process

In many industries silos are used for heating, cooling and/or degassing of bulk solids by injecting a gas flow in the lower part of the silo which passes the material in a fixed or moving bed. For the degassing the product first needs to be heated to process temperature. As the hot air slowly heats up the pellets, a heat transfer zone forms and moves upwards while continuing the heating process. To optimize the design and operation parameters, it is necessary to know the shape and location of the heat transfer zone as well as the gas and temperature distribution.

The fixed bed is modelled as a porous media thermal non-equilibrium. The air supply is located at two levels in the silo cone, wherefrom the gas enters the fixed bed through an annular gap and leaves the silo at the top in the center.

The simulation is based on a pilot plant test, followed by a parameter variation of air supply and geometric modifications of the inlet geometry. The analysis of the results in Fig. 1 shows that the two-stage air supply results in an uneven temperature distribution.



Reason is a delayed heating of the lower cone when using a low gas volume flow there, which hinders the even heating of the silo core. This is coupled to a preferred heating of the product close to the wall by the upper air supply. If the product is only heated by the lower gas inlet, the result is theoretically a uniform temperature distribution. However, for reliable operation, the air velocities shall not exceed the minimum fluidization velocity. This is the reason why it is not possible to feed the entire air volume via the lower air supply only. By optimizing the air inlet geometry and consequently increasing the air flow rate accordingly to the maximum possible velocity, the formation of a cold core can be counteracted and thus a better temperature distribution can be achieved.

Conceptual Design of a Novel Grasping Gripper with actively stimulated Particles

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(1) TU Delft, (2) TU delft

Keywords | Soft gripper, Soft actuator, Environment stimulus, Active deformation, Particles jamming, Stiffness variation

Abstract

Compared with rigid grippers, soft grippers show adaptability and flexibility in grasping irregularly shaped and fragile objects. However, low stiffness and passive deformation of the soft grippers are the main limitations for the development of large-scale applications, especially for heavy objects or objects with sharp edges. Thus, the design of a novel grasping gripper based on packing particles is proposed to overcome this limitation. The proposed gripper element has two chambers made of membranes, filled with deformable particles with different shapes and sizes potentially made by shape memory polymers. In addition, the complete gripper is a combination of various elements to achieve sophisticated grasping tasks. The experiments with different gripper elements are carried out using simply shaped particles of different sizes to verify the feasibility of the structure. The results indicated: that the proposed grasping gripper exhibited the property of autonomous deformation by deformed particles which were stimulated by changes in environmental conditions. In addition, the bending angle of the elements increased with the growth of the difference in the particle equivalent diameters, structure length, and elastic modulus of the membrane. In conclusion, the grasping gripper can realize the dual functions of stiffness improvement and autonomous bending but performs differently under different influence parameters. Thus, the proposed novel active robotic gripper can provide guidance for the development of soft grippers in multi-scale physical scenarios.

Keywords: Soft gripper, Soft actuator, Environment stimulus, Active deformation, Particles jamming, Stiffness variation

Micromechanical analysis of flow and energy dissipation during the discharging process of a rectangular hopper handling polydisperse particles

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Keywords | Hopper discharge, GPU-based DEM, polydisperse, particle size distribution, energy dissipation

Hopper discharging dynamics have been extensively studied under different geometries and particle properties, yet many problems are still recurrent. One of the most widely used methods of studying hopper flows is the Discrete Element Method (DEM). Limited by computational loadings, the previous DEM studies have focused on single and binary sized particle systems. However, polydisperse systems are very common in nature and industry. This talk will present a numerical study of the hopper discharging process of spherical polydisperse particles. The model is a Graphical Processing Unit (GPU) based DEM, which is necessary to realize large scale simulations to consider polydisperse particles. The results are analyzed in detail to develop a comprehensive understanding of the effects of polydispersity, especially on discharging flow dynamics, microdynamic response, and energy dissipation mechanisms. The particle size distributions (PSD) considered follow a discretized log-normal distribution, characterized by the particle size fractions d_{10} and d_{90}. Two configurations of the PSD are investigated, namely number-based and mass-based distributions with a constant arithmetic mean particle size and variable spread parameter, yielding particle size ratios d_{90}/d_{10} from 2 to 8. The energy analysis includes conservative and dissipative terms driven by particle-particle and particle-wall contacts. The dissipative mechanisms are differentiated for collisional and frictional interactions. The results show that the change of PSD spread yields opposite trends of the discharging flow rates for number- and mass-based distributions,

demonstrating decreasing and increasing discharge rates, respectively, as the PSD spread becomes wider. Consequently, the packing and flowing structures change significantly as the size dispersity increases. For instance, the average contacting forces increase for the number-based distributions but decrease for mass-based ones. However, with a widening PSD spread, the total dissipated energy increases for both PSD configurations to a similar degree. On the other hand, the spatial distribution of the dissipating energy shifts significantly towards the hopper centreline for the number-based PSD and concentrates along the walls for the volume-based one. Although the energy dissipation in the hopper varies spatially, their total value does not change much with respect to size distribution.

An Investigation into moisture migration through two distinct moisture layers of iron ore fines

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(1) University of Newcastle, (2) West Australian Iron Ore

An Investigation into moisture migration through two distinct moisture layers of iron ore fines
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This research investigated the migration of moisture of layered iron ore on a belt conveying system. A wet layer of material was sandwiched between two rested moisture layers, the material bed underwent a period of oscillation and then moisture migration was assessed. The experimental equipment used for this investigation is an oscillatory type drainage tester designed and built at the University of Newcastle. The experimental system is comprised of a drainage bench and an oscillatory frame. The drainage bench accommodates six material columns. For this project, each material column was stacked up with six Perspex cells, which is reflective of the burden depth of the ore on the conveyor belt. The inner diameter of each cell is 140 mm and the height of each cell is 80 mm.

Two different motions were analysed for the testing performed in this report. The frequency was held constant at 3 Hz and the peak accelerations tested were 1.5 m/s2 and 2.0 m/s2.

A summary of project findings include:

- There was minor water migration in both directions, with somewhat more moving towards the bottom.
- There was more migration of water into the 2% base moisture cells than the 4%.
- There was no water accumulation through the porous base during the time period tested.
- There was no evidence of fine particle migration in either direction

Transport and deposition of fine particles using electrostatic acceleration in a powder process

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Keywords | particle charging, transport, electrostatic acceleration, particle motion control, deposition

The precise manipulation of fine particles with high adhesion and cohesion is required for solid-state coating and additive manufacturing. In this study, alumina particles were continuously charged, dispersed, transported, and deposited on surfaces. Fig. 1 shows the system with a powder feeder, an electrostatic nozzle, an electrostatic accelerator, electrodes to control particle motion, and a target electrode to deposit them. First, particles were charged by induction with the same polarity as that of the nozzle where voltage was applied, followed by downward ejection. Thereafter, the direction and motion of particles were controlled using the extraction and screen electrodes. Multiple mesh electrodes were used to homogeneously distribute the particles in space. Fig. 2 shows the particles uniformly deposited on the target electrode. The specific charge of the particles measured with a Faraday cup increased with increasing applied voltage. Furthermore, using square-wave voltage for the nozzle, the charge of the deposited particles was maintained at a constant value, thereby suppressing back discharge.

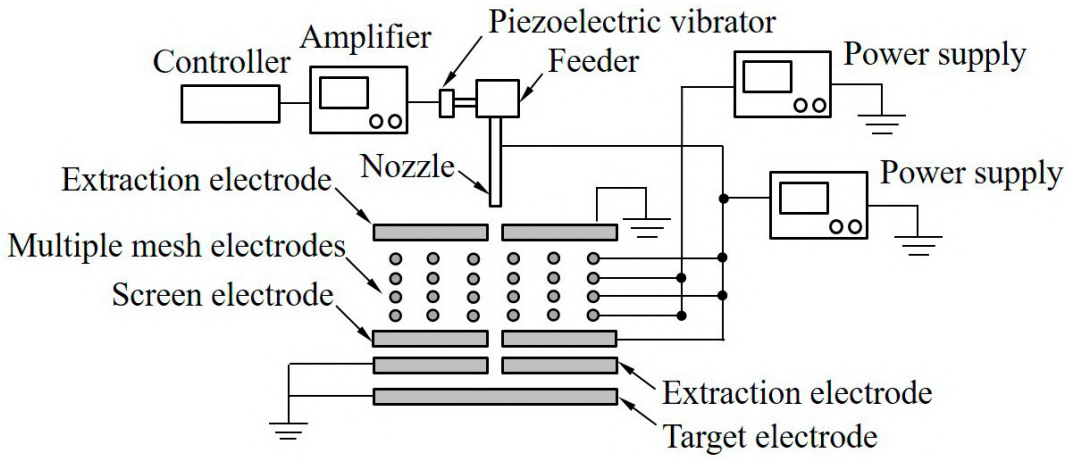


Fig. 1. Particle deposition system.

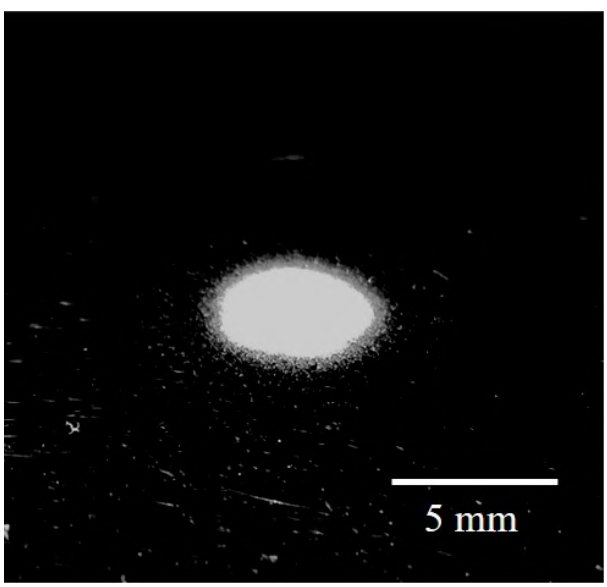


Fig. 2. Particles deposited on the target electrode.

Collision Characteristics of Non-Spherical Particles on Ductile Surface under Normal Impact: Numerical Investigation

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Keywords | Particle-wall collision; Normal impact; Coefficient of restitution; Non-Spherical particle; Granular materials

Particle-wall collisional behavior is considered to be one of the most important factors governing particle movement in multiphase flow simulations. The coefficient of restitution (COR) is frequently used to characterize particle-wall collisional behavior. The more accurate the COR considered for numerical simulations, the more accurate the resulting outputs will be. Calculating COR for spherical particles is simple; however, calculating COR for non-spherical particles, which are common in real-world applications, is more difficult due to the particles' unpredictable behavior after impact. In this paper, the COR during the normal impact of a rigid prolate ellipsoidal particle on the target wall is investigated using the finite element method (FEM). The simulations are conducted with a particle of sphericity 1, 0.9, 0.7, and 0.5 impacted at different orientation angles (angle between particle major axis to the horizontal plane) in the range 0o to 90o on four different ductile surfaces, namely Aluminum, OFHC Copper, Steel 1006, and Armco Iron. The loss in kinetic energy of the particles after impact is used

to analyze the COR. The effect of particle sphericity, particle orientation before impact, and impact velocity on COR are determined. The present study helps to provide important engineering insights to calculate the motion of non- spherical particles in multiphase processes using the discrete element method (DEM).

Measure Particle Velocity in Pulse Injection Pneumatic Conveying via Electrical Current Signal Analysis

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(1) University of Ottawa

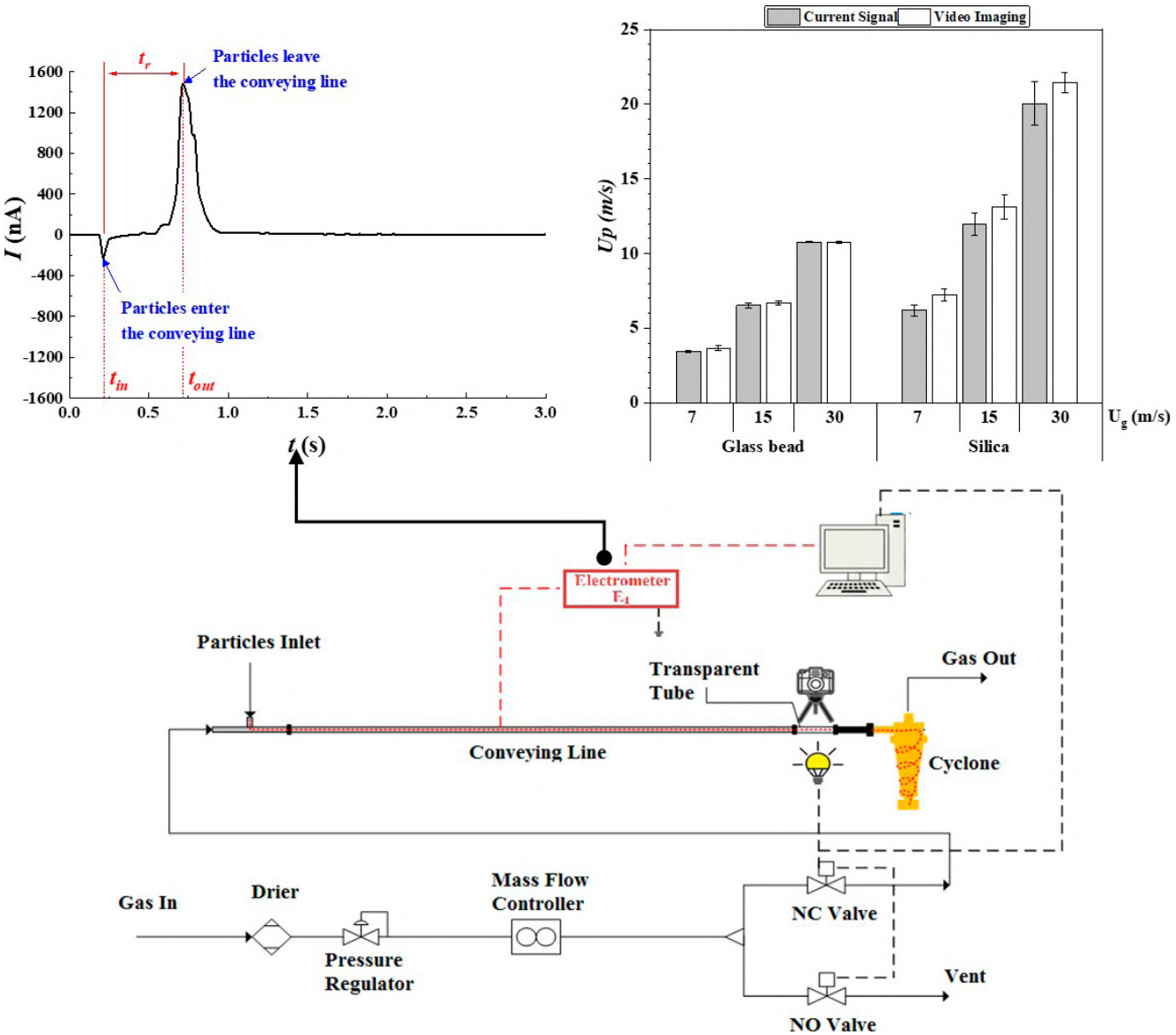
Keywords | Electrostatics, particle velocity, pneumatic conveying, video imaging

The goal of this work was to measure particle velocity (Up) in pulse feeding pneumatic conveying utilizing electrical current signal analysis with a new approach. To measure particle velocity using electrostatic method, the cross-correlation function of signals measured by two electrodes is plotted, and the maximum peak in the cross-correlation function corresponds to the residence time of particles between the two electrodes which in turn is converted to particle velocity (Yan et al., 1995). The accuracy of the particle velocity measured by this method could be influenced by the distance selection and calibration of the two probes, and ill-defined peaks (Yan, 1996). In this work, this technique is modified to improve the simplicity (i.e., eliminating signal conditioning and cross-correlation calculations) and minimize the associated drawbacks in measuring particle velocity in pulse injection pneumatic conveying. In this new approach, the conductive tube wall is directly connected to an electrometer. The residence time of particles (tr) is calculated from the two peaks in the electrical current signals directly measured from the conveying tube as the solids enter and exit the line. Knowing the conveying tube length, and the particles' residence time, the particle velocity is then calculated. The results from this method were compared with a video imaging technique.

0.1 g of glass beads and amorphous silica particles were pneumatically conveyed in a 4.57 mm (1/4 inch) in inner diameter and 3 m in length stainless-steel 316 tube, electrically isolated from the system at its inlet and outlet. Dry air was used as the conveying gas with velocities (Ug) of 7, 15, and 30 m/s. The results showed that particle velocities measured by the two techniques were in a good agreement for all the operating conditions tested. The new proposed method based on electrical current to measure particle velocity in pulse-injection pneumatic conveying has simplicity and reliability.

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Tubular Push Conveyor – a new mechanical conveying system with gap effect

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Keywords | mechanical conveyor, gap effect, motion resistances

The tubular push conveyor, which is developed and marketed by the Bühler AG under the brand name TUBO, is an innovative conveying system for the transport of non-mineral bulk materials. It sets new standards, especially in agricultural and food technology, in terms of flexible conveying routes and simple design. However, the conveying principle is also applicable in chemical and process industry if low-abrasive materials are conveyed. Unlike today's systems, the bulk material is transported in a closed tube without a tension device (rope or chain) but by the help of push elements [1].

The paper will present the general layout of such conveyors as well as the basic calculation approaches for the motion resistances. The calculations will be compared with experimental measurements on an industrial sized test rig. Out of this analysis the gap effect dependent on the particle size distribution and the gap size between push elements and tube can be highlighted as a source for additional motion resistances. This is similar to other mechanical conveying systems like tube chain conveyors, screw conveyors and trough chain conveyors.

The paper will present a general overview of the gap effect in mechanical conveying systems and the latest results regarding the prediction of the gap effect.

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Wear deformation of a convex pattern surface using DEM and deformable geometry technique

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Keywords | wear; convex pattern surface; deformation; DEM

A convex pattern surface has been proposed (Chen et al., 2017) and optimized (Yan et al., 2021) to reduce the sliding wear of bulk handling equipment, by adjusting the flow behaviour of the bulk material. In this study, the deformation process of the convex pattern surface will be modelled. We combine the discrete element method (DEM) with an Archard wear model and a deformable geometry technique to capture the sample deformation. The accuracy of this method is verified by modelling the deformation of a plain surface caused by one particle (Yan et al., 2022).

Figure 1 indicates a graphical abstract of this work, including a deformation technique, simulation setup and main findings. The simulation results include stability analysis of the simulation setup and wear result comparisons. Stability of the simulation setup is analysed by evaluating the flow behaviour of the bulk material and the wear rate of the sample. For the wear results, the wear volume is compared between a plain surface and the convex pattern sample. The resulting wear distribution is shown to reflect the effect of the convex pattern on adjusting bulk flow behaviour. Also, a dynamic deformation process of the sample is reconstructed. Through the wear result analysis, we will conclude to what extent the convex pattern sample can keep reducing wear compared with a plain surface with the deformation of the sample.

Cohesive Bulk Solid Discharge Behavior from Various Belt Conveyor Configurations

Del Cid, Liz (1)
(1) Jenike & Johanson, Inc.

Keywords | Trajectories, DEM, Cohesive Materials

Guidelines for the discharge trajectory of material off a belt conveyor can be closely approximated by CEMA (1) or Roberts (2, 3) assuming circular segments or parabolic sectional profiles, respectively. Typically, belt discharge trajectories fall into two categories: slow-speed and high-speed trajectories. The influence of bulk solid material properties, such as cohesive materials and non-idealized shapes (e.g., spherical), is still missing from these guideline approaches. In recent years, advances in the calibration process of discrete element method (DEM) models have improved confidence in these simulation predictions and can with greater confidence be used to investigate the influence bulk solid material properties have on belt trajectories.

The focus of this study evaluates the impact cohesion and particle shape have on trajectories of varying belt inclination angles, and trough geometries (e.g., flat belts, picking idles, and 35- and 45-degree idlers) and compares those to an estimated guideline in the absence of transitional geometry. The trajectory model used in this study is based on CEMA's transfer point discharge trajectories and the DEM model on a linear-Thornton contact model (3). The work presented shows the changes in the trajectory path and cross-sectional profile as influenced by cohesive material characteristics.

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Experimental measurements of the pressures exerted by wood pellets in a model silo with corrugated steel walls

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Keywords | Corrugated silo wall; friction; experimental tests; wood pellets

Steel silos with corrugated walls represent a technical solution widely used in industrial applications due to a more economic consumption of material and smaller weight, compared to the smooth wall solution. However, corrugations affect the friction between the stored material and the wall, resulting in changes in the flow of the stored material and the pressures exerted over the silo walls. Based on the findings of previous experimental works, the European standard EN 1991-4 theorized that the value of the coefficient of friction for a corrugated steel surface can be placed between the coefficient of friction for a smooth metal sheet wall and the coefficient of friction between grains, although important issues are not yet fully addressed when calculating the pressures exerted by the stored granular materials against corrugated walls.

In this research, experimental tests have been performed in a rectangular model silo 150 cm high with a 45 cm square cross section. Two of its walls were made of transparent plastic, and the other two of corrugated steel sheets. Tangential and normal forces exerted by the stored granular material at different heights were measured by sensors attached to the corrugated steel sheet. The vertical section of the model silo is supported on load cells, in order to register the vertical loads exerted by the stored material during the discharge. Cylindrical biomass pine-wood pellets of 6 mm diameter and an apparent mean density of 660 kg/m3 were used to carry out the tests.

The effective coefficient of friction exerted by the stored material over the corrugated wall and the flow rate of the granular material stored in the silo were obtained and compared with those proposed by the EN 1991-4 standard.

Experimental measurements implemented in this research will allow the validation of numerical models developed to study the influence of the mechanical properties of granular materials at different handling processes and a better understanding of the actual behavior of granular materials stored in corrugated wall silos.

Analyzing and Structuring Silo Failures

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(1) Wilms-ITC, (2) Zeppelin Systems GmbH

Keywords | Silo Design, Silo Failure Analysis, Silo Loads

Silo malfunctions and failures, both operational and structural, cause plant downtime, significant implications on plant operation and immense cost for modification or repair. Thus, analysis of respective failures needs to be structured and as quick as possible. However, there are only very few failures with a clear single cause. Typically, only a combination of wrong design assumptions or operating conditions can explain a failure.

Numerous failures have been analyzed in the past and some cases with typical failure causes are presented. Failure causes are design faults, operational mistakes, wrong assumption of solids flow properties and malfunctioning feeders. Experience from a variety of failures is used to establish a check list on potential failure causes. This check list (see example below) can be used to investigate silo failures in the future more quickly.

Causes of Silo Failures	Possible cause	Likeliness	Impact	Comment
Assuming a too low bulk density and a too low total weight of the silo content	yes	low	low	deviation up to 10%
Assuming a wrong wall friction coefficient/wall friction angle or a wrong surface qualtiy	no	low	low	within assumed range
Assuming a wrong flow profile and thus a wrong pressure distribution	no	no	no	-
Assuming wrong flowability and ignoring discharge flow problems	no	no	no	honking has little influence
Ignoring non-uniform flow and eccentric flow channels due to unknown discharge or operation modi	yes	high	very strong	semi-open slide gate valve
Eccentric discharge	yes	high	very strong	operation of sample nozzle
Collapse of arches and stagnant zones	no	no	no	-
Settlement or uneveness of the foundation	unknown	medium	strong	grouting procedure unknown
non-uniform support or non-uniform stiffness of the support structure	unknown	medium	strong	grouting procedure unknown
High wind loads or internal vacuum	no	low	low	vacuum from aspiration in case of blocked vent hood
Overpressure or explosions	no	no	no	-
Excessive earthquake loads	no	no	no	-
Influence from temperature of humidity	no	no	no	-
Loads from super-structures or conveying systems	yes	medium	medium	additional aspiration pipe support
External forces from walkways or connctions to buildings or other silos	no	no	no	walkways not fixed
Constraints from rigid connection to other silos or structures	yes	no	no	Rigid aspiration pipe connection
Loads from and on internals	no	no	no	-
Disregard or wrong interpretation of design rules	no	no	no	-
Disregard of operating manual	yes	high	strong	discharge through sample nozzle
Insufficient weld quality or wrong weld seam classification	no	no	no	-
Insufficient reinforcement steel	no	no	no	-
Prestress resulting from assembly of poorly aligned or fitting segments	yes	high	high	hopper insertion weld seam; fit of hopper
Unfavorable execution of assembly	yes	medium	medium	hopper insertion weld seam; fit of hopper
Assuming wrong design properties and permissible stesses of material of construction	no	no	no	-
Special influences / parameters	no	no	no	-

The evaluation of failures by means of such a check list can also assist in attributing the responsibility for certain design or operational mistakes to the respective party and thus this proposal may serve as a tool to settle any economic dispute resulting from repair or modification cost.

Biomass off gassing and self heating

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(1) University of Newcastle, (2) NSW Department of Primary Industry

An Investigation into Off-Gassing, Self-Heating and Aging of Biomass Materials Stored in an Anaerobic Environment
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Wood pellets, wood chips and other biomass materials were tested for their self-heating, off-gassing and aging properties during storage. Approximately 15 L of material was placed into the storage containers that were then sealed and equipped with a thermocouple and a gas-collection port. The containers were stored at ambient temperatures over a period of six months – the temperature was not controlled and allowed to fluctuate freely.

The temperatures in the buckets were continuously monitored and gas samples taken to be analysed using GC-TCD to obtain concentrations of carbon dioxide (CO2), carbon monoxide (CO), oxygen (O2) and methane (CH4). At intervals of approximately 10 days, 1, 2, 3, 4 and 6 month intervals, the samples were open and tested to obtain compactive bulk material properties. It was found that biomass produces significant and potentially harmful amounts of the CO and depletes oxygen. Some CO2 and CH4 generation were also observed. No significant self-

heating at this scale was observed, however some sample showed mass gain, which is likely due to oxidative bonding.

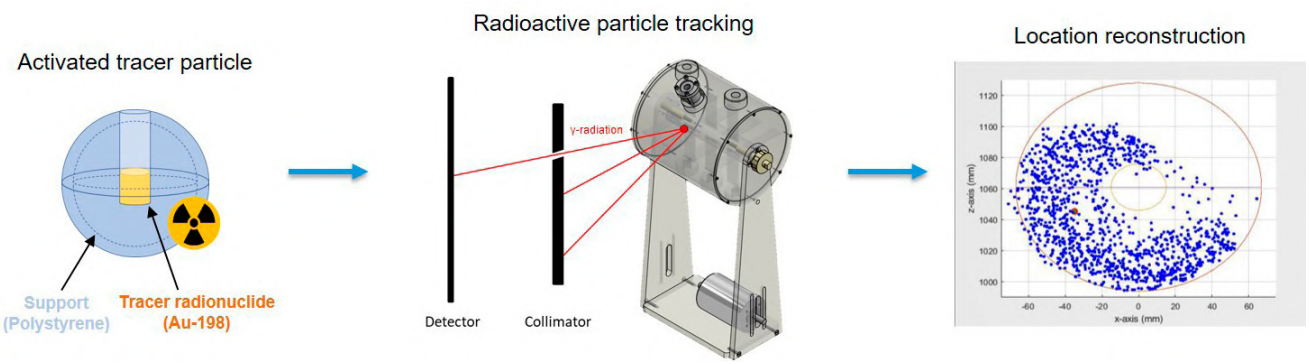
Elucidating particle flows in horizontal stirred bed reactors by radioactive particle tracking

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Keywords | Horizontal stirred bed reactor, radioactive particle tracking, solids mixing

Horizontal stirred bed reactors are commercially employed in polypropylene manufacturing processes. The reactors generally contain a bed that is mildly stirred by a series of paddles attached to a center shaft. The agitation induces a forced circulation, which brings particles to the top of the bed. At the top of the reactor, condensed propylene monomer is sprayed in order to remove the heat originating from the strongly exothermic polymerization reaction in the bed via evaporative cooling. It is crucial to have a well-mixed system with a narrow particle cycle time distribution throughout the reactor to avoid the formation of hotspots that could lead to expensive reactor shutdowns due to lump formation. It is therefore of great importance to understand the particle flow in horizontal stirred bed reactors. Simple optical techniques are limited to investigating particle flows close to the wall region, because the large fraction of the particulate phase makes the dense flow opaque to visible light.

In this work, we developed a lab-scale horizontal stirred bed reactor that enables particle flow studies in non-reactive environments. Since optical techniques are inadequate to study the dense flows, we used our in-house radioactive particle tracking setup to study the trajectories of a representative tracer particle inside the reactor. The setup consists of three scintillation detectors that form a field of view around the reactor. Within this field of view the location of the tracer particle can be reconstructed via single-photon emission computed tomography (SPECT) with sub-millimetre and sub-second accuracy. We studied the effect of the reactor filling (35 % - 55 %) and the agitator rotation speed (10 RPM – 60 RPM) on the particle cycle time distribution, axial dispersion and velocity field. At higher reactor fillings and higher rotation speeds the particle has a shorter cycle time, which would lead to improved heat removal in the process. The results from this study contribute to better understanding of the flow behaviour in horizontal stirred bed reactors and design processes for optimal performance of reactors in the industrial manufacture of polypropylene resins.



Understanding the mixing behavior of dry and wet soda ash powder in horizontal rotating cylinder

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Keywords | mixing, wet powder, dry powder, soda ash, sodium carbonate

Conversion of sodium carbonate to sodium carbonate monohydrate is important process in soda ash industry. The monohydrate crystals are obtained from the phase transformation process of sodium carbonate particles to sodium carbonate monohydrate crystals in presence of water. For continuous large scale production of monohydrate a horizontal rotating cylinder is used in soda ash industry. The particle flow

in the rotating cylinder is a complex system and hence difficult to predict or model. The residence time distribution is one of the useful tool utilised in chemical engineering to characterize the mixing of particulate matter in equipment.

In the present work residence time distribution is obtained for dry sodium carbonate particles and wet monohydrate crystals moving along the horizontal rotating cylinder. The variance of the distribution shows that wet monohydrate crystals have higher values than dry, indicating higher degree of mixing. In addition, for wet monohydrate crystals, the variance is function of amount of water present in the particulate matter and other operating conditions have little effect.

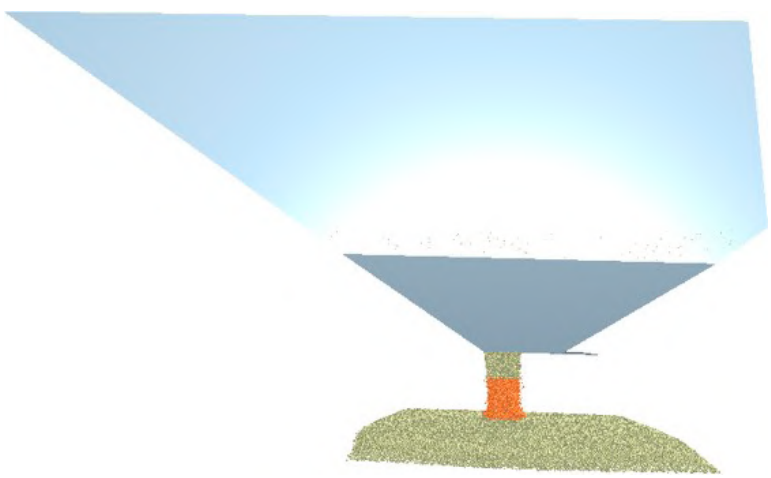
Mechanisms of Dust Generation During Grain Handling

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Keywords | Dust; grain handling; DEM modeling

Corn is the largest crop grown in the U.S. and supplies most food, feed, fuel ethanol, and pharmaceutical industries. Due to the large and growing volume of grains handled, and based on the nature of grain handling and processing equipment, there is an increased hazard to the health and safety of workers from the dust generated. On average, 1 metric tonne of grain contains about 1-5 kg of dust. The essential role of grain dust in causing grain dust explosions is clearly established, but there is limited understanding of the adhesion mechanism of dust particles onto the grain and surfaces, the mechanism of separation of dust from grain kernels, their inter-particle bonds, and the dispersion pattern of grain dust. Therefore, the objective of this study is to use fundamental tools and engineering concepts to study the mechanisms of dust generation during handling of corn.

The adhesion force that holds grain dust particles to the grain itself is an important factor in better understanding and mitigating dust separation from grain kernels and the resulting dust cloud generation. The dust particle attachment strength ranged from less than 4.6 × 10-10 N to 2.1 × 10-8 N. In addition, results showed that freshly harvested corn samples contained a higher presence of small particles with low circularity than older, lower quality samples. The dust particles that were more strongly attached to corn kernels tended to have lower surface roughness than those that were weakly attached for the freshly harvested grain. In order to find out ways to suppress dust emission, a discrete element model (DEM) was developed (Fig. 1), and experiments were conducted using corn kernels on determining the emission rates of total suspended particulates (TSP) at three-grain flow rates and two drop heights during receiving operation at a grain elevator. The results indicated that the dissipated energy contributed to the dust separation from corn kernels. The TSP emission rate increase with increased grain flow rate and increased grain drop height. Decreasing the exposure of grain from stream of air significantly decreased the TSP emission rate.



End-use properties of powders from flowability to dustiness

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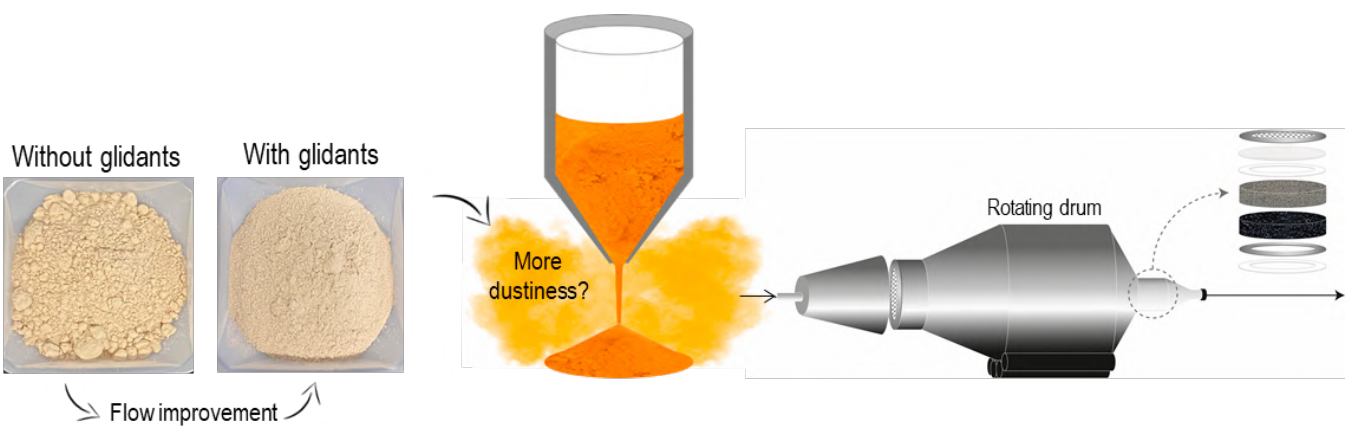
Keywords | Flowability, dustiness, glidants, colloidal silica

Powders are part of the design, production, and final products of different industrial sectors. Their handling, packing, and storage are subjects of interest because of the difficulties arising during manufacturing due to flow problems. Powder flowability can be improved by: facilities modification, which is a seldom universal solution; by the powder's fluidization, involving separation operations and increasing process complexity; and by the addition of nanoparticles, which is considered the more effective solution with the best benefit/cost ratio. Nowadays, nanomaterials are essential to the product conception of many industrial sectors, such as medical, cosmetic, food, and aerospace. Silica nanoparticles (S-NP) are widely used as excipients for different purposes. From a technological point of view, the addition of glidants in powders has been based on practical experience, trial, and error, leading to about 0.5 to 2% wt. of S-NP. From a safety point of view, the regulation entities consider S-NP as non-toxic and nonirritant, and the FDA[1] and the EFSA[2] approve S-NP as a food additive.

Notwithstanding, their toxicity has been a persistent concern over the years. Several in vitro studies show that S-NP are toxic in different types of human and animal cell lines. The effects of nanoparticles on individuals and the environment have not yet been thoroughly evaluated, and this will only be successful with a worldwide scientific and legislative effort.

Our project aims to determine the relationship between two end-use properties of powders, their ability to flow, enhanced by the use of glidants, and dustiness. Here, we use four flow additives to improve the flowability of four industrial powders (microcrystalline cellulose, wheat flour, joint filler, and glass beads) and evaluated dustiness using a rotating drum and a vortex shaker. Our findings suggest that adding glidants to improve flow behavior increases the sample's dustiness, composed of colloidal S-NP suspensions.

[1] US Food and drug administration
[2] European food safety authority



Inertization of ignition in biomass dust layers

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Keywords | Inertization, dust layers, safety biomass flammability

The use of biomass has heavily increased in the past years, and so has the number of accidents related to its storage, transport, and use. In this context, it is important to define the flammability and explosion characteristics in order to have a proper knowledge of material's behaviour and prevent accidents (Eckhoff 2003). The present work aims to study ignition inertization in biomass dust layers. To do so, wood pellets were milled and sieved obtaining a < 1 mm particle size sample. The sample's flammability characteristics were defined trough minimum ignition temperature of dust layer and cloud (MIT-l and MIT-c) and minimum ignition energy (MIE). Moreover, the characterization was completed using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) so moisture, volatile content, maximum

weight loss temperature, etc., were defined.

Furthermore, the present study focuses on a possible solution to biomass flammability tendency by adding solid inert material (Danzi, Marmo, and Riccio 2015; Janés and Carson 2013). In particular, two different inert materials were used (sodium bicarbonate and recycled glass) and mixed with biomass at different concentrations (30%, 50% and 70%). Once the mixed samples were produced, minimum ignition temperature of layer (MIT-l) was defined for each sample, so the inerting effect was clearly noticed. Additionally, the samples (both raw and mixed with inerts) were tested using TGA and DSC techniques in order to analyze their thermal behaviour, thus allowing the definition of a threshold that indicates the optimal inert concentration that significantly increases MIT-l while the heating value is not substantially reduced.

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Analysis and reduction of the risk of fire explosion in pyrotechnic stores

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Keywords | Safety, fire explosion, pyrotechnic, flammability risk

A fire inside a pyrotechnics magazine can cause the simultaneous initiation of the stored articles, with the wave of shock that is produced promoted by the pressure of the gases generated in its interior. The purpose of this research is to analyse the phenomena that develop as a result of a fire inside the pyrotechnics stores and establish possible effective prevention and protection measures to reduce the risk of explosion.

An analysis of the risk of explosion and fire consequences in a pyrotechnic magazine, testing to real scale, was initially carried out. The measurements of Reflected Pressure recorded in the tests are consistent with the predictions of the mathematical model in the TM 5-130 manual for black powder (Department of the Army, Department of the Navy, and Department of the Air Force 1990). Limiting the maximum permissible load, considering the volume of the store, would reduce the seriousness of the consequences in the event of an accident. However, the maximum permissible levels should be so low as to make their use for retail sale of pyrotechnic products unviable.

Therefore, and in order to reduce this risk of explosion, it is necessary to minimize the possibility of a fire inside a pyrotechnic magazine. Different systems of detection and automatic extinction of fires in pyrotechnic magazines were evaluated, testing to real scale. Once a fire has started inside a pyrotechnic product packaging, it is very difficult to achieve its extinction, so it is necessary to try to prevent it from spreading to nearby packaging by a rapid detection (using a smoke detector, optical, ionic and/or suction) associated with automatic extinguishing and an extinguishers agent with a high cooling capacity which prevents the spread of the fire (water or foam).

The results obtained have allowed the Ministry of Industry, Energy and Tourism, update the current regulation (Complementary Technical Instruction 17 of the Regulation of Pyrotechnic Articles and Ammunition, approved by the Royal Decree 563/2010, of May 7).

FLASH COMMUNICATIONS

Numerical analysis of granular dynamics in a full-scale continuous blender using DEM

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Keywords | DEM, Continuous blending, Residence time distribution, Powder flow, GPU computing.

Continuous powder blending is an essential unit operation during continuous pharmaceutical manufacturing. However, the complex granular dynamics in the blender, which is valuable for understanding the continuous powder blending process, is still poorly understood. This study employs a graphic processor unit (GPU) enhanced discrete element method (DEM) to analyse the granular dynamics in a full-scale powder blender with the forwards-alternating-forwards blade configuration. The impact of impeller speed on the blending performance, such as the mean residence time, the residence time distribution (RTD), the hold-up mass, the mean centred variance, and the number of blade passes, is systematically investigated.

Numerical results indicate that only a small fraction of powder distributes in the upper region of the blender when it operates at the steady-state. In contrast, most of the powder distributes in the middle and lower regions of the blender as shown in Figure 1(a). It is found that the impeller speed has a significant influence on the hold-up mass and the time needed to reach steady-state, which can be identified in Figure 1(b). Besides, Figure 1(c) reveals that the total path of individual particles has a strong correlation with the corresponding residence time. This study further demonstrates that the GPU-enhanced DEM is a powerful tool to simulate continuous powder blending process at scale and can be promising to analyse powder flow behaviours during continuous pharmaceutical manufacturing.

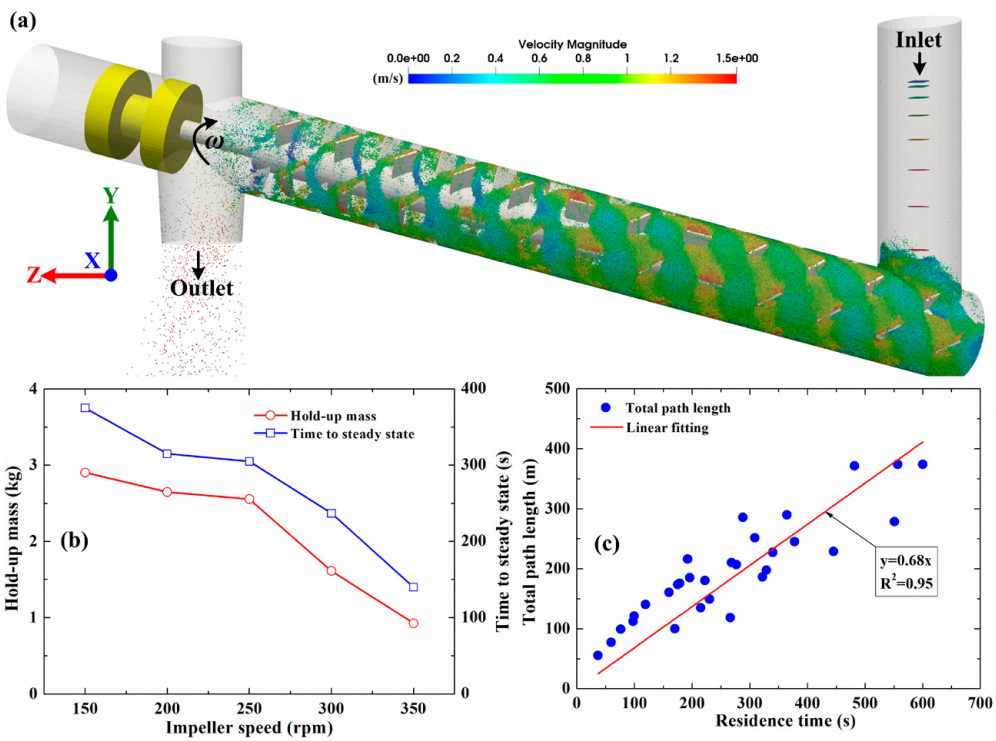


Figure 1. (a) Powder flow profile inside the blender; (b) Hold-up mass and time to steady state under various impeller speeds; (c) Relationship between total path of particle and the corresponding residence time.

Energy Efficiency: Economic use of compressed air in pneumatic conveying for bulk solids

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Keywords | blow tank; energy efficiency; pneumatic conveying; pressure vessel; solids feeder.

Pneumatic conveying of powders is a unit process extensively used in industries for the handling of particulate material of several segments.

The studies started from dilute phase pneumatic conveying and, in order to produce better results in industrial settings, it has evolved to include energy efficiency as a significant part. Many works were done to understand and model pneumatic conveying systems and bulk characteristics. However, they are highly empirical and the conclusions are, in most of the cases, limited to experimental conditions. This paper uses computational mechanics, mathematical and applied sciences to introduce a systematic method to select the air pressure and flow necessary to operate an energy optimised pneumatic conveying system. The method has been tested and applied to a pressure conveyor fed by a compact blow tank of 100 L in a 133 m long pipeline with diameter of 3 inches. It has been demonstrated with advances in engineering the possibility of controlling the conveying system with only two input parameters in order to operate at the desired condition of pressure and airflow and consequently the respective conveying rate and energy efficiency.

2 PARTICLE AND PARTICULATE SYSTEMS CHARACTERIZATION

KEYNOTE LECTURES

Application of bootstrap method to particle size distribution (PSD) analysis

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Keywords | PSD, uncertainty, statistics, log-normal distribution

How many particles shall be counted, i.e. sample size, is always a question in particle size distribution (PSD) analysis. To answer it, distribution information is necessary in advance: thus this is a 'chicken and egg' question. The question, therefore, may be converted to how uncertainties (due to sample size) can be evaluated after a PSD measured. Conventionally they were evacuated through parametric analysis, but here bootstrap method can be used as a powerful tool, because of features such as (1) non-parametric analysis, (2) flexibility to evaluate of statistical quantities of interest and (3) simplicity of computational protocol to process a real data set.

Bootstrap procedure is simple:

(1) One-time real measurement obtains a set of particle size, of sample size (number of the data) N.

(2) Resample, with replacement, randomly form the real data set to produce bootstrap sample of sample size N. Note that this step is only made as a data processing, not as real measurement.

(3) Repeat the step (2) for B-times to produce B sets of bootstrap sample.

(4) Evaluate uncertainties of any statistical quantities of interest from B sets obtained in (3).

In other words, bootstrap method is an approximation method to use virtually reproduced data sets for statistical analysis instead of repeated real measurement of B times.

Comparisons between real sampling and bootstrap approximation were performed by numerical simulation using log-normal distribution, and it was demonstrated that the bootstrap method is useful, as far as PSD is given as number based. Next challenge must be how the bootstrap method can be applied for mass (volume) based (mass weighted) distribution. Again numerical evaluations were performed with rog-normal distribution. The detailed results will be reported: (1) bootstrap method is effective for mas weighted distribution (2) when distribution is wider, the required number of particles increases a lot, as usual known. (3) when the sample size is too small, bootstrap method gives too optimistic results; diagnosis strategy for this case has to be developed.

Monitoring particle interactions in heterogeneous processes with FBRM

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Keywords | Particle interactions, flocculation, particle monitoring

Heterogeneous processes involve many interactions among particles and between these and polymers or other soluble compounds in the continuous phase. The results of these interactions often determine the performance of the process. Focused Beam Reflectance Measurement is a technique that can be used to monitor many of these processes. It gets chord lengths distributions of particles or bubbles even each 2 second. This allows monitoring any change in their aggregation, size or shape.

The aim of this communication is to share the experience of the Cellulose and Paper Research Group of UCM in developing new methodologies based on FBRM measurements for studying particle interactions in different sectors:

- Interaction among particles, a key to develop optimal methods to prepare slurries containing new particulate additives, as for example, the use of mineral rheological modifiers in cement (Jarabo et al, 2010).
- Interactions of particles with polymers in flocculation/dispersion processes (paper, cement, sludge, water treatment, colloidal silica removal,....). Studying the interaction of polymers with different particles in suspension under different shear forces allows determining floc properties and identifying flocculation mechanisms (Hermosilla et al, 20012; Fuente et al., 2005).
- Interactions with air (pulp and water treatments by dissolved air flotation). Monitoring the bubbles coalescence during the process allows identifying the optimal conditions for flotation process (Saarimaa et al, 2006).
- Interactions of particles with nanofibers, to replace flocculants in papermaking and dispersants in cement (Figure 1). Interaction of nanofibers with contaminants and polymers, in water treatment, metal and dye removal. (Balea et al, 2019).
- Monitoring of multiphasic catalytic reactions (modelling of kinetics, intensification of chemical processes, ...) (Esteban et al., 2014).

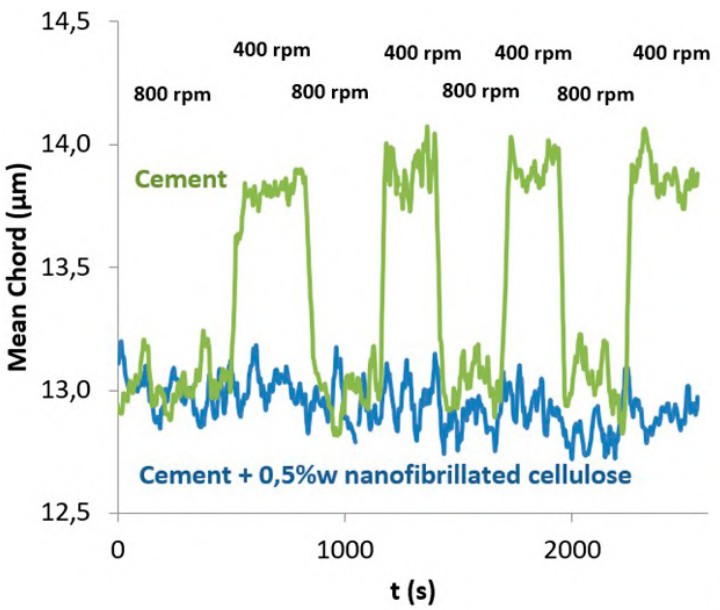


Figure 1. Interaction between cement and nanofibrillated cellulose. Monitoring the mean chord length during 800 rpm-400rpm stirring cycles reveals that the nanoparticle avoids cement aggregation at 400 rpm.

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Simultaneous determination of number-based particle size distribution and concentration by forward and sideward light scattering of single nano- and

microparticles: Validation, measurement range, coincidence and estimation of uncertainty

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Keywords | Particle size distribution, particle counts, particle concentration, uncertainty, SPLS-Technology, LUMiSpoc, Scattering simulation

Smart liquid products consist of various materials and featuring of a wide range of particle sizes. Especially, innovative technology demands high resolution of number-based size distribution of nano and submicron-particles together with the level of concentration. Spectroscopic ensemble methods as well as fractionating techniques do not have such capabilities. Small sub-populations and minimal particle size differences are not detected effectively. Furthermore, on a global scale, regulatory requirements for risk evaluation and nanomaterials classification warrant such data.

We describe a new innovative counting method, measuring simultaneously the intensity of forward (FSC) and sideways (SSC) light scattering of each single particle (SPLS-Technology®) for a wide dynamic size range (35nm to 8.000nm) with a size resolution of about 5nm. Intensities are classified in up to 3.6 million bins (real time). It is achieved by hydrodynamic focussing in conjunction with a high-end optical and an electronic measurement system and allows the analysis at a rate of 10.000 particles/second at particle concentrations between 10^3-109 particles/ml. Counting accuracy amounts to less than 3%. Browser and server-based software SepView® facilitate user control, project, SOP management and enables data acquisition as well as analysis of the recorded forward and sideways intensities.

In the second part we focus on metrological key figures. Precision (standard deviation) of nanogold particles (nominal 40nm, measured 37,24nm) amounts of 0.24%. Method was validated by comparison of experimentally obtained intensities of FSC and SSC and MIE simulation for 8 monodisperse reference PS-particles (143nm–3000nm). In addition, obtained particle size of certified reference particles corresponds well with the certified ones between 35nm to 1.600nm (R=0.9995). Measured concentration of diluted samples and expected values reveals an R-value of 0.9997. Coincidence was investigated based on DIN58932-4 and dilution experiments. At concentrations below 5x108 particles/mL the coincidence is generally smaller than 2%. Expanded uncertainty of sizing amounts of 4.03% and of particle concentration 7.59%, respectively.

ORAL COMMUNICATIONS

A machine learning application for coal tailings spectroscopic classification for soil amendment potential

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Keywords | Characterisation, spectroscopy, machine learning

A Machine Learning Application for Coal Tailings Spectroscopic Identification for Soil Amendment Potential
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Australia is currently the fifth largest coal producer in the world and is the second largest coal exporter. Coal production and processing produces a high amount of waste called tailings, usually in the form of unwanted fine organic matter or rock. The global estimated amount of mineral waste produced exceeds 100 billion tons per year, with the majority of these tailings either stored in Tailings Storage Facilities (TSFs) or dewatered at high cost and emplaced at the mine site. Historically, both methods have the potential to cause both safety and legacy issues at the site, if not managed properly.

An initial characterisation showed that high levels of nutrients and organic matter in the tailings would be well suited for use as a soil amendment, particularly to offset the arid conditions of many mine sites. With proper characterisation and management, the potential to convert this waste to a resource will reduce the impact on the environment and reduce the

high costs required for dewatering and storage. Partnering with the Global Centre for Environmental Remediation, and local regulators, the goal of this collaborative research project is to identify coal tailings suitable for this purpose, and the best way to characterise them.

This paper discusses the common analysis methods of coal waste, with a specific focus on infrared spectroscopic techniques. Near-IR and MID-IR spectroscopy techniques are coupled with supervised machine learning methods to identify tailings characteristics that are key to soil health and plant growth. This methodology is used to test several domestic tailings samples, to identify the best classification method for coal mine waste.

Dissipation of energy in granular materials

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(1) Lahti, (2) LUT

Keywords | Thermal discrete element method (TDEM), energy storage, packed bed, conduction

One of main characteristics of granular systems is the rate at which thermal energy will be dissipated. Considering a granular system with a high constant temperature at $t=0$ s, the reduction of energy of system should be an exponential function with a relaxation time depending on physical properties of particles including thermal conductivity, density, diameter, particle shapes, etc. Keeping this in mind, an interesting question is the physical mechanisms with which one can fasten or slow down the rate of energy dissipation in the system. Therefore, the aim of this paper is to shed some light on these mechanisms to understand the physical methods that could be utilized to control the energy release rate of a thermal granular source.

Evaluation of Deep Learning methods for particle characterisation from in-line imaging and chord length distribution measurements

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(1) University of Strathclyde, (2) Pfizer, (3) Takeda, (4) Eli Lilly, (5) AstraZeneca, (6) UCB

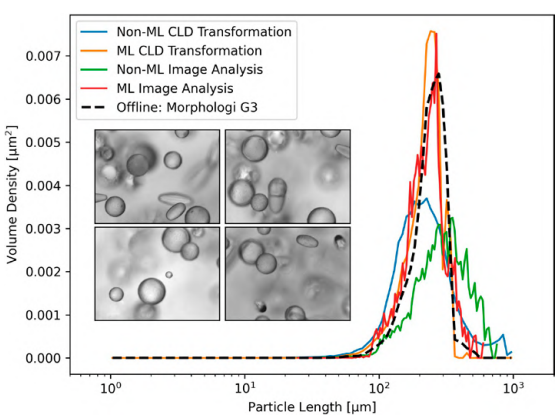


FIGURE 1. Polystyrene spheres and ellipsoids (sieved to 250-355um) Particle Size Distributions from in- and off-line measurements.

In-line Process Analytical Technologies (PAT) are useful for measurement of particle characteristics (e.g. particle size distribution, PSD) non-destructively and with high time-resolution (inaccessible with off-line techniques) which can be essential for process monitoring and accurate population balance modelling.

This work is concerned with assessing in-line imaging and CLD for PSD measurement. Imaging is limited by resolution (small particles are difficult to measure), subject focus, and field-of-view (particles touching image frame). CLD sensors (e.g. FBRM) can detect particles of smaller sizes but struggle with fast flow (undersized chords), large particles (chord splitting), and shiny particles (specular reflection).

In-line measurements were taken with the Mettler Toledo FBRM (CLD) and PVM (imaging) probes of Polystyrene Standard Spheres, a mixture of Polystyrene Spheres and Ellipsoids, and Lactose particles. In-line-derived PSDs from a range of crystal sizes and concentrations were compared with ground truth (off-line microscopy or manufacturer specifications) as shown in Figure 1. Image analyses: a machine learning (ML) method (Detectron 2 (Wu et al 2019)) and a traditional approach (ImagingApp (Cardona et al 2018)). CLD analyses: a statistical approach (Agimelen et al 2015), and a machine learning method. Sensors and analyses are evaluated using Root Mean Square Error (RMSE) and Integral Absolute Error (IAE) of the Cumulative Density Functions (CDFs).

Statistical CLD analysis is found to be sensitive to non-uniform particle shape distributions, and to artefacts introduced by the sensor. ML CLD analysis yields improved results, but is heavily reliant on the training data. The selection of image analysis approach has less of an effect on the resulting PSD than the characteristics of the image sensor itself where resolution and field-of-view limitations play a larger role.

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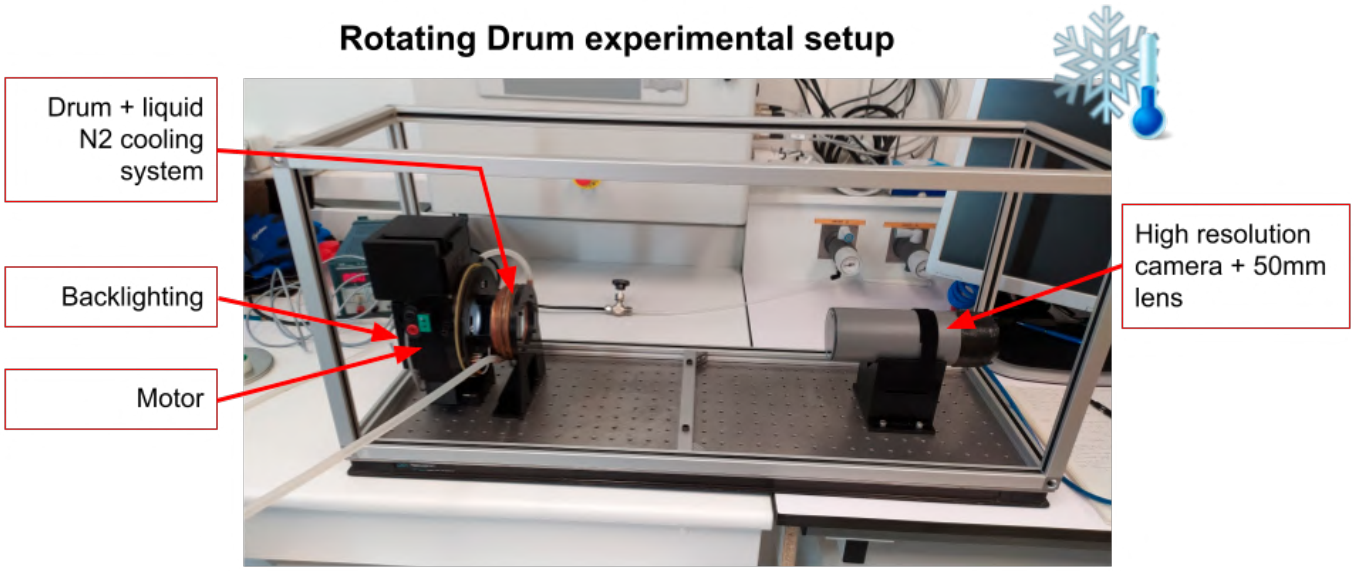
Cohesion of ice powders at very low temperatures : dynamical characterization of analogs of icy regoliths of the solar system

Jabaud, Benoit (1); Artoni, Riccardo (1); Tobie, Gabriel (2); Le Menn, Erwan (2); Richard, Patrick (1)
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Several planetary surfaces in the solar system are covered by granular ice, with properties unique to each body depending on their mode of formation and the surface environment. For example, on Enceladus, a moon of Saturn, jet activity and deposition of very fine ice grains (~10µm) in low surface temperature conditions (~60-80K) suggest the formation of stable powder-like deposits (Choukroun et al., 2020). Anticipating the properties of the icy surfaces is important for understanding the evolution of surface morphologies and minimizing the technical issues that future missions may face when landing and/or sampling.

We are therefore interested in the characterization of the mechanical behavior of micrometric ice powders, on a wide range of temperatures that span martian to Saturn moon conditions.

Ice powders were produced by spraying a fog of water droplets directly into a liquid nitrogen bath (~77K). Different process conditions were used, yielding different, well-controlled particle size distributions. For characterizing the evolution of powder cohesion with temperature, we developed a rotating drum, inspired by Lumay et al. (2012), but which we can operate on a wide range of low temperatures (~90-150K) via liquid nitrogen cooling. We show that in water ice powders, cohesion increases with the temperature. This is quite surprising but seems to be in agreement with some previous planetary science studies (Musiolik & Wurm, 2019). We discuss the possible origin of this peculiar behavior, the effect of particle size and present the other tools with which we intend to unlock the secrets of icy regoliths (tapped density, shear tests, discrete element simulations).



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Impact of particle size distribution on the extraction of key compounds in coffee

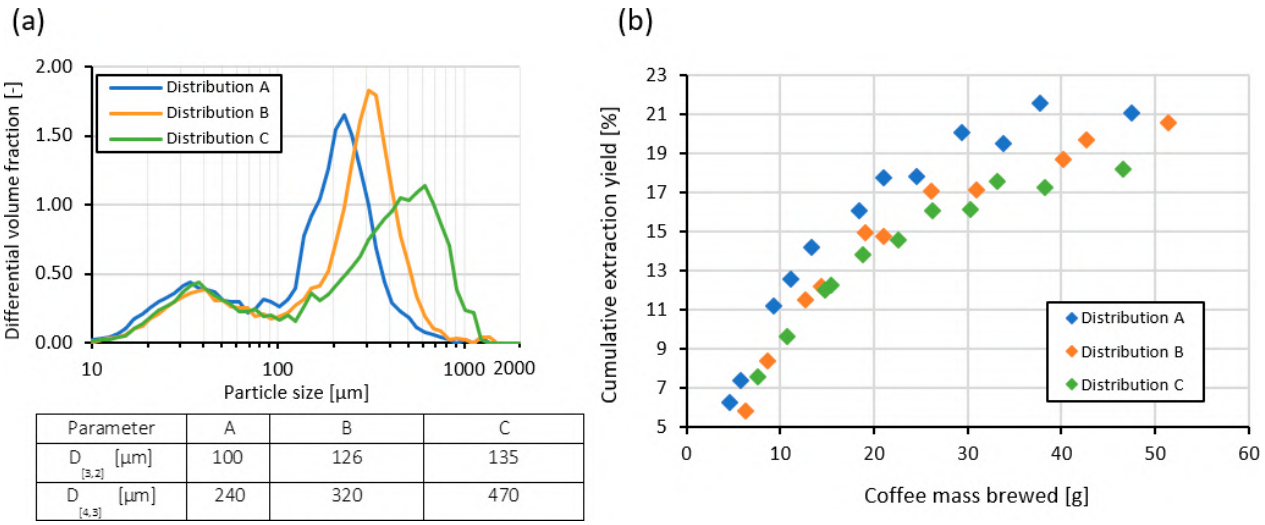
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Keywords | Coffee, espresso, particle size distribution, extraction, key marker compounds, pressure drop, volume flow

The extraction of coffee compounds is a convection-diffusion process wherein water flows through the intra- and inter- particulate pores. For that reason, the corresponding solid-liquid mass transfer is highly influenced by the particle size distribution. Different particle size ranges and size homogeneity of the coarse fraction (>100µm) has found to have a direct impact on extraction conditions at the steady state, mainly the flow rate and the pressure drop. The latter impacts the final extraction composition. In this work, three size distributions (see Figure 1a) were selected based on intrinsic criteria such as constant fine to coarse ratio and different size spread from 220 to 470 µm. The objective is to study the impact of these intrinsic characteristics on the extraction of key marker compounds: caffeine, trigonelline, and 5-CQA chlorogenic acid.

Extraction experiments were carried out in a modified espresso machine with all the other parameters kept constant to brew a double espresso (50ml) with a coffee dosage of 19-20 g. The extract was collected as portioned samples in 5ml intervals to study the extraction kinetics of compounds as a function of time and mass brewed. For each sample the total dissolved solids content, extraction yield and concentration of key marker compounds was analyzed.

Although distribution C is characterized by two-times larger particle size (D4,3) compared to distribution A, the same pressure drop (14 bar) and hence similar volume flow (2 ml/s) during extraction are achieved. However, by increasing the particle size, the obtained concentration of caffeine, trigonelline, and 5-CQA is on average 25 % lower for distribution C, with 5-CQA having the largest deviation. Difference in the kinetics was found at the beginning of the extraction, with distribution C having the lowest and distribution A the largest extraction rate. The overall trend can be seen in Figure 1b.



Parameter	A	B	C
$D_{[3,2]}$ [µm]	100	126	135
$D_{[4,3]}$ [µm]	240	320	470

FIGURE 1. (a) particle size distributions used in the current study (b) cumulative extraction yield in function of the extracted coffee mass for the three selected particle size distributions at a constant temperature of 88°C, volume flow rate 2.4±0.5 ml/s , pressure drop 14±1 bar.

Towards intuitive titania photocatalyst design: Using particle size distributions-based characterization for determining Hansen parameters

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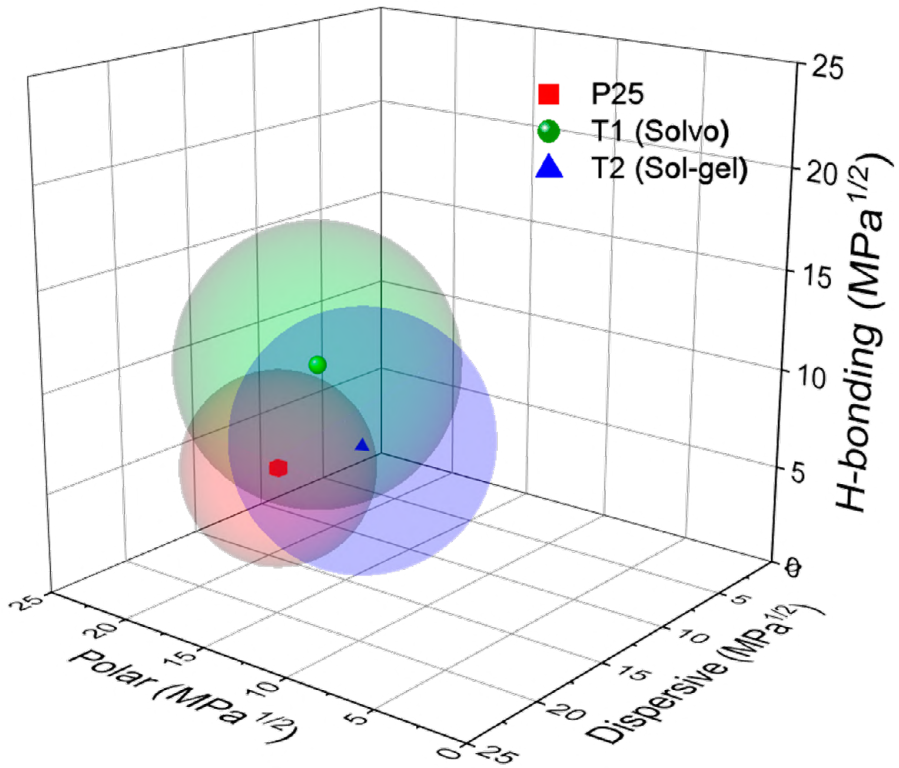
Keywords | Particle size analysis; Photocatalysis; Hansen parameters; characterization; surface behavior; surface properties; solvothermal synthesis; sol-gel synthesis

Every year, close to seven million people lose their lives due to air pollution [1]. As counteractive measures, titania photocatalysts have been used for degrading volatile organic compounds (VOCs). To tailor-make better photocatalysts, we need an understanding of the specific processes occurring at the particle surface during VOC adsorption. In this work, a characterization scheme based on particle size distributions (PSDs) is presented that can be used to calculate Hansen solubility parameters (HSPs) for comparison between photocatalysts made from different syntheses.

Ten probe liquids (PLs) were chosen for making dispersions to provide data for Hansen parameter calculation. Dispersions of titania P25, solvothermal titania [2], and Erbium-doped sol-gel titania [3] were prepared and underwent sonication followed by PSD determination by analytical centrifugation. A PL ranking based on product ratios (PR) developed by us was calculated

$$Product\ ratio\ = \frac{x_{10} \times x_{50} \times x_{90}}{\frac{\pi}{6} D^3}$$

where x10, x50, and x90 are the percentage fractions of the PSD and D is the primary particle size. Hence, PR is inversely proportional to good particle-liquid compatibility. The PRs can be used to sort PLs into 3 categories – clearly good PLs, uncertain PLs, and clearly poor PLs. For the uncertain PLs, combinatorics is employed earlier developed by Bapat et al. [4], the Hansen parameters are calculated and reported for all well-fitting Hansen spheres alongside outliers. The HSPs (Fig.1) show that the two titania samples prepared in the liquid phase are closer together in comparison to P25 synthesized in the gas phase. Thus, in line with our expectations, the synthesis process greatly affects the surface properties of the photocatalyst.



	P25	Solvo	Sol-gel
Disperse interactions δD (MPa $^{1/2}$)	17.7 - 19.0	13.1 - 16.8	13.0 - 16.5
Polar interactions δP (MPa $^{1/2}$)	15.6 - 17.0	14.4 - 18.8	11.4 - 17.6
Hydrogen bonding δH (MPa $^{1/2}$)	5.2 - 7.7	7.8 - 14.7	5.0 - 14.5
Sphere radius (MPa $^{1/2}$)	3.9 - 5.4	4.0-11.5	3.9-11.6
Total combinations possible	1024		
Combinations chosen for HSP	6 out of 8	14 out of 16	7 out of 16

Fig. 1: Hansen spheres (above) and HSPs (below) of the three titania materials alongside proper reporting of tried combinations and outliers

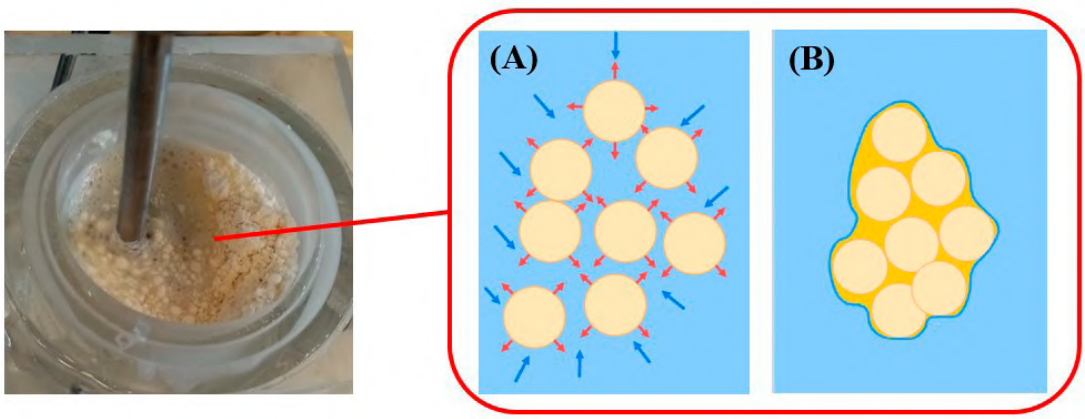
In conclusion, a new method for PL categorization for HSPs is presented. HSPs can assist in understanding the correlation of VOC adsorption in photocatalysts to particle surface is being explored further.

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Influence of plant material on plant-based milk powder characteristics

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Keywords | Food powders; plant-based milk; reconstitution process; lump formation; material characteristics



Food products in powder form are widely produced industrially due to advantageous properties including better physical and chemical stability, easy weighing, efficient transport and smaller volume of storage (Cuq et al., 2011). Milk powder in particular has numerous applications in various products, but a growing number of consumers are tending towards plant- based alternatives. The plant-based milk (PBMA) powder investigated in the project, consisting of maltodextrin, plant protein, vegetable oil and soluble fiber source, exhibits to lump formation during reconstitution. This lump formation is most likely correlated with the bulk structure as well as the viscosifying and swelling properties of the plant ingredients.

Figure 1: Lump formation during reconstitution process; (A) particles start to swell (red arrows) due to water contact (blue arrows); (B) formation of a viscous outer layer leading to the aggregation of swelling particles

For more efficient wetting behavior and facilitated water imbibition, the fine food powders are often agglomerated. To design targeted particle structure, PBMA powder is produced by drying homogenized emulsions using a spray-drier and agglomeration is performed by fluidized bed processing. Agglomerates are generated by fluidizing the solid plant-based milk powder with an upward heated air flow and spraying water forms liquid bridges when the particles collide before drying. The solvent evaporates as the drying occurs and a solid bridge binds the particle together.

In order to obtain an optimized structure of PBMA powder for subsequent applications, the influence of material properties on the formation, structure and qualities of the agglomerated powder has to be investigated. Therefore, the material characteristics regarding particle size distribution, glass transition temperature, wettability etc. are examined in dependency of the water content and temperature at different formulations due to the complex powder composition consisting of partially amorphous components. The results show that the plant proteins and fibers have a predominant influence on the process and product properties. A simulative approach was also chosen to gain further knowledge of the involved micro- mechanisms and the influence of the components on the agglomeration process.

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Selective liquid-liquid extraction of fine particles

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Keywords | PP 2045, adsorption of hydrolyzed metal cations, adsorption of surfactants, selective hydrophobization

Separation of fine particles is still a challenge. The known separation principles are based on volume- or area-related forces, which lose their effectiveness as well as selectivity during separation of small particles [Sandmann, 2020]. For this reason, an alternative to the known separation principles are investigated.

The PP 2045 deals with the multidimensional separation of fine particles smaller than 10 µm. The aim of subproject A3 is the investigation of material- and size-specific separation of particles smaller than 1 µm, which will be realized via phase transfer. Since the materials are hydrophilic, they have to be hydrophobized for the phase transfer step. The chosen materials are all negatively charged over a wide pH range. Ionic surfactants cannot be used that easily, because they will not adsorb on the particle surface selectively. This means that no selective hydrophobization and thus no separation of the mixture is possible. Therefore, the selective adsorption of surfactants is investigated, which is shown in figure 1.

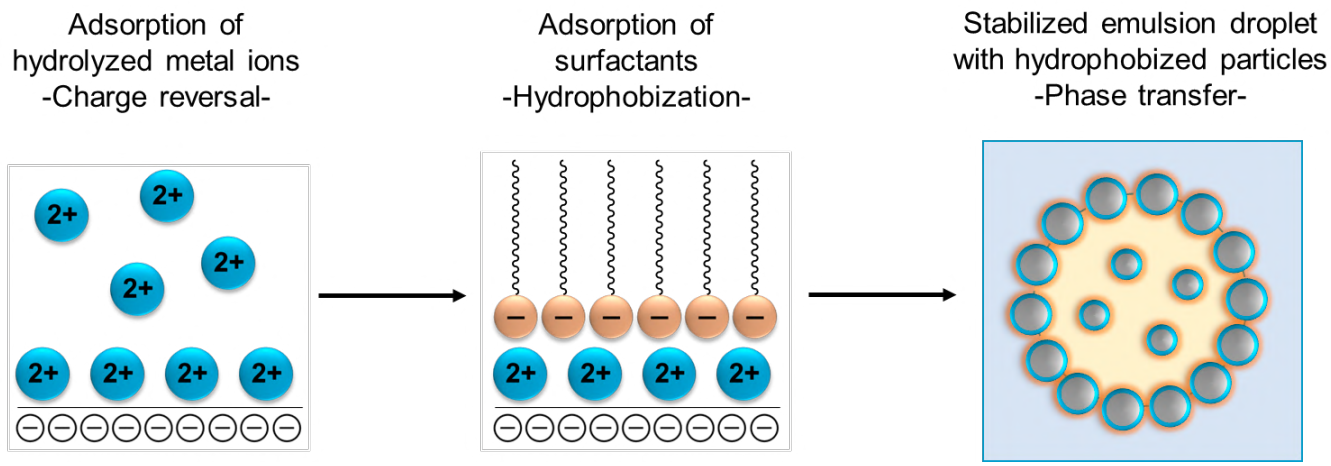


Figure 1: Short overview of the individual steps: Adsorption of hydrolyzed metal ions on the negatively charged surface (left) [similar to de Vos, 2019], adsorption of surfactants (middle) and the stabilized emulsion droplet with hydrophobized particles (right).

The selective adsorption of surfactants can be achieved with the help of hydrolyzed metal cations, which act as a bridge between the particle and the surfactant. The decision regarding which hydrolyzed metal ions are suitable is determined by means of ChemEQL [Müller,1996]. The necessary charge reversal of the particles, which is achieved with hydrolyzed metal cations, can be seen in the change of sign of the zeta potential. After adsorption of surfactant and therefore a selective hydrophobization, the transfer from the aqueous phase to the organic phase takes place. The optimum conditions will be determined for the phase transfer under variation of type and amount of hydrolyzed metal ions and surfactants.

Hydrogen Electrolyzer Recycling - Characterization of the layers and particle components of membrane electrode assemblies to assess the ultrafine particle separation ability

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Keywords | Characterization, recycling, particle separation, proton exchange membrane electrolyzer cell, high temperature electrolyzer cell, critical raw materials, wettability

In water electrolyzer cells, active materials contain various fine-grained critical raw materials such as platinum group metals or rare earth elements. Although the readiness level of water electrolysis technology is high and large scale of hydrogen production is targeted worldwide, there has been no significant research on recycling of end of life (EOL) water electrolyzer cells. For a functioning circular economy, recycling processes for these valuable materials have to be developed especially on the fine particle scale below 100 µm.

The in-depth characterization of water electrolyzer cells and their components allows to assess their liberation behavior and subsequent separability based on different particle properties, e.g. size, density, wettability, etc. Thus, this paper aims at identifying the material composition and properties of proton exchange membrane electrolyzer cell (PEMEL) and high temperature electrolyzer cell (HTEL) before processing. In PEMEL, critical raw materials such as iridium, ruthenium, and platinum are used, while nickel, lanthanum, and yttrium are

used in HTEL. Techniques such as automated mineralogical analysis (MLA), X-ray fluorescence spectroscopy and microscopy (XRF) and laser diffraction are used to identify the possibility of liberation. To ensure a high recovery rate of critical raw materials, the surface properties of individual component have been studied. Additional contact angle studies by means of pressed bubble and the particle attachment on single air bubbles revealed the wettability of membrane electrode assemblies and significant differences between the components. Furthermore, pH and salinity show to influence the wetting behaviour of the components. These findings provide the design of the separation study for EOL electrolyzer cells.

Fine particle separation in Lithium Ion Battery Black Mass Recycling - the challenge of particle wettability characterization

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Keywords | Froth Flotation, lithium-ion batteries, wettability, recycling, particle characterization, inverse gas chromatography

One of the main tasks to ensure the secure supply of critical raw materials is the efficient recovery and recycling of secondary resources. Lithium-ion batteries (LIB) are the key technology nowadays and in the future to enable the human energy revolution. Therefore, the recycling of spent LIBs is of great interest. A major challenge in spent LIBs recycling, is the beneficiation of fine powder resulting from the battery crushing, so-called “black mass”, which contains the valuable lithium metal oxides (from the cathode) and the critical graphite (from the anode). One promising way to separate the graphitic material from the lithium metal oxides is by means of froth flotation, which separates the particles according to their differences in wettability. In order to improve the separation process, one must first have a proper understanding of the particle properties. In particular, the lithium metal oxides are commonly declared as hydrophilic and should therefore be easily separated from the hydrophobic graphite. Nevertheless, many studies report on their recovery into the froth product, along with the graphite, thus lowering its grade, which is most probably due to the residual hydrophobic binder that causes a change in their wettability.

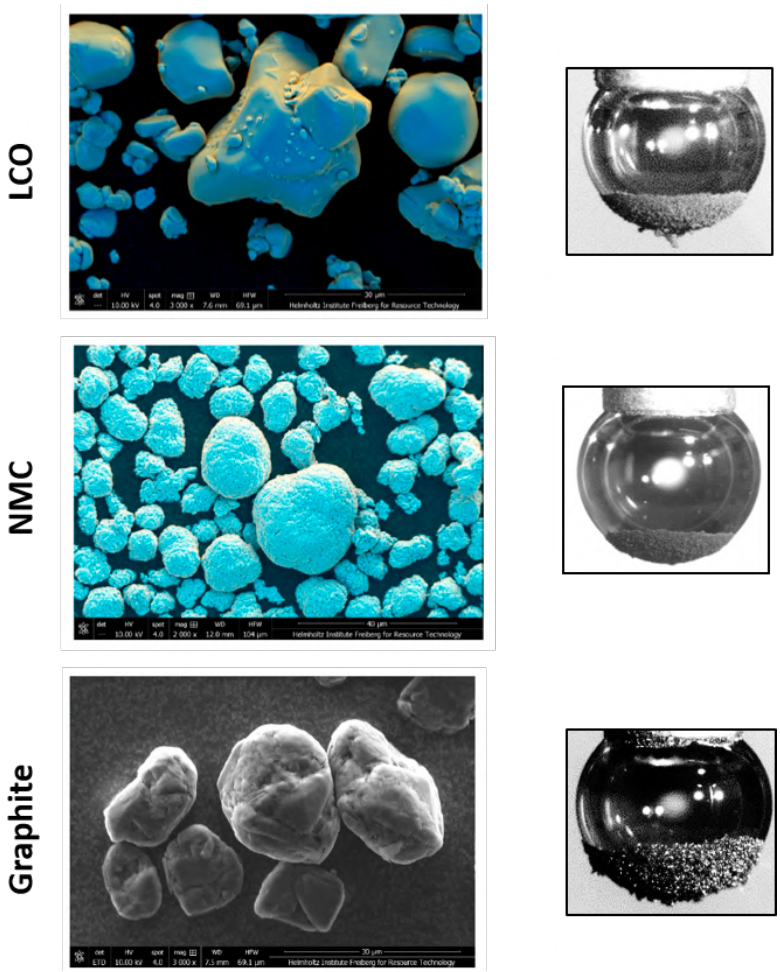


FIGURE 1: shows false colored SEM images (left) of different cathode active particles (LCO and NMC) as well as the anode graphite and their attachment to bubbles without reagents.

In this study, different battery materials, including pristine lithium metal oxides and graphite, as well as real spent materials from LIBs wastes were used. The micro particles were analysed for their wettability and (de)wetting ability using optical contour analysis, inverse gas chromatography, particle attachment to bubbles, analytical particle solvent extraction as well as the famous Washburn method. These results are set into context with flotation tests and shed light on the particle wettability and its effect on the flotation separation efficiency, as well as the difficulties that arise when it comes to the recycling of spent LIBs.

Experimental and numerical investigations of the mechanical properties of particles for cold spraying of wall surfaces

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Keywords | Cold spray Nanoindentation Particle compression Mechanical properties

The change of the surface morphology of components plays an important role in special technical applications. Therefore, the creation of a defined surface morphology is important for the optimization of pocesses in the field of mechanical and process engineering.

A suitable method for the change of the surface properties is cold spraying [1]. With this method, particles are accelarated to velocities up to 1000 m/s and impact on a substrate. The high kinetic energy leads to plastic deformation and mechanical interlocking during the impact [2]. As a result a strong bond at the interface between the particle and the solid substrate is formed. To obtain high particle velocities

a pressurized gas is preheated and then expanded through a Laval nozzle. The use of inert process gases and the low process temperatures makes the cold spray technique suitable for a big variety of powders.

For the optimizaton of the process the mechanical properties of particles and component materials at process temperatures are needed to understand the mechanisms during the impact. Therefore, nanoindentation experiments are conducted on steel samples (single particle and substrate material) to analyse the mechanical behaviour (Fig 1a,b). With the used device it is possible to get important mechanical characteristics like the Young modulus or hardness of a sample at different temperatures and strain rates.

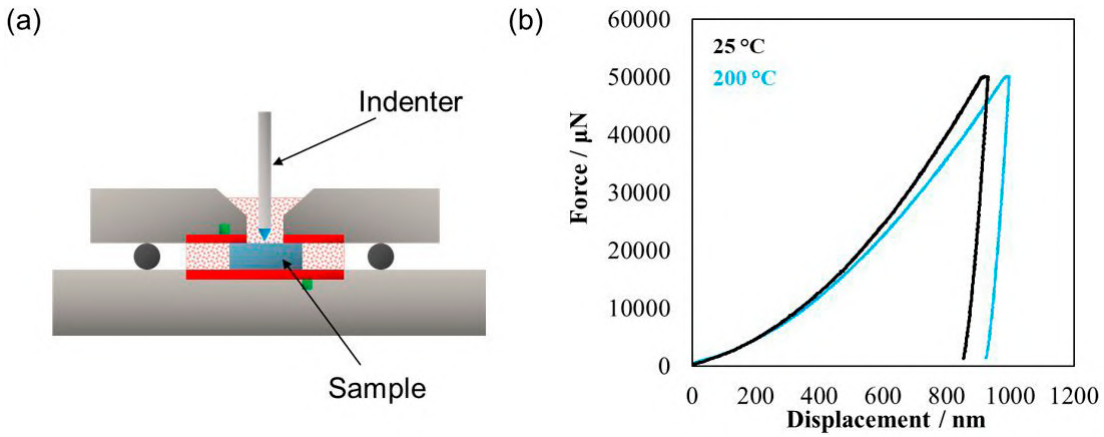


Figure 1: Scheme of the (a) heating stage setup for nanoindentation and the obtained force-displacement curve (b) of a stainless steal surface at 25 and 200 °C.

The obtained properties are used as parameters in the simulation of the particle impact with the finite element method (FEM). The simulations show a good agreement with the experiment, which enables a further optimization route for cold sprayed components (Fig. 2).

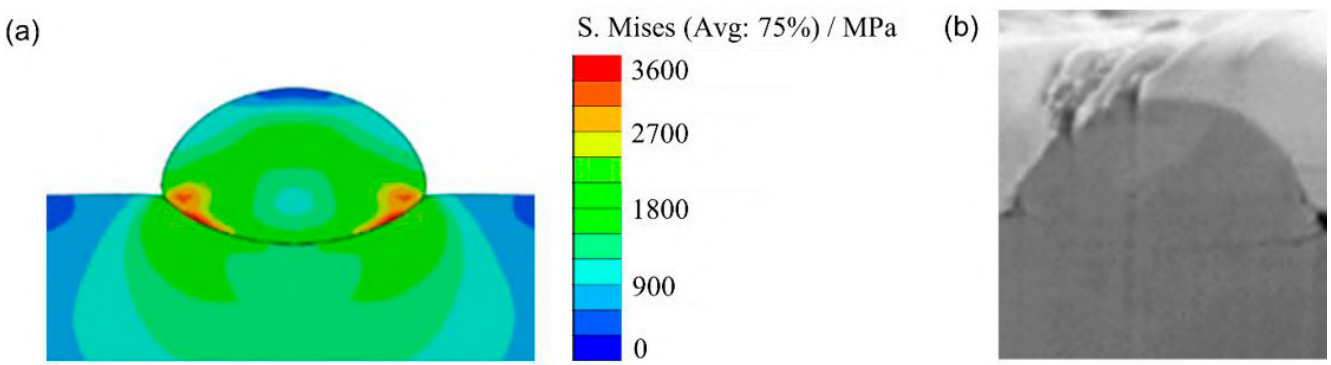


Figure 2: Comparison of the particle deformation behaviour with (a) FEM simulation and (b) FIB cut.

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Multidimensional characterization of patchy particles

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Keywords | Analytical ultracentrifugation, plasmon resonance, single particle scattering, scanning electron microscopy

Patchy particles are a novel class of disperse system in which the component particles are designed to have surface regions with completely

different physical or chemical properties. Such an arrangement opens up exciting opportunities for particle- based materials and devices in a range of application areas. To achieve this, however, novel fabrication processes need to be established and optimized. A major challenge here concerns the characterization of the patchy morphology. We urgently require new techniques, particularly those offering the possibility to rapidly and quantitatively determine multi-dimensional morphological parameters and their dispersity.

The present contribution will contrast the opportunities and challenges of different techniques for the case of patchy particles comprising a spherical silicon dioxide core with a diameter between a couple of hundred nanometers and a few microns and one or more thin (< 20 nm) silver patches. These are produced in a continuous flow process in which the conformal growth of silver is achieved via an electroless plating reaction. This approach results in both uncoated core particles and a distribution of patch thicknesses and coverages. While scanning electron microscopy can estimate, to a certain extent, these parameters, this is both arduous and inaccurate and is complicated by the fact that patches can assume all possible orientations in a sample. Due to the obscuration of unfavourably oriented patches, even the patch yield, defined as the fraction of all core particles having a patch, cannot be accurately determined by microscopy. However, we will show briefly how multiwavelength analytical ultracentrifugation and, in more detail, single particle extinction and scattering can determine this parameter. In the former case, the measured sedimentation coefficient is related to the patch to core mass ratio and details of the reaction feed and assumed stoichiometry applied to arrive at an estimate of the yield. In the latter case, the complex components of the forward scattered electric field vector measured for individual particles enable uncoated and coated particles to be easily identified. Moreover, we will show how aspects of patch morphology and its dispersity, extremely challenging to obtain by microscopy can be estimated by both new techniques.

A particle shape-based segmentation method to characterize spray dried materials by X-Ray microtomography

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Keywords | Particle structure, X-Ray microtomography, Image analysis, Porosity distribution

During spray drying, the liquid is atomized into small droplets, which are then dried in the spray drying chamber. Dependent on the drying conditions, particles with different shapes, porosities and sizes are produced. Detailed knowledge of structure is of interest as it determines, in combination with particle size distribution, the handling properties such as particle strength, reconstitution behaviour and bulk density. The structure of the generated particles does not only differ between batches but also within a single batch due to particles being exposed to different local drying conditions dependent on particle size and their pathway in the drying chamber. Thus, for accurate description of structure, distributed, or at least size-resolved, parameters are required.

Using X-Ray microtomography, the internal structure in 3D can be visualized. For a quantitative analysis, multiple particles have to be analysed, e.g. by measuring a particle bed. Particle segmentation by image analysis is then required to obtain information such as porosity, contact number and concavity for individual particles.

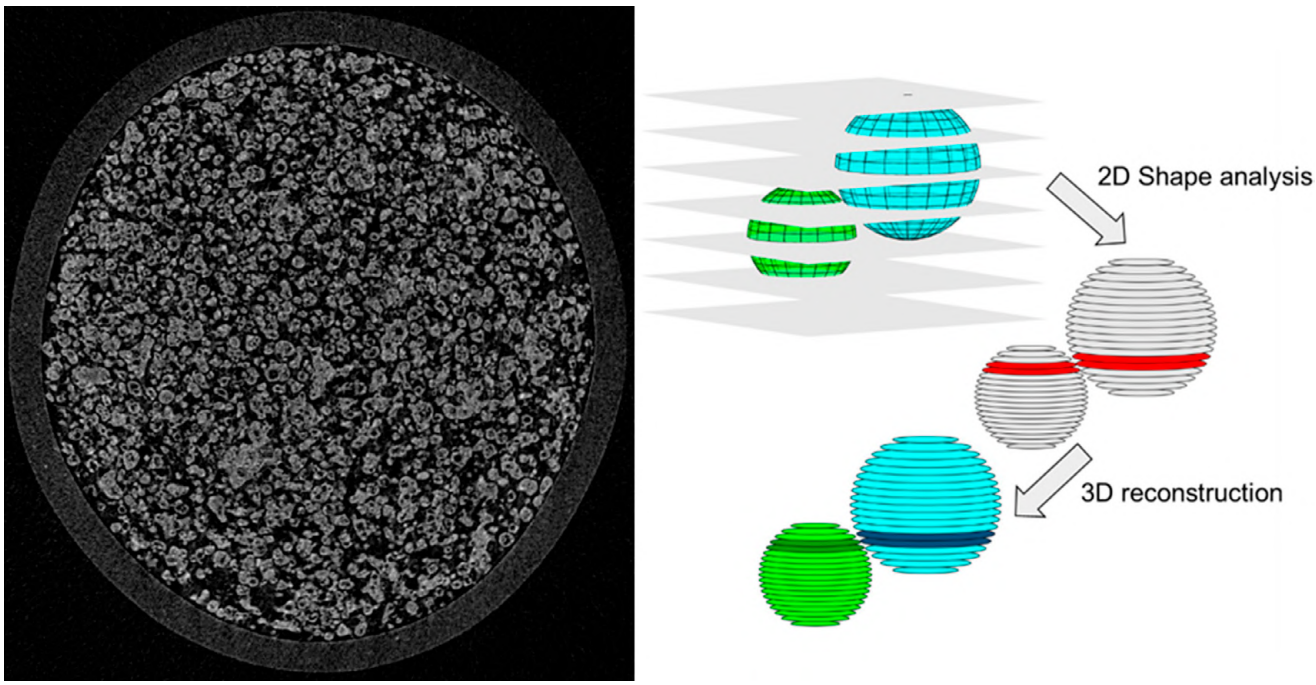


FIGURE 1: X-Ray microtomography image of particle bed of spray dried milk powder (left) and principle of proposed algorithm for segmentation (right)

For spray dried systems, however, the particle size distribution is broad. Thus, standard segmentation techniques from literature, which have a size-based criterion, tend to fail.

Therefore, in this study, we present a new segmentation technique for spray dried powders based on analysis of shape. Here, 2D connected particle layers are identified based on a-priori knowledge of particle shape. These particle layers are deleted and a 3D reconstruction of the particle is performed subsequently. For each of the particles, information such as porosity and contact points can be obtained and correlated to the particle size.

The algorithm was successfully validated and the suitability of different methods for sample preprocessing was evaluated.

The impact of various process (e.g. air temperature and moisture content) and product (e.g. initial concentration) parameters on the particle structure in spray drying was investigated.

The technique may be extended to other particle systems as long as a shape classifier can be specified, e.g. by machine learning techniques.

Morphological Study of Microagglomerates Obtained by Spherical Agglomeration Using X-ray Microtomography

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Keywords | Spherical agglomeration, X-ray microtomography, particle morphology

The spherical agglomeration technique is based on a three-phase system consisting of two liquids with a miscibility gap (suspension liquid and binding liquid) and a suspended solid phase which can either be homogenous or heterogenous. After addition of the binding liquid, which wets the particles better than the suspension liquid, the agglomeration starts.

The process itself is divided into three different phases. In the wetting phase the particles collide with binding liquid droplets. Due to the wetting properties, particles and binder droplets stick together and form the so-called microagglomerates. Due to capillary forces and enough binding liquid, these microagglomerates grow to the final agglomerate size in the fast-growth regime. When the agglomerates reach a certain size, the binding forces and the shear forces of the turbulence balance each other and no further growth of the agglomerate size takes place. In the past, these three different process phases are characterized over the time-dependent agglomerate size. Additionally,

Schreier & Bröckel (2021) showed that the process phases can also be characterized via torque measurements during the agglomeration process. It is shown that at the end of the wetting phase the torque decreases and then increases again (see Fig. 1).

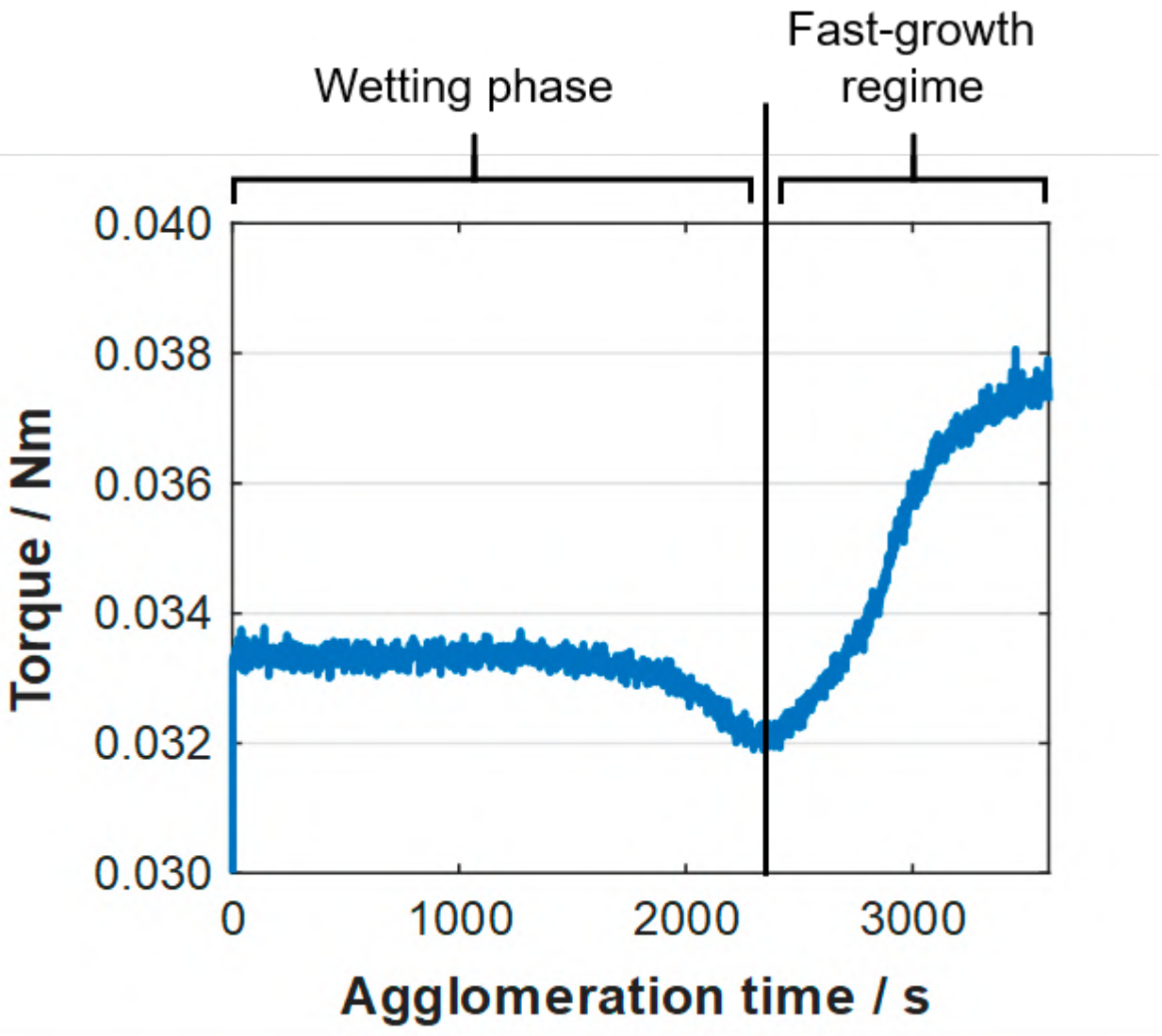


Figure 1:

Example of time dependent torque values during the spherical agglomeration Snabre & Mills (1996) showed that the shear stress is proportional to the fractal dimension and the packing density of flocs under constant shear rate. These two morphological parameters as well as parameters like volume of the microagglomerates can be obtained by X-ray microtomography. In order to minimize the impact of preparation on the microagglomerate morphology, the sample is frozen and scanned in the X-ray microtomography.

Specific objective of this presentation is the adapted sample preparation as well as the image analysis routine. Furthermore, the first results of the morphological parameters depending on the agglomeration time will be discussed.

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Methods from machine learning for the structural analysis of Li-ion electrode particles

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Keywords | Super-resolution, convolutional neural network, stochastic geometry, digital twin, Li-ion battery, numerical charge simulation, continuum damage

Microscopy techniques like scanning electron microscopy (SEM) or X-ray computed tomography (CT) can provide detailed image data of electrode particle microstructures. Followed by a quantitative structural characterization such data allows for the investigation of structure-function relationships, i.e., the influence of an electrode particle's microstructure on its properties like its mechanical or electrochemical behaviour. However, for the structural characterization by means of image data nontrivial processing tasks are often necessary. In this talk several applications of machine learning methods and stochastic geometry are shown for the structural characterization of particles in lithium-ion battery electrodes imaged by SEM, CT and focused ion beam (FIB) - electron backscatter diffraction (EBSD). In the first application, generative adversarial networks are deployed to perform super-resolution on SEM-images of cycled cathode particles such that fine features like cracks within particles can be more reliably characterized to investigate the state of degradation. The second application shows how convolutional neural networks can be used to achieve a grain-wise segmentation of FIB-EBSD data of polycrystalline electrode particles (Furat et al., 2021b). The third application deals with the structural multi-scale modeling of cathode particles to overcome limitations of different imaging techniques (Furat et al., 2021a). Therefore, a stochastic geometry model is calibrated using CT data depicting the outer shell of cathode particles and FIB-EBSD data of the grain architecture of a cathode particle, see Figure 1. Then, the model can be used to perform structural scenario analyses, i.e., to generate arbitrarily many digital twins with statistically similar shape and grain architecture as the particles observed in the image data. These digital twins are used as input for numerical charge simulations to investigate their degradation behaviour (Allen et al., 2021).

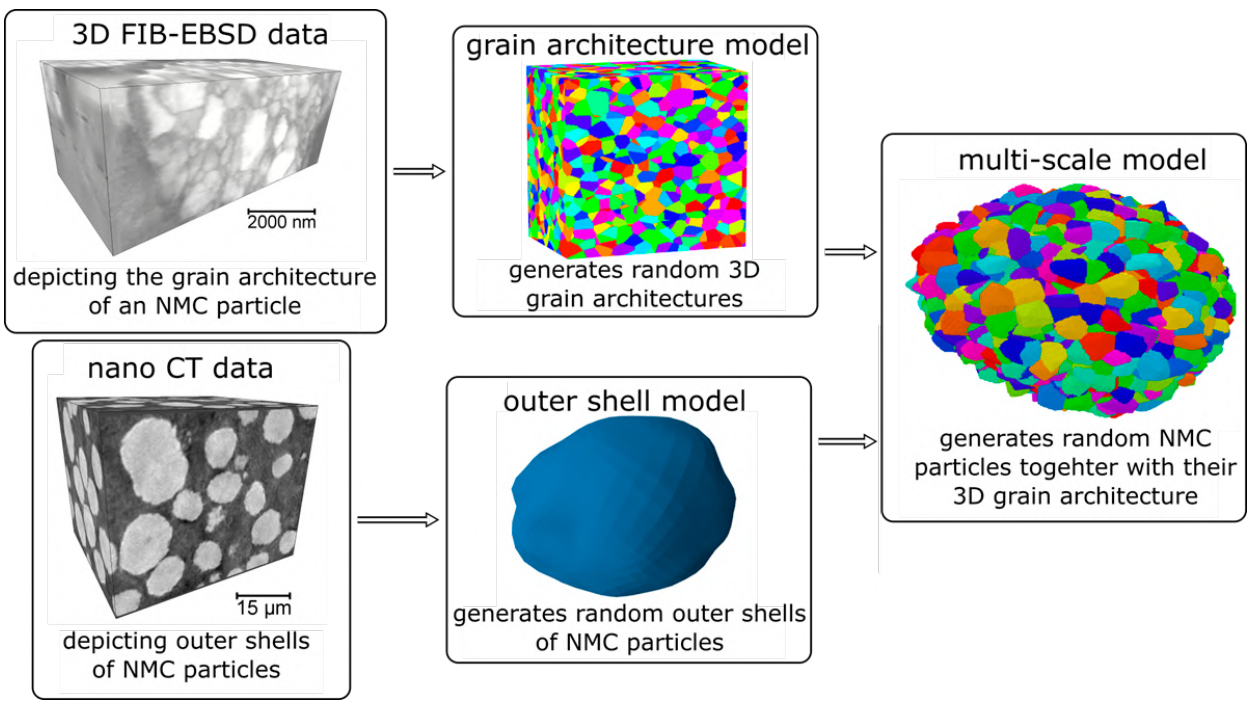


Figure 1: Modeling approach: FIB-EBSD data is used to calibrate a grain architecture model (top). From nano-CT data an outer shell model is fitted (bottom). By combining both models, we can generate representative particle architectures (right) (Furat et al., 2021b).

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Modelling morphology and fibrillation of lignocellulosic micro/nanofibers during

mechanical isolation using artificial neural networks

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Keywords | ANN; nanocellulose; aspect ratio; morphology;

Despite the huge efforts that were made in the last 20 years for development of reliable and robust monitoring techniques for lignocellulosic micro/nanofiber (LCMNF) production, most LCMNF properties cannot be measured on-line. Besides, there is the difficult task of relating the raw material properties and final end-use properties (mechanical performance, recyclability, barrier properties, etc.). These challenges can be faced efficiently with the help of machine learning modeling techniques, which make use of computational resources and statistical tools for efficient handling of massive datasets. Among these modeling techniques, artificial neural networks (ANN) have been long recognized as one of the most powerful.

The present work proposes the use of neural networks to model the relationship among a set a of easy-to-measure operation variables and initial fiber characteristics to the morphological features of mechanically obtained LCMNFs. The calibration dataset was constructed using data from Pine pulps described in an earlier work of this group (Serra-Parareda et al., 2021). The selection of input variables of the fibrillation process was carried out thoroughly, combining with the interpretation of the behavior of the actual process conditions, allowing to identify that the aspect ratio could be properly described by four main inputs: cellulose contents, applied energy, the mean fiber length and the mean diameter of the pre-treated pulps. The ANN based on these 4 inputs and on 4 neurons in the hidden layer showed excellent correlation with the dataset used in the calibration (R=0.9993), validation (R=0.9989) and testing (R=0.9984) stages. As a further proof of validity of this ANN model, additional tests were performed for different species, including samples of spruce BTMP and also of non-wood species. The neural network model provided excellent predictions of aspect ratio for hemp samples, and adequate predictions for spruce BTMP and (to a greater extent) for sisal fibers, which encourages the use of the neural network approach to the modeling of the fibrillation process during the mechanical production of micro-nanofiber materials from different sources.

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Formation and stability of particle-bubble heterocoagulates containing poorly wetted alumina particles investigated by Dynamic Image Analysis and Atomic Force Microscopy

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Keywords | particle-bubble interaction, heterocoagulation, Dynamic Image Analysis, Atomic Force Microscopy

The formation and stability of particle-bubble heterocoagulates is of major importance for industrial processes like mineral floatation or the cleaning of metal melts. Results on the heterocoagulation behavior of poorly wetted spherical alumina particles with diameters between 1 µm and 20 µm in electrolyte solutions, on both the meso- and the micro-scale will be presented. The particle wettability is altered by silanization which leads to a poorly wetted particle system with a tendency towards agglomeration and a high affinity for the air-water interface.

On the meso-scale heterocoagulation experiments were performed in a baffled stirred tank which is aerated with a two- component jet nozzle, producing spherical gas bubbles in the size range of 50 µm to 350 µm. The characterization is done by dynamic image analysis with images obtained from an inline probe. The measurements exhibit a number of different types of heterocoagulates with varying degrees of surface coverage and deviations of the spherical bubble shape. Structural parameters are related to the initial state of particle dispersion as well as the electrolyte concentrations, and estimates of the effective heterocoagulate density are provided.

Colloidal Probe Atomic Force Microscopy experiments on sessile bubbles and particle laden bubbles with varying degrees of particle load are performed to gain a fundamental understanding of the micro processes which govern the formation and stability of the heterocoagulates observed in the meso-scale experiments. The particle laden bubbles are generated by a solvent exchange procedure and full force distance

curves are measured at varying approach velocities. At low coverage the interaction is considered to be of particle–bubble type. At high surface coverages the interaction may show a solid-solid like behavior. The results are analyzed with respect to the adhesion force, dynamic contact angles and the equilibrium position of the colloidal probe in the interface.

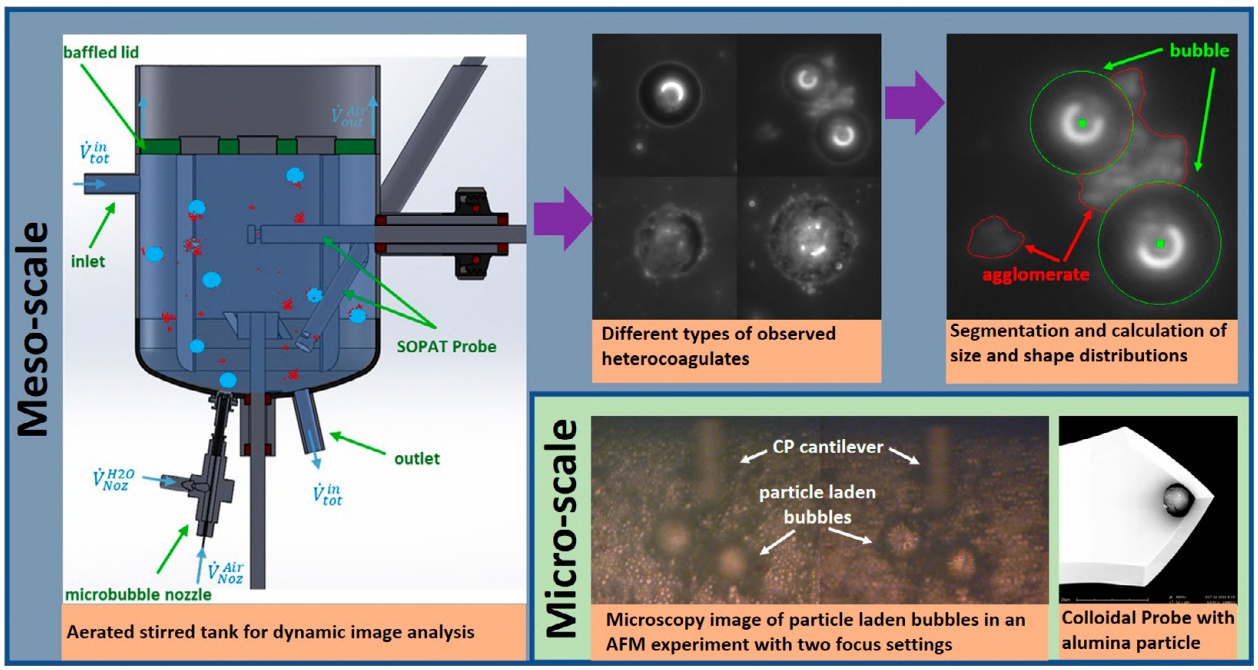


Fig.1: Concept of heterocoagulation experiments on the micro- and meso-scale

Tensor-based, contact-model-agnostic approach to reconstruction of granular bulk's contact forces from its macro behaviour and contact network fabric

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Keywords | Contact network fabric, contact forces, stress tensors, strain tensors

Experimental investigation of the connection between granular material's contact network fabric and its evolution and the characteristics of macro behaviour of granular bulks has so far been restricted due to limitations when retrieving contact network and particles' kinematics. Hence simulations are employed to obtain whits of the required information. Numerical methods used for the purpose of granular material research (e.g. Discrete Element Method) are based on an a priori made assumption about the particles' contact properties and require knowledge about particles' material properties. It is followed by calibration of numerically produced values according to the experimentally obtainable data. A straight forward way to better explain and reconstruct contact forces from contact network fabric and the characteristics of macro behaviour of granular bulk, is still lacking.

Here we employ a novel, tensor-based approach to calculating contact forces between particles. This approach, unlike common DEM ones, requires no prior knowledge about features of particles' material and therefore reproduces contact forces without any assumptions regarding contact properties - it is a contact-model-agnostic approach. Moreover, it considers a Voronoi tessellated continuum specimen and uses a connection between its contact network fabric, stress and strain tensors; it links them on different levels of description (bulk, particle, interaction - contact), and dissects a priori known information about the specimen's bulk pressure (i.e., the trace of the average stress tensor). The dissection outcome represents per particle and per interaction stress tensors, which are later combined with a complementary area vector. This combination enables reconstruction of contact force network within the specimen. The novel approach was applied to experimental data taken from x-ray tomography, obtained in 3SR Laboratory, Grenoble, France.

Adhesion forces between particles in the gas-phase

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Keywords | Nanoparticles, adhesion, van der waals, capillary, forces

Adhesion forces between particles in the gas-phase

The behavior of particles in processes such as fluidization, filtering (e. g. in FFP2 masks), and separation (e.g. recycling) is strongly driven by their large surface-to-volume ratio. This leads to the fact that particle behavior in the gas phase is mainly determined by adhesion forces due to the van der Waals interactions and the capillary forces. Till today we lack in a basic understanding of these forces for particulate systems on a fundamental as well as on a practical level. This missing knowledge affects all process dealing with particles. Therefore, I will present our research about measuring and simulating adhesion forces from a fundamental level, starting at a simple particle-particle system. Here I point out that water is the major component determining the adhesion and has to be understood on a molecular level, while the state-of-the-art continuum models rather arguable to make reliable predictions. Especially the usage of the Hamaker constant as a very simple way to describe particle-particle interactions will be discussed critically.

Finally, I will highlight how we can use this knowledge to go to a complex system containing thousands of particles and predict their behavior correctly which is on major importance for industrial systems.

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Triboelectric charging of powders

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Keywords | Triboelectric charging, Additive Manufacturing, process

Triboelectric charging of powders is an important topic in granular science. Indeed, powders are widely used in industrial applications and processes, which requires good powder flowability. However, the grains constituting the powder has often the ability to build up charge during powder flow, due to the triboelectric effect. During a collision or friction, a exchange of charge can occur between a grain and another one or with a surface. This phenomenon changes the powder properties like the flowability as the interactions between particles increase due to the Coulomb force. Then, powder charging represents one of the major problems in processes handling powders.

While the powder charging is known since a long time, it remains difficult to predict how a powder builds up charges. Indeed, the mechanism controlling the triboelectric effect remains poorly understood. For this reason, it is important to develop tools that allow to measure the ability of a powder to charge.

We present here results on a metal powder during conveying through the different parts of a Laser Metal Deposition (LMD) machine. The electrostatic charging has been investigated with the GranuCharge. The GranuCharge consists of a steel bend pipe, which modelize most of possible charging way, i.e. friction between grains or with the tube and collisions. The powder flows in the tube and fall in a Faraday cup which measures the total electric charge of the powder.

Powder samples are taken at different locations of the powder conveying stage to assess their respective influence on the charge build-up. We highlight the influence of the powder distributor speed on the charge build-up as illustrated in Figure 1.

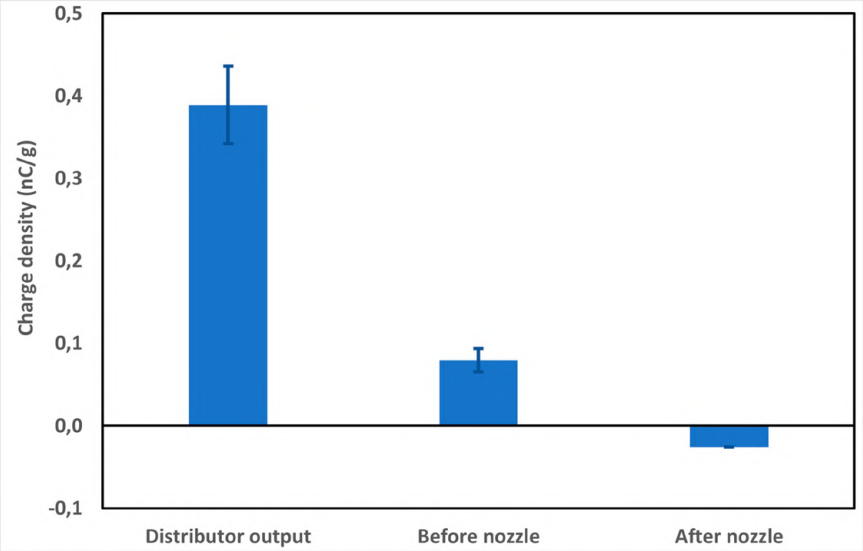


FIGURE 1- Charge density measured at different stages of the process.

A Study on the Charge Neutralising Effect of Aluminium Stearate in Triboelectrification

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Keywords | aluminium stearate; triboelectrification; electrostatics; charge neutraliser

Aluminium stearate is a common metallic soap that is widely used as a thickener in paint, a water repellent and a lubricant in various industries. Depending on its synthesis process, aluminium stearate can exhibit three different crystalline forms of mono-, di- and tri-stearate, the most common one being aluminium di-stearate. Apart from having superior lubricating properties, it is also an excellent charge neutralising agent for preventing wall fouling in a fluidised bed reactor caused by electrostatic charge build-up. However, the underlying charge mitigating mechanism of this metallic soap is still not very well understood and hence it is a topic which requires further investigation.

The long stearic acid chains present are structured very differently for the three crystalline forms of aluminium stearate. The focus of this work is to study the role of structural difference of these surface stearic acid chains in electrostatic charge reduction. Assemblages of glass beads are made hydrophilic and hydrophobic through hydroxylation and silanisation. These glass beads are then dry coated with the three different stearates, separately and evenly, using the ProCepT, a high shear mixer. A novel method based on aerodynamic dispersion technique is then used to characterise the charge level of these blended mixtures of aluminium stearate and glass beads, of which the results will be presented and reported here.

Triboelectric charge build-up mechanisms in granular particle mixtures

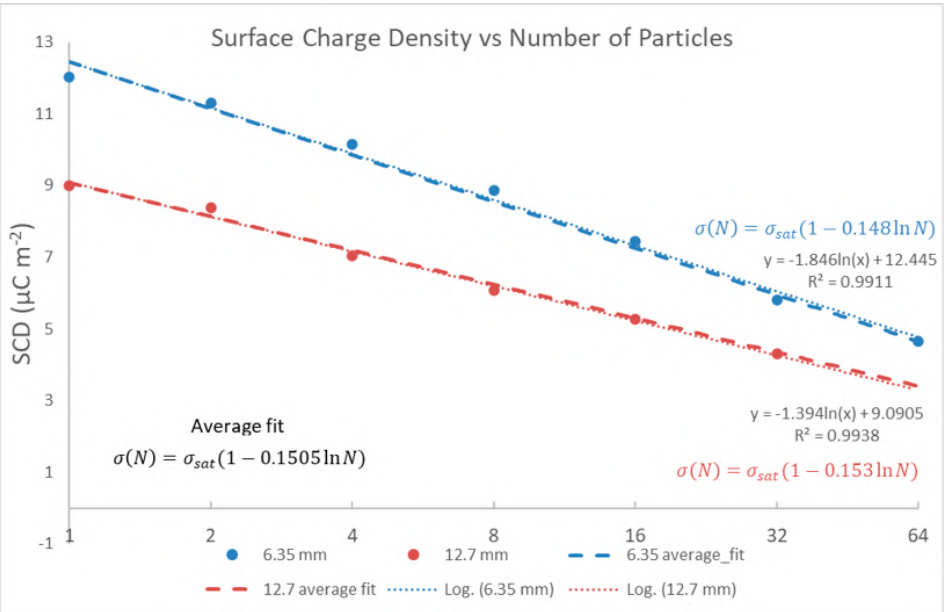
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Keywords | Triboelectric Charging

The build-up of triboelectric charge can be utilized for numerous technologies, while creating problems in a range of environments. It is desirable to reduce charge build-up in many industrial processes, from dealing with pharmaceutical powders to mitigating dust adhesion to surfaces. On the other hand, the control of surface charge on particles of different material can be exploited to enable methods such as electrostatic mineral separation. This widespread manifestation of triboelectric charge means that an understanding of the charging process, and accurate prediction of charge attainable by materials is important.

The objective of this work is to develop a new method of continuously measuring triboelectric charge build-up on particles in real time as charging occurs. This new method is implemented in order to investigate the effect of packing density and external electric fields on the final saturation charge that bulk collections of polymer particles can gain. We look at PTFE and Nylon spheres from 3-12 mm diameter, charging in batches of 1 – 2000 particles against stainless steel. The aim is to build upon results for single particle charging under similar conditions in which a 1/r relationship was found (Cruise et al 2022).

It has been found that increased packing density of particles can lead to reduced saturation charge per particle under triboelectric charging (Kolehmainen et al 2017). From theoretical develoment and experimental results, it was found that the work function of a conducting material can be altered by induced or applied voltages. This is applied experimentally to control the charge acquired by particles. This novel approach allows the saturation charge on particles to be carefully increased or reduced. This technique gives greater control of the charge behaviour of many materials in a wide range of industrial applications.



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Electrodynamic Dust Shield with Vibration Assistance for Cleaning Lunar Regolith Particles

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Keywords | lunar regolith, electrostatic traveling wave, space application, vibration assistance

The Apollo astronauts were suffered from lunar regolith which is particulate material covering the Moon surface. Owing to the vacuum and low gravity environment of the Moon, regolith particles were easily lifted by extravehicular activities and ended up with depositing on spacesuits and exploration equipment, causing some problems, such as obscuration of astronauts' vision and mechanical malfunctions (Wagner 2008). Therefore, regolith mitigation technologies have been required, and electrostatic systems have been attracting researchers and engineers.

One technique is an electrodynamic dust shield (EDS) which utilizes an electrostatic traveling wave generated on a cleaning target in which parallel electrodes are embedded separately from one another (Figure 1). It has a glass lamination with transparent ITO electrodes and can be placed on optical elements and solar panels without disturbing their functions. When AC high voltages with shifted phases are applied

to the electrodes, the resultant electrostatic field removes particles. In previous studies, EDS can remove most of regolith simulant particles, while some of smaller particles with larger adhesion remain on the target (Kawamoto and Hashime 2018).

In this study, we employ a vibration to reduce adhesion of particles to improve the EDS performance. Our setup consists of EDS and a stand vibrating in x- and z-axis (Figure 1). While smaller particles cannot be removed enough without vibration (Figure 2), the assistance improves the cleaning performance (Figure 3). Moreover, a vibration in shear direction (x-axis) is more effective to the cleaning than that in vertical direction (z-axis) since the shear vibration reduces the adhesion force. In addition, larger particles suppressed due to gravity can be cleaned more with vibration assistance. Since EDS requires no mechanical drive, liquid, and gas, it has advantages for space application. This result indicates that EDS with vibration assistance would be promising for future lunar explorations.

References

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Wagner, S., 2008. An Assessment of Dust Effects on Planetary Surface Systems to Support Exploration Requirements. NASA/ TM–2008–213722.

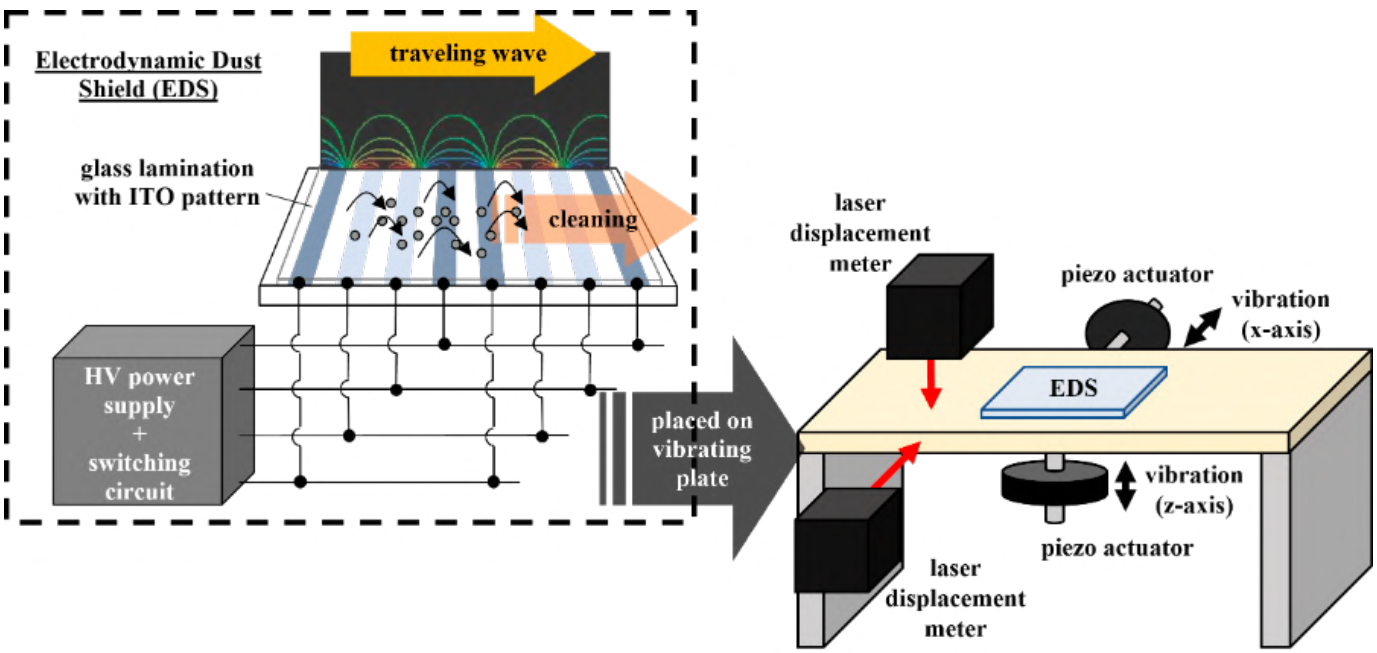


Figure 1 Experimental setup of an electrodynamic dust shield (EDS) with vibration assistance. The EDS is placed on a plate vibrating in x- and z- axis.

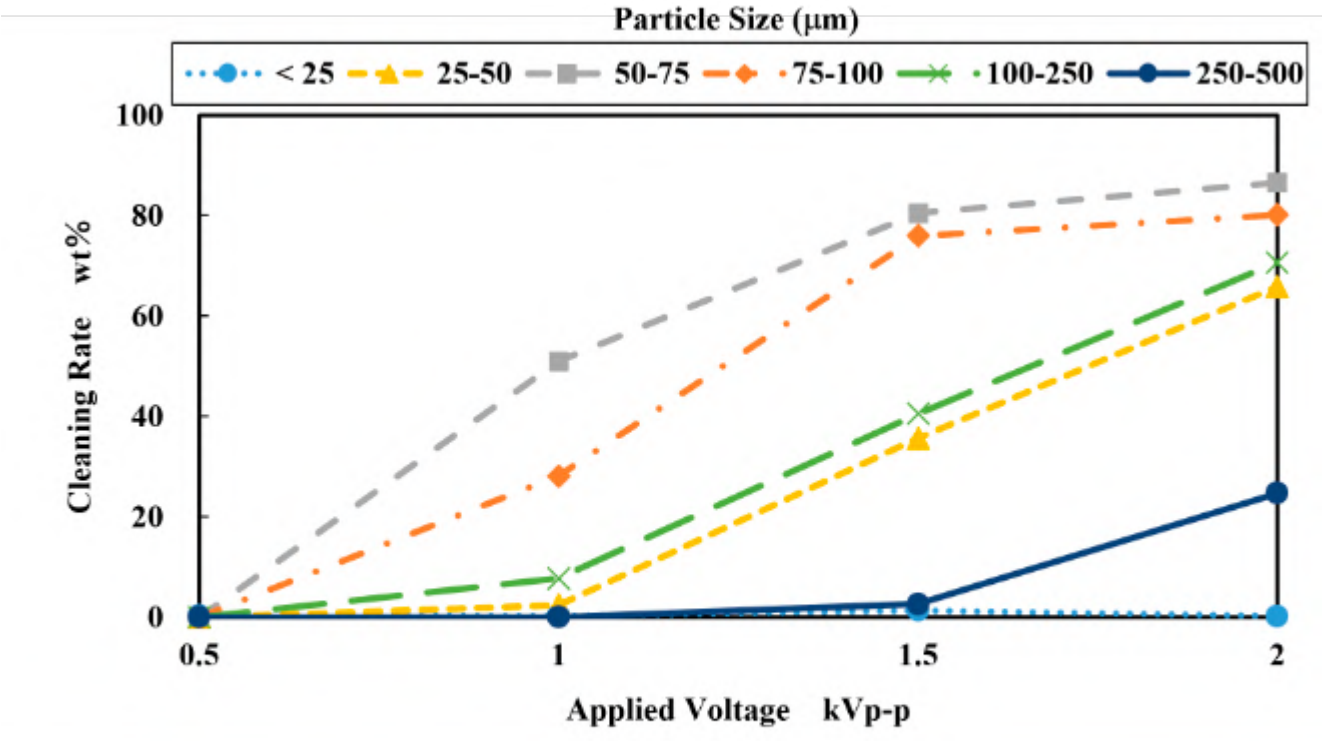


Figure 2 Cleaning performance of EDS without vibration assistance. The regolith simulant particles are sorted in size in advance of the cleaning experiments, an applied voltage and the size range are varied as experimental parameters.

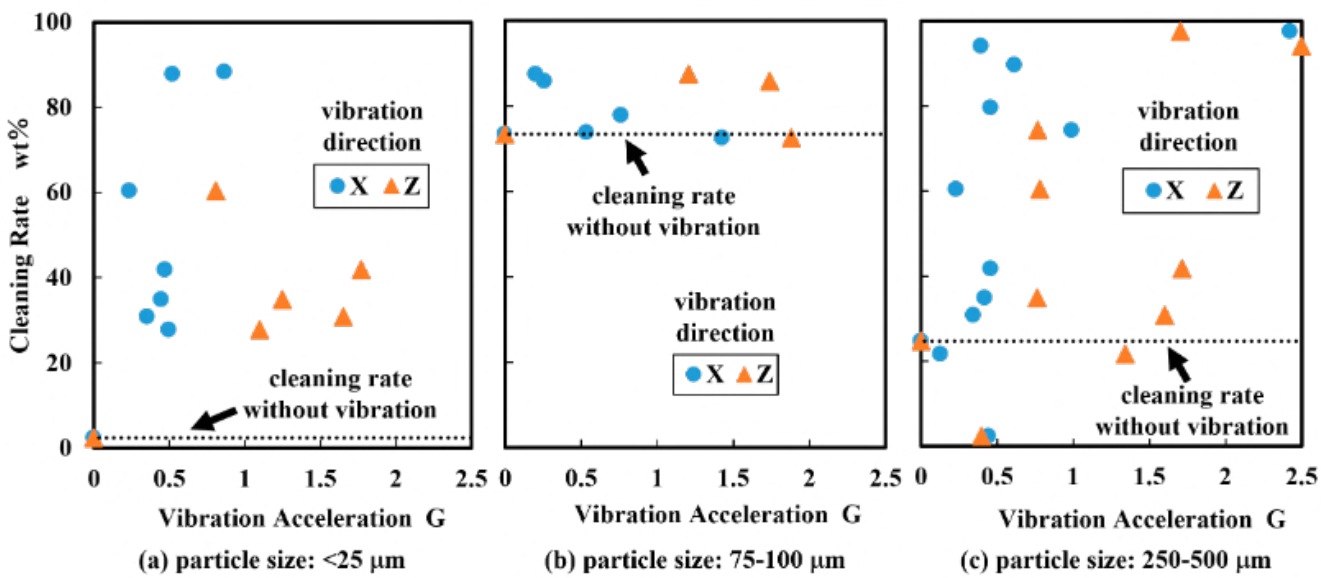


Figure 3 Cleaning performance of EDS with vibration assistance. Three types of sorted particles are used; (a) less than 25μm, (b) 75-100μm, and (c) 250-500μm. The dashed line in each graph shows the cleaning rate in case of not using vibration assistance.

Novel method for evaluating the effect of elevated temperature on powder flow properties

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Keywords | Powder Flow, Elevated Temperature, Powder Properties, Flowability

An increase in ambient temperature can affect powder properties in multiple ways: solid particles may deform or partially melt, moisture may evaporate, and the density of the entrained air can decrease significantly. In an industrial setting, many processes involve heating powders to extremely high temperatures, but even within normal ambient ranges uncontrollable temperature changes can lead to stoppages, downtime, and sub-quality products. The combined effect on powder flow properties of all the factors that are affected by a change in temperature is very complex to model and predict from particle characteristics.

We present a testing procedure to measure directly the effect of temperature on the powder flow properties measured with an FT4 Powder Rheometer® (Freeman Technology, UK). In the proposed setup powders can be heated to temperatures as high as 200°C using a robust silicone heating jacket. The temperature and flow energy of the powder itself is monitored in real time, so the flowability of the powder is safely characterised as a function of temperature.

We investigate the changes in flow properties during both heating and cooling processes for a variety of powder samples. We show that heat can affect powder flow properties in very distinct ways and at different speeds of heating, and we are able to interpret results to differentiate the effects of moisture loss and particle softening. By testing powders at operating temperatures, the methodology developed in this work can quantitively support the prediction of powder behaviour in industrial processes involving elevated temperatures.

Effect of the mixing sequence and adsorption behavior of binders on rheological characteristics and packing ability of aqueous graphite slurries

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(1) Hosei University

Keywords | Lithium-ion battery, Electrode slurry, Mixing sequences, Particle dispersion, Adsorption

It is well known that various properties of the products fabricated from powder strongly depend on the particle dispersion state in the slurry. For example, the performance of lithium-ion battery electrodes is influenced by particle dispersion in the slurry used for their production; on the other hand, its mechanism is not clearly. In this study, we elucidate the effects and mechanism of the binder-mixing sequence on the characteristics of the graphite aqueous slurry in order to establish the slurry preparation guidelines to achieve the desired particle dispersion state.

The graphite aqueous slurries in which added Na-CMC and SBR were prepared using the four mixing sequences. CMC→SBR: graphite was mixed with the CMC solution, whereafter the SBR solution was added. SBR→CMC: the SBR solution was mixed with graphite, whereafter the CMC solution was added. SBR+CMC: CMC and SBR solutions were mixed, whereafter graphite was added. SBR: graphite was added to the SBR solution. We measured the adsorbed amount of binder, and discussed its effect on packing and flow characteristics.

During the preparation of the graphite aqueous slurries, the state of the adsorption of the binder to the particle changes when the sequence of binder addition is changed. The change in the adsorption state of the binder influences the particle dispersion in the slurry, rheological properties of the slurry (Fig. 1.). The slurry generated by the SBR mixing sequence became too clumpy for its viscosity to be measured. We constructed a model for the adsorption of the binder and the change in the particle dispersion state and considered the optimum mixing sequence.

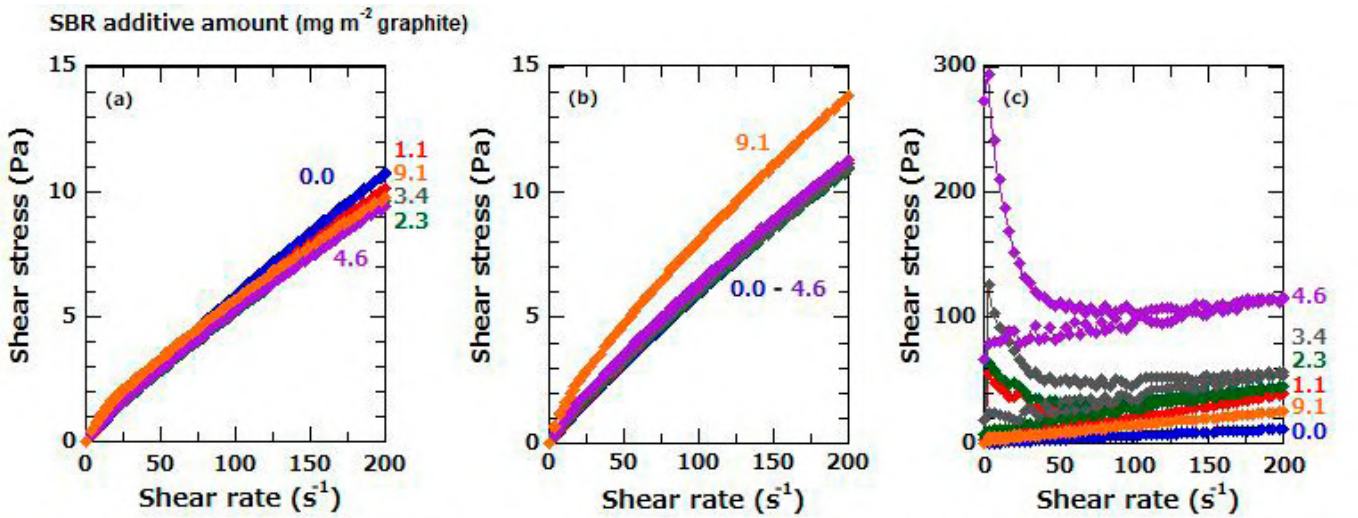


Fig. 1. Flow curves as a function of SBR additive dosage in each mixing sequence: (a) CMC SBR, (b) CMC+SBR, (c) SBR CMC. (The slurry generated by the SBR mixing sequence became too clumpy for its viscosity to be measured.)

From the above, we drew the following conclusions:

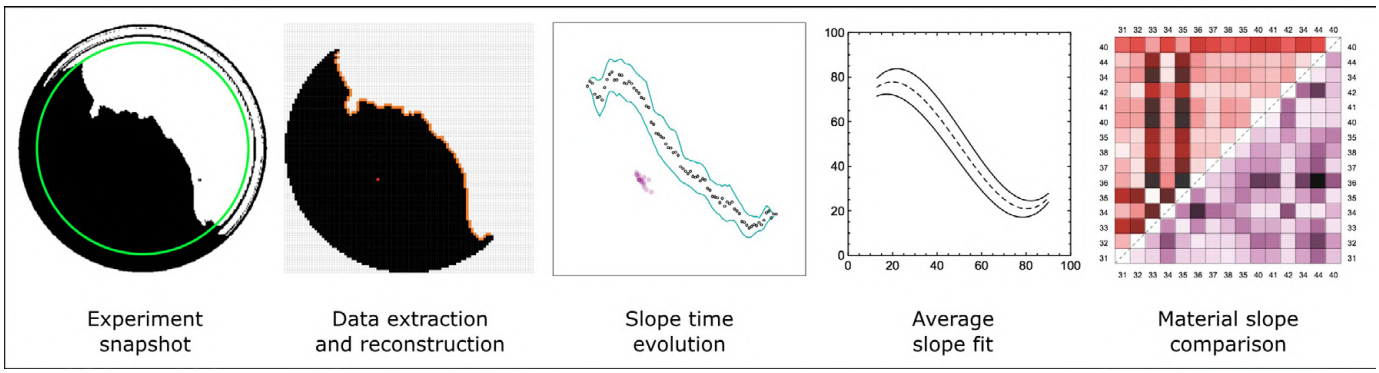
The state of the adsorption of the binder to the particle changes when the sequence of binder addition is changed and the change in the adsorption state of the binder influences the particle dispersion in the slurry.

If SBR is added before the other components to form aggregates, it is difficult to create a state in which the particles are uniformly dispersed.

A general framework for data extraction and analysis of dynamic angle of repose tests

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Keywords | Dynamic angle of repose; Cohesive powders; Flow profile; Material properties; Numerical models calibration.



The dynamic angle of repose test is a common powder rheology experiment performed to measure the flow properties of granular materials under negligible consolidation stresses. Materials are placed inside a rotating drum and pictures of their slope are taken at periodic intervals. The slope depends both on material parameters, such as friction and cohesion, and geometry, such as shape and size distribution, alike. Despite the rich physics involved in the flow, this test is mostly used to compute the angle of repose by means of a linear fit of the material profile.

While this measurement is very useful and reliable for free-flowing materials, when it comes to cohesive powders the profile becomes proportionally lumpy and rough. The profile texture also changes in time as uneven lumps of aggregated material are transported along the flow. This makes the extraction of the dynamic angle of repose an almost impossible task, since a linear fit of the powder profile is impossible and the interplay between avalanching period of the lumps and sampling time affects the statistics.

In our work we present a generalised framework where various observables are extracted from powder profiles, such as slope and its fluctuations, center of mass evolution and average avalanche volume. We show the procedure behind the image analysis and how data are extracted and processed to obtain physical observables of interest. These quantities go beyond the simple angle of repose, thus allowing richer and broader knowledge to be obtained from this simple and ubiquitous test. We relate the former to particle properties, and we formulate concepts of similarities between flows to compare powders. We also illustrate another added value of this framework: the flexibility to analyse experimental and numerical data alike. This point, in addition to the observables variety, is particularly useful when comparing numerical models to experiments, and can be easily and quickly exploited by numerical calibration procedures based on machile learning.

Dispersion in Horizontal Stirred Bed Reactors

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(1) University of Twente

Keywords | Horizontal Stirred Bed Reactor (HSBR), discrete particle model, dispersion

In this work, we investigated the mixing of particles in an industrial horizontal stirred bed reactor (HSBR) based on a discrete particle model using the open-source software MercuryDPM (Weinhart et al., 2020), see Fig. 1. The goal was to compute and investigate axial and radial dispersion of the particles in the HSBR. HSBR is a type of reactor for powder mixing with a series of paddles connected to a central shaft that agitate particles.

The dispersion coefficient measures the amount of spread of a material volume in a given time interval, i.e., how far an average particle travels from the position predicted by the streamline. The parameter is used in the continuum, advection- diffusion models to predict the mixing of components in a bulk material efficiently. Thus, measuring the dispersion coefficient is a good method to transition from a discrete to a more efficient continuum model.

For the HSBR process, we were interested in the axial (z) and radial (r) dispersion. To compute the dispersion coefficient, we compared and contrasted the methods previously presented by Yang (2018) and Ingram (2005). Both can be applied to extract the axial dispersion, but only the latter is easily applied to radial dispersion. We observed sub-diffusive behavior. To explain this behavior, we also analyzed a simple drum, and found a normal diffusion; thus, we concluded that the sub- diffusivity in the HSBR was likely caused by the blades. To validate the results, we predicted the Lacey mixing index for the measured axial dispersion coefficient and compared it with the Lacey mixing index based on particle tracking. We observed that they were in good agreement, see Fig. 2.

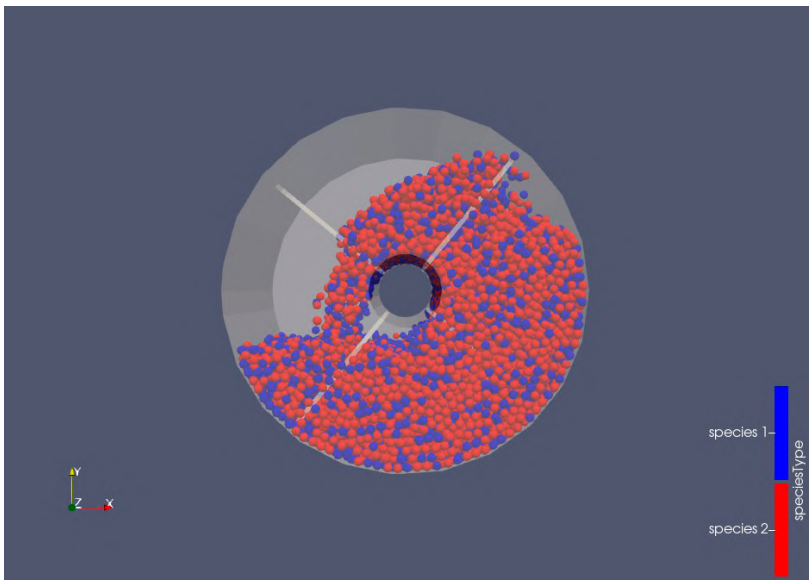


Fig. 1. Simulation of the mixing process in the HSBR.

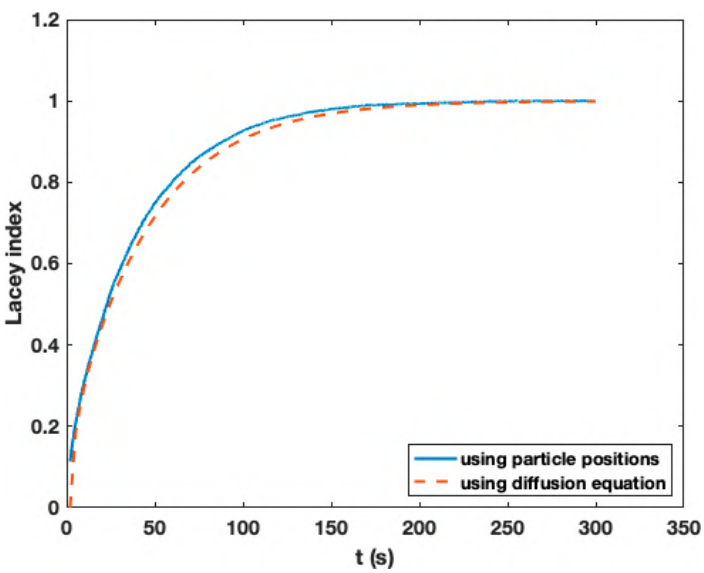


Fig. 2. Lacey index for axial mixing.

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Investigating the Shear Stability of Spray-Dried Si/C Microagglomerates in Lithium-Ion Battery Anode Slurries

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Keywords | Lithium-ion batteries, Silicon nanoparticles, Spray-drying, Anode slurries, rheological characterization

An ever-increasing demand for green energy solutions accelerates research activities for materials with enhanced properties in various energy related applications [1]. In this context, silicon and silicon-based composites emerged as most promising replacement of graphite as anode material in lithium-ion batteries (LIB) [2,3]. However, due to inherent large volume changes in the course of lithiation / delithiation cycles rapid degradation of cells is usually observed for silicon-type electrodes. One approach to overcome this issue is through nanostructuring of the active material. Still limited by some unfavorable phenomena (e.g. low tap density and low packing), additional steps to improve the overall electrochemical performance through hierarchical structures by the use of spray-drying can be introduced [4].

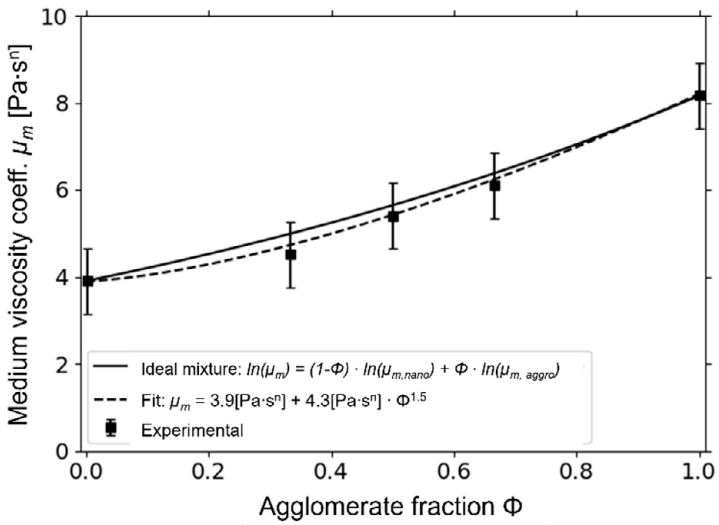


Fig.1: Medium viscosity coefficient μ_m as a function of agglomerate fraction Φ depicting a characterisitic rheological parameter for Si/C-based full slurries

In this context, we report on the development of a detailed rheological flow model for hierarchically structured silicon-carbon composite materials. They are composed of nanostructured Si/C forming secondary, spherically shaped microparticles in the course of spray-drying. Bound together by a suitable binder, the µ-agglomerates are exposed to high shear forces during the slurry mixing procedure. Their mechanical stability is an important parameter in their subsequent processing towards final electrodes. Hence, a quick quality control parameter on slurry stage is sought.

Our approach involves the rheological investigation of model slurries with defined fractions of unstructured Si/C nanoparticles and secondary µ-agglomerates simulating possible agglomerate breakage during slurry mixing. By this, we parametrize a structural kinetic model for the shear-thinning slurries and analyze their thixotropic properties. This enables us to identify key indicators for rheological features as a function of agglomerate fraction (Fig. 1).

To conclude, our investigation shows how advanced rheology can help to fully utilize materials already known to be very promising in LIB. Both already existing mixing processes for Si/C can now be assessed in a fast way and new process developments (e.g. spray-drying) can be established through the availability of key rheology indicators. In future, we will develop our rheological model towards more fundamental levels and expand its applicability to other energy-related materials that are a key part in formulation science and particle technology.

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Relevance of hight temperature packing dynamics for Additive Manufacturing characterization

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Keywords | Packing, high temperature, Additive Manufacturing

Powder characterization is of huge importance for improving Additive Manufacturing process. Methods based on selective layer melting preheat the powder bed higher than the ambient temperature to decrease the amount of energy needed for the laser irradiation to melt the powder, especially with polymer powders (Lumay et al., 2020). For this reason, powder characterization at the same temperature than the manufacturing process would be relevant in the future (Neveu et al., 2021).

Packing is a popular method to characterize powder flow and density. We investigated how temperature can influence the packing dynamics of plastic powder. We performed packing dynamic measurement with the GranuPack high temperature. The experimental protocol is the following: a steel tube, in which is poured the powder, performs a succession of free falls of 1mm of height, called “taps”. After each tap, the height of the granular pile is measured by a laser and the bulk density is computed from the knowledge of the tube diameter and the mass of the powder. The bulk density curve as a function of the taps number is then obtained. The temperature of the tube is controlled by a surrounding electrical heating jacket which can heat from ambient temperature to 200°C.

Significative changes between bulk density curves are observed when temperature increases, affecting both the packing range and the packing kinematics, as illustrated in Figure 1.

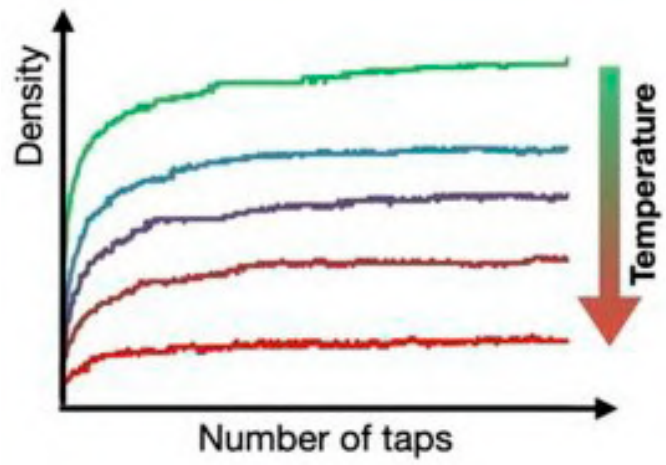


Figure 1: Evolution of the bulk density curve with the temperature.

Indeed, we observed that the initial density and the tapped density after 500 taps, decrease with the temperature. Moreover, the Hausner ratio, generally used to describe the packing dynamics, decreases as the temperature increases. These results indicate an evolution of particle properties and highlight the importance of temperature in powder characterization.

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Suitability of eroded particles from electro discharge machining (die-sink EDM) for additive manufacturing – characterization and processing of waste stream products

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Keywords | additive manufacturing, electro discharge machining, eroded particles, particle characterization, recycling / upcycling

In additive manufacturing (AM) specific metal powders are used in different techniques, which are only produced via the cost- and energy-intensive process of powder atomization of metal melts (Tsunekawa et al., 2013). Therefore, an alternative way of supplying and generating metal powders for AM-processes is indispensable. When postprocessing components by electro discharge machining (EDM) (Ho et al., 2003), which takes place in a fluid dielectric, erosion sludge or debris accumulate. EDM is based on an erosive character of electrical discharges, caused by the transformation of electrical energy into thermal energy leading to a material removal. Molten material congeals in the dielectric, which leads to different particle generation mechanisms. The aim of this study is to extract these particles from the oil phase, followed by a detailed analysis and perform an upcycling for a possible usage as a secondary AM-powder for selective laser melting or electron beam melting processes. Experimental techniques such as laser diffraction and dynamic image analysis, filtration and washing experiments as well as magnetic separation steps were performed, to show the theoretical suitability for an AM- usage. Furthermore, investigations with SEM combined with EDX and X-ray CT were carried out. Additionally, optical emission spectroscopy (ICP OES) was used for identifying the chemical composition as well as thermal analysis coupled with FT-infrared spectroscopy. In general, it could be shown that recycled particles are able to fulfill main requirements for AM- powders, which indicates a possible usage. All the results were contrasted with an intensive literature review of references dealing with eroded particles in another context, starting from the 80's to present times, to underline the AM-suitability, as stated in (Berkowitz et al., 1987) or (Khanra et al., 2007). After procedures of sieving & sifting two powder fractions will be processed later on in AM-machines and properties out of tensile or compression tests will be compared to reference materials.

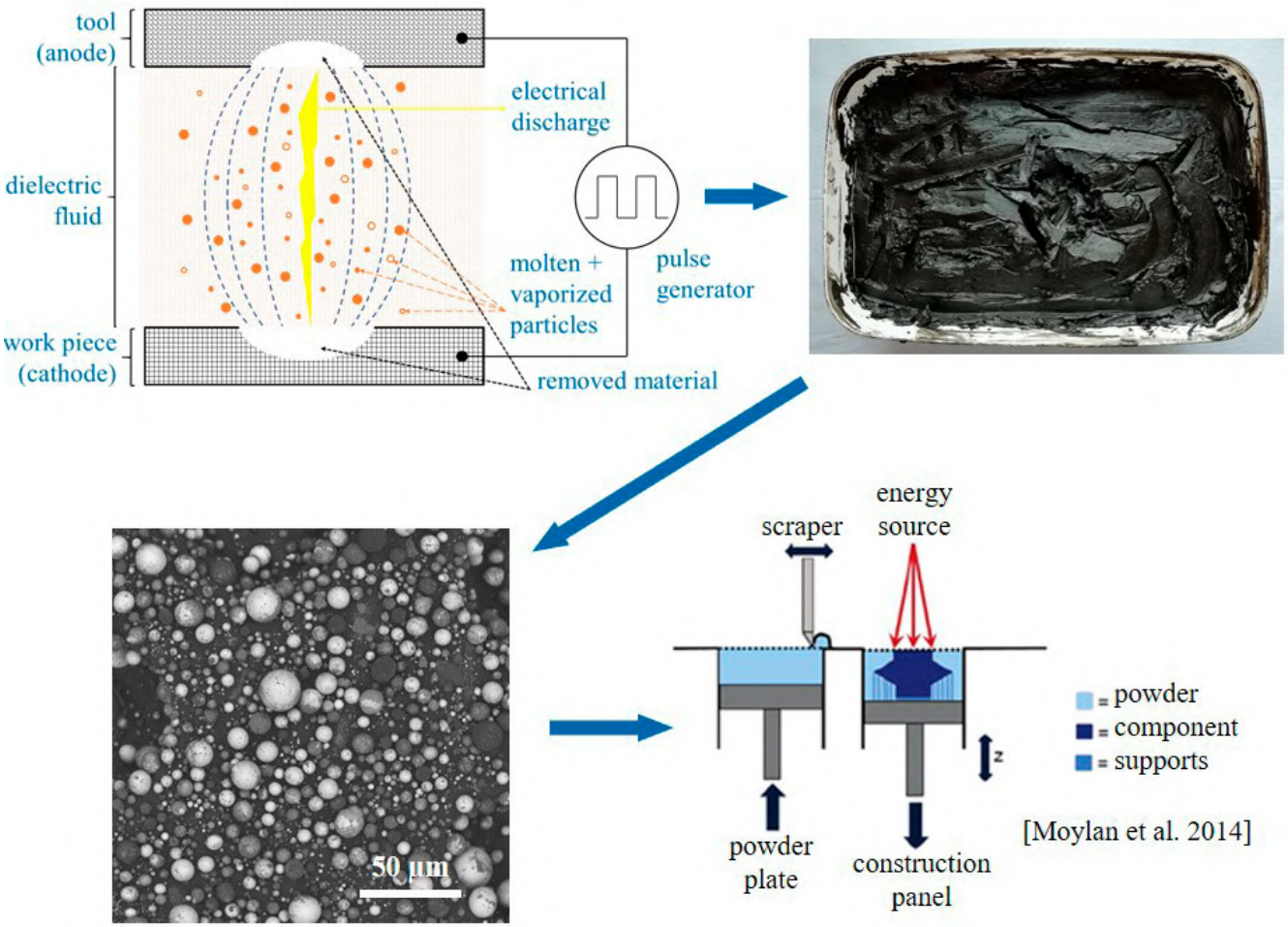


Figure 1 / graphical abstract: Flow sheet of study aim: particle generation mechanism, oily sludge (both top) processed particles and AM-process (both bottom)

References are included in the attached file

The role of Temperature and Moisture on Polymer Materials for Additive Manufacturing, and their Implications for the Process

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Keywords | Additive Manufacturing; Temperature Control; Polymer Powders

Polymeric materials in additive manufacturing add a unique difficulty in characterization to a field that is used to deal with metal powders.

Due to the molecular structure of these polymer powders, moisture and temperature change their behavior to a larger degree than is to be expected with the more common metal feedstock.

Like in Metals Manufacturers have addressed this by bringing the temperature of the feedstock chambers to elevated levels, therefore reducing or eliminating the moisture effects. However, unlike metals, polymers show a substantial divergence from their room temperature This in turn creates the problem of reliably measuring the feedstock at these elevated temperatures to give an accurate picture of their properties during use. And in addition the problem of moisture influence during storage prevails.

In this work we will show measurements of polymeric feedstock under actual processing parameters and delve into the problems arising

during drying and storage.

It will consist of measurements with temperature and moisture controlled ring shear cells as well as data from our novel temperature-controlled stirrer-based cell with fluidized methods.

Pre-alloyed powders of Ti-Fe ultrafine eutectics for laser Additive Manufacturing

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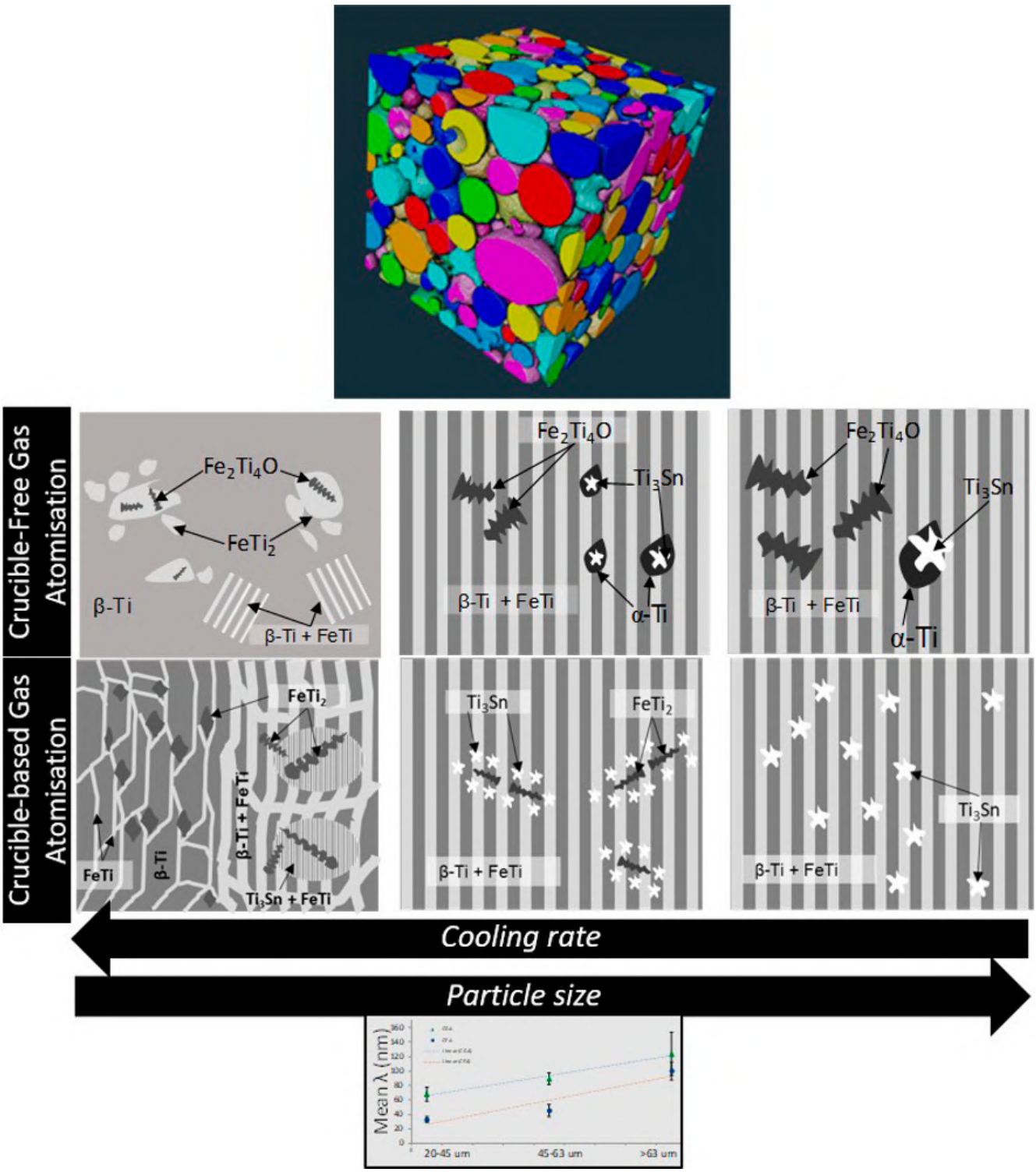
Keywords | metal powders, gas atomisation, ultrafine eutectics, characterisation, additive manufacturing, XCT

Years of academic research has gone into developing Ti-Fe based ultrafine eutectic and near-eutectic alloys with remarkable mechanical properties (Das et al., 2005). However, conventional casting methods have only managed to produce small specimens because of their strict solidification criteria requiring fast and uniform cooling rates. Selective Laser Melting (SLM) seems to be the ideal, and yet largely unexplored processing route for bulk ultrafine eutectics. Pre-alloyed powders of near- eutectic compositions containing Ti-Fe-(Nb, Sn) were produced by three standardised gas atomisation methods, named as Crucible-based Gas atomisation (CGA), Crucible-Free atomisation (CFA) and Arc-melting Atomisation (AMA) for SLM.

Part of the success in powder-bed based techniques like SLM lies in the physical properties of the powder particles used as raw material, such as particle morphology, size distribution and porosity, since these determine the critical powder behavior in terms of flowability and apparent density. This work covers the complete characterization of gas-atomised powder particles performed using conventional techniques like Scanning electron microscopy and laser diffraction particle size analysis. In addition, a novel methodology is proposed based on X-Ray Computed Tomography (XCT) of powders and 3D image analysis. This advanced routine allows the determination of several features like porosity, size and shape distribution of powders in a single step with much higher accuracy than conventional powder characterisation methods.

Eutectic alloys are quite sensitive to compositional fluctuations and solidified via different pathways in the three methods resulting in different microstructures. Electron Microscopy, X-ray Diffraction, image analysis and nanoindentation were used to understand the solidification behaviour in powder particles obtained by different gas atomization methods and across different sizes, thus, different crystallisation kinetics. Further, these results were compared to SLM-produced microstructures of the same alloy. Presence of meta-stable phase in powders and ultrafine inter-lamellar spacing (< 190 µm) of the eutectic matrix, which increased with powder particle size, highlight the potential of gas atomisation as a method to study rapid solidification.

Das, J., Kim, K. B., Baier, F., Löser, W. and Eckert., J, 2005. "High-Strength Ti-Base Ultrafine Eutectic with Enhanced Ductility." Applied Physics Letters 87 (16): 1–3.



Spreadability versus Flowability: Transient Jamming Makes Them Different

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Keywords | spreadability; flowability; DEM; rheology

In additive manufacturing by application of radiation energy to a powder bed, fine powder is first spread by a blade or a roller to form a

thin layer. The quality of layer in terms of the uniformity of the particle spread is critical for the quality of the final product, being free from defects. The ability of powder to be spread uniformly as a thin layer is described by spreadability. This depends on particle properties as well as the dynamic of the spreading process, and is commonly assessed by common flowability testing. However this approach might not give a full indication of powder spreadability because flowability testing is essentially a bulk testing method, where the failure in a natural bulk plastic zone gives an indication of flowability. There are two shortcomings in this approach which can affect the outcome, one is the absence of a narrow gap, in which transient jamming occurs, as prevailing in the powder spreading process, and another is the powder rheology in narrow gaps, which cannot be assessed not only by flowability tests, but also by powder rheometry. This requires an understanding of transient jamming and powder rheology in narrow clearances, a topic of investigation here.

In this presentation, both experimental and numerical analysis by Discrete Element Method have been carried out comparing the outcomes of flowability and spreadability assessments using a powder rheometer (Freeman Technology FT4) and also spreading by a blade. A special methodology is suggested for assessing the spreadability of a powder.

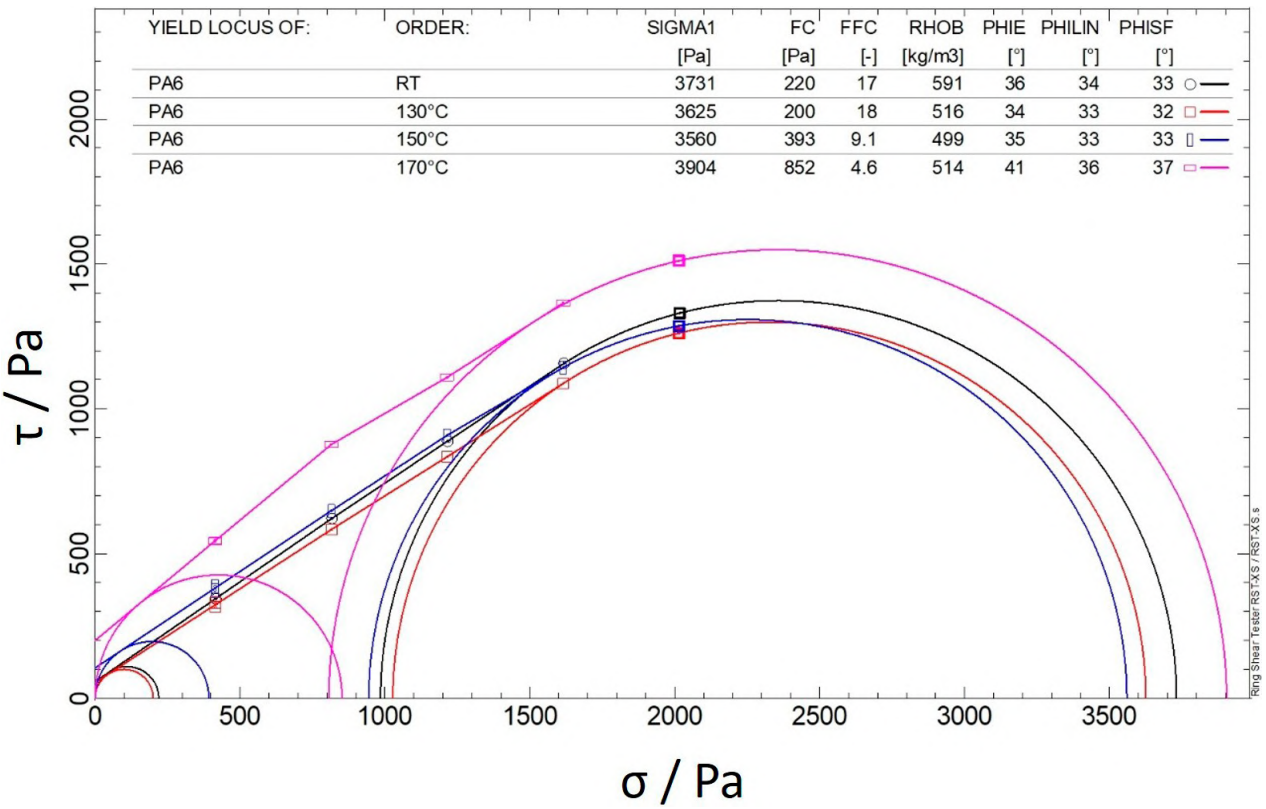
Flowability of polymer powders at elevated temperatures for additive manufacturing

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Keywords | Flowability of bulk solids, additive manufacturing

Measuring the flowability of bulk materials is important wherever powders must be transported, stored, or dosed. A new field of application is represented by powder bed-based additive manufacturing processes. These processes, for polymers called selective laser sintering, first apply a thin layer of the powder and selectively fuse it using a laser. This is repeated until a complete structural component is created. The application of the powder is particularly critical in this process, as imperfections in the powder layer can lead to defective components or construction job failures. One reason for this can be insufficient flowability of the powder material used. For process control of the SLS process, knowledge about the flowability of the material is of great importance.

Because of its importance in different fields, various methods to quantify the flowability were established like shear tests or avalanching. In order for the measurement of flowability to provide representative results, it is vital that the measurement methods use similar consolidation stresses to those used in the present application. Additionally, it is mandatory to investigate the flowability of the polymer powders depending on the temperature, which is not an option with all measuring instruments available on the market due to the lack of heating possibilities. For these reasons, a self-modified ring shear tester and a Revolution Powder Analyzer (RPA) were selected for further analysis. Both instruments were used to measure different SLS materials at temperatures up to 170°C. First results for a ring shear test at 2000 Pa pre shear stress for different temperatures are shown below and implicate an increase in flowability for a temperature of 130°C in comparison to lower and higher temperatures. To get a further insight into the temperature dependent flow behaviour, additional measurements at different pre shear stresses were carried out and the results will be presented. A comparison between the two measuring instruments will be provided as well as new methods to analyse and evaluate the RPA results. Finally, application tests in an SLS machine will provide information on which properties are best suited for evaluating the flowability of SLS materials.



Role of temperature on the polymeric powders flow properties in selective laser sintering

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Keywords | Selective Laser Sintering, High Temperature, Powder flowability, Flow Function, Bond number

Selective laser sintering (SLS) creates 3D sintered products by adding powders layer by layer. In order to reach an acceptable quality of powder bed and decrease the possible defects on the final artifact, the proper flow behavior of powder is required, which makes to feed and distribute powder perfectly.

Heating up the sintering chamber in SLS can have some advantages including reduction of the thermal gradient to avoid crack formation and distortion in the final product, saving the costs and energy by lowering the required laser power, and increasing the densification of a sintered part. On the other hand, a significant raise in temperature can have negative effects on powder flowability. In other words, wider contact areas between particles during plastic deformation, with increasing temperature, will be led to poor flowability. Consequently, this may cause an increase in the intensity of van der Waals interparticle forces. At higher temperatures, melted phases formed on the surface of particles could make more intense capillary forces between particles arise.

In this work, the effect of temperature on the flowability of four polymeric powders including Polyamide 6, Polypropylene, Polyamide 6 black, and Thermoplastic polyurethane was studied by Anton Paar Shear Cell at different consolidation stresses. Yield loci and flow functions were obtained from 27°C to approximately approaching the melting point. According to the outputs, powder flowability classification was done by calculating the granular Bond number which is the ratio between the interparticle force, estimated from the measured frictional and cohesive flow properties at the bulk scale, and the particle weight due to gravity. Generally, results highlighted that increasing the temperature reduces the powder flowability, especially near the melting temperature.

In conclusion, the polymeric powders' results emphasized that the highest amount of van der Waals interparticle forces occurred at the peak temperature and most considerable consolidation stress, which means poor flow properties were revealed.

Characterisation of Spreadability Behaviour of Ti6Al4V Powders for Additive Manufacturing

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Keywords | Spreadability, additive manufacturing

Powder-bed based Additive Manufacturing (AM), also known as 3D printing, is an emerging technology to produce high quality end-parts at a cost and time effective manner as compared to the traditional subtractive manufacturing processes. The study of metal powder-bed processes usually involves two critical steps which includes the spreading of the powder and the fusion of the layers. Spreadability of powders is an essential characteristic in determining the total build time and quality of the final product. The spreadability is thought to be linked to the powder characteristics, mostly the flow behaviour, which is influenced by the individual particle properties and environmental conditions.

This work endeavours to investigate the spreading behaviour of two samples of titanium alloy (Ti6Al4V) powders, produced by Gas Atomisation (GA) and Hydrogenation-dehydrogenation (HDH) methods. The spreading behaviour of each sample has been investigated using an in-house spreading rig set up at the University of Leeds, with a set of parameters, including the gap size between the blade and build plate (ranging between 191 mm to 508 mm) and spreading velocity (ranging between 50 mm/s to 200 mm/s) as the variables, while the mass of the fed powder as a constant. The bulk layer density and mass per area are the two measures of spreadability introduced in this study.

It is found that the quality of the spread layer is significantly influenced not only by the powder properties, but also the process parameters. GA powder exhibited spherical shapes, which in turn created homogeneous layers due to improved packing behaviour compared to HDH powder which were characterised by irregular shapes impeding powder flow.

Additionally, the bulk layer density of GA powder decreased when the spreading velocity was increased, but this correlation was not established for HDH powder. An increase in the spreading velocity also resulted in a gradual reduction in the values of the mass per area, suggesting that higher velocities jeopardise the quality of the spread layer.

The spreading behaviour of stainless steel powders for additive manufacturing

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Keywords | Spreadability, additive manufacturing

Additive manufacturing (AM) is a rapidly developing technology that transforms the manufacturing toolbox and operation of industrial companies. In this technology, powder is selectively added layer-by-layer in order to create a complex part that is not easy to be produced in subtractive methods. Physical and chemical behaviour of powders used in additive manufacturing is a key element in industrial applications. Hence, it is necessary to accurately control and optimise the processing techniques with precise powder characterisation. There has been a considerable attention to the bulk powder behaviour in different fields of powder technology, but there is a lack of extensive research on the spreadability of powders in additive manufacturing (AM). While quantifying powder spreadability is a vital step for AM, there is no standard strategy for this purpose. Lack of a generally agreed definition for spreadability in AM might be related to the heterogenous nature of the powders which may differ by powders types, method of AM application and processes conditions. Therefore, precise monitoring of the spread behaviour of powders seems to be very challenging since powders may change their spreading dynamics at any period of time during the experiments which results in uncertainty.

In this project, the combined effect of powder characterisations and spreading process parameters such as gap size between the blade and the built plate, spread velocity and environmental conditions on the spreadability of stainless steel powders have been investigated. The samples are two different batches of 316L stainless steel with the particle size distributions in the range of 15-105µm. Series of experiments are conducted through using an in-house spreading rig to quantify the spreading behaviour of two different samples. This work investigates the spread quality of powders through obtaining the bulk density of spread layer on the build plate and form a comparison to the initial bulk density of the respective powders.

Acknowledgement

The authors would like to acknowledge financial support from EPSRC-UK Future Manufacturing Hub in Manufacture using Advanced Powder Processes (MAPP) (EP/P006566/1, www.mapp.ac.uk).

Using Jenike Shear Cell to Measure Wall Friction of Particles immerged in Water

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Keywords | Jenike Shear Cell, Wall friction, Van-der waals force

The cohesion value is strongly influenced by the interparticle cohesive forces including Van der Waals forces and liquid bridge forces. In order to investigate the contribution of the cohesion forces, and the friction mechanism for wall friction measurements, a number of tests were conducted using Jenike shear tester at various conditions. The preliminary experiments were conducted for glass spheres in several diameters.

The test conditions were aimed to test various effects on the wall friction measurements. First, the standard measurement was performed with dry particles. As expected, the wall friction increases as the particle size decreases, mainly due to Van- der-Waals forces. In order to test the effect of the cohesion forces, a test was conducted while the Jenike Shear cell was fully immersed in water to eliminate Van-der-Waals forces. Indeed, the coefficient of friction was found to be lower also due to reduced friction coefficient between the two materials. This effect was further investigated by testing more materials.

Multiple scattering effects on intercept, size, polydispersity index, and intensity for parallel (VV) and perpendicular (VH) polarization detection in photon correlation spectroscopy

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(1) Malvern Panalytical

Keywords | light scattering, materials characterization, particle size and distribution, particle characterization, polarizers, multiple scattering

Dynamic light scattering (DLS) also known as Photon Correlation Spectroscopy (PCS) and Quasi-electric light scattering (QELS) is well established for rapid size, polydispersity, and size distribution determination of colloidal samples from approximately 1nm to a few microns. The technique is prevalent in vast array of sample types from emulsions, primary materials, biologics, surfactants, and drug delivery platforms including liposomes, dendrimers, and polymer nanoparticles. With the ease of use of today's commercial DLS instrumentation comes an inherent danger of misinterpreting or misapplying results from these various sample types. We show how comparison of different polarization components can help reveal undesired multiple scattering, which can potentially disguise a sample's true mean size and polydispersity. We show that the contribution of multiple scattering events effectively reduces both the measured scattering intensity and the apparent size from the autocorrelation function. The intercept of the correlation function may serve as an indicator of relative strength of single to multiple scattering. Furthermore, the abundance of single scattering events at measurement positions close to the cell wall results in an apparent increase in uniformity yielding a lower polydispersity index (PDI) which is more representative of the physical system. These phenomena were exemplified by studying milk as a real-world sample and latex spheres for confirmation.

Insights in the rehydration kinetics of powdered food ingredients through Broadband Acoustic Resonance Dissolution Spectroscopy (BARDS)

Deleris, Isabelle (1)
(1) Cargill

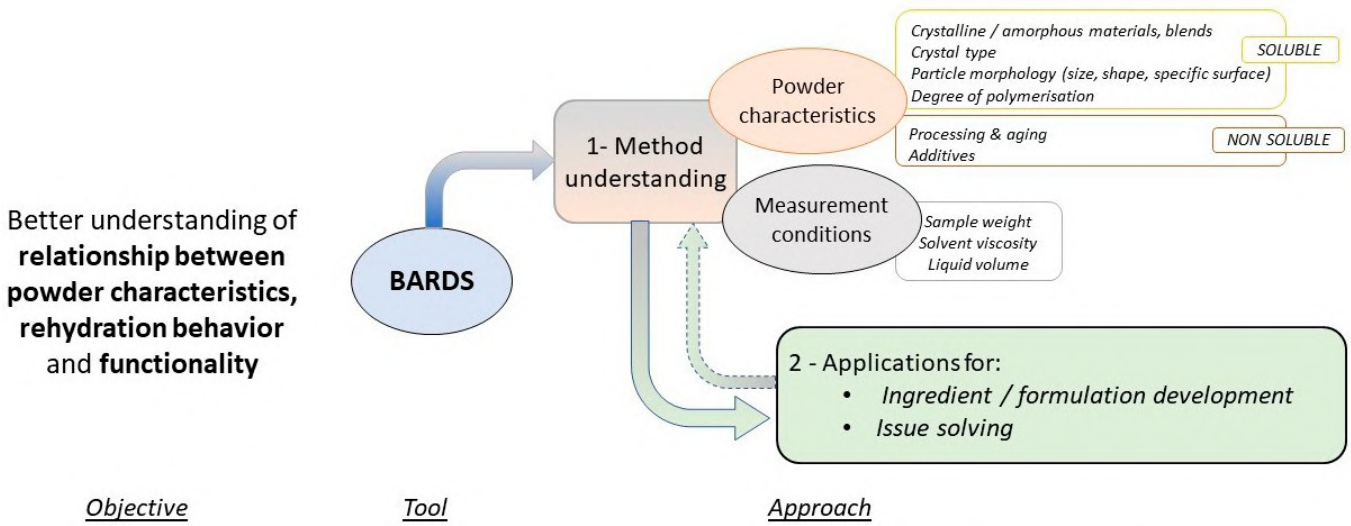
Keywords | rehydration kinetic, BARDS, wettability, dispersibility, functionality

When dealing with development of powdered ingredients for food and non-food applications, one key element to consider is their behavior during reconstitution, final step allowing functionality development (texture, color, taste,...).

Powder rehydration has been largely addressed in literature. The main challenges to establish correlations between powder characteristics and rehydration performances come from process multiscale and dynamic aspects and the dependence of results on characterization method and type of products.

Recently, BARDS has been developed and successfully applied to get more insights in rehydration mechanisms of a broad range of compounds. In addition to the continuous and quantitative information that it provided, the advantage of this method is the mechanistic description associated to it (Cronin, Wu et al. 2021).

The objective of this work was to apply this method on key products to better understand their rehydration behavior. Due to the complexity of the rehydration process and the diversity in the type of ingredients to characterize, a two-step approach was applied, focusing first on method understanding and then applying it to specific cases (figure below).



From methodological perspective, a guideline was established to allow the selection of the optimal measurement conditions, including the type of solvent, the amount of sample, depending on powder type and properties,

From powder perspective, the effect of particle shape and size as well as crystal arrangements on rehydration profiles was confirmed for crystalline soluble materials. For amorphous materials, we showed that BARDS rehydration profiles could discriminate maltodextrins with different DEs. When non soluble materials were considered, interesting information were also obtained, with distinct rehydration profiles depending on processing conditions, presence of additives or powder age. All these elements are key information to consider for the development of products with improved rehydration properties.

Overall, by combining experimental characterization and mechanistic modelling, BARDS constitutes a powerful tool to better understand the key or limiting factors for optimal rehydration and thus orientate product/process modification for functionality improvement.

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Image-Based Particle Analysis via Deep Learning

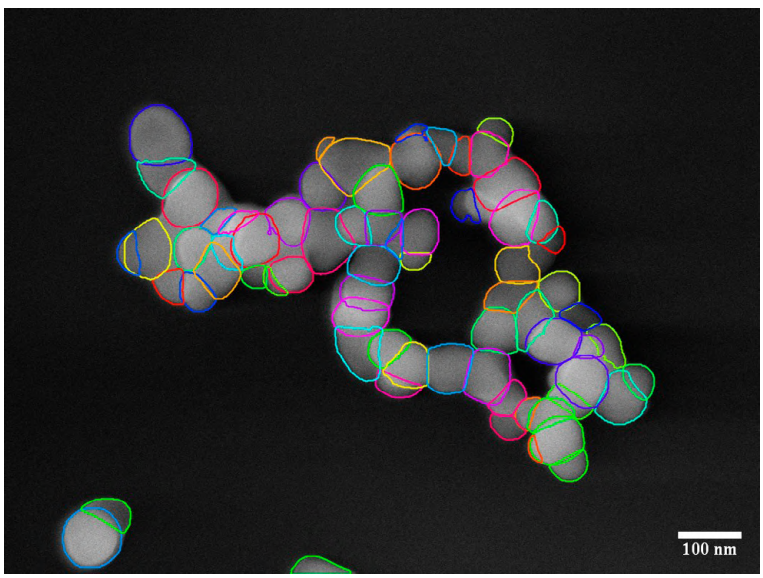
Frei, Max (1); Kruis, Frank Einar (2)
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Keywords | deep learning imaging particle analysis particle mixture analysis agglomerate analysis fiber analysis

Image-based analysis is a straight-forward route to information about the sizes and shapes of particles. However, it still poses a major challenge, especially when dealing with aggregates, or non-spherical primary particles. While acceptable results can be achieved with conventional automated algorithms if the involved image processing parameters are carefully tweaked, they are susceptible to changes in the imaging conditions, which results in a frequent retuning. Consequently, a laborious manual annotation is often the last resort to achieve robust, high quality results.

Using deep learning-based methods, the tweaking of image processing parameters can be delegated to a learning algorithm, which finds the optimal parameters based on a set of already annotated training images. In the presented work, we therefore evaluated the applicability of state-of-the-art deep learning architectures for image-based particle analysis, by carrying out four case studies concerning the size analyses of:

1. soot aggregates (Sipkens et al., 2021)
2. aggregated silica spheres (see Figure; Frei et al., 2020)
3. carbon nano tubes (Frei et al., 2021)
4. dense particle mixtures (Frei et al., 2022)



While the data-driven design of deep learning architectures is key to their effectiveness, it is also their major drawback, since it requires large data sets of annotated images, which are typically not available. Hence, we also examined ways to alleviate this drawback using image synthesis.

Acknowledgement

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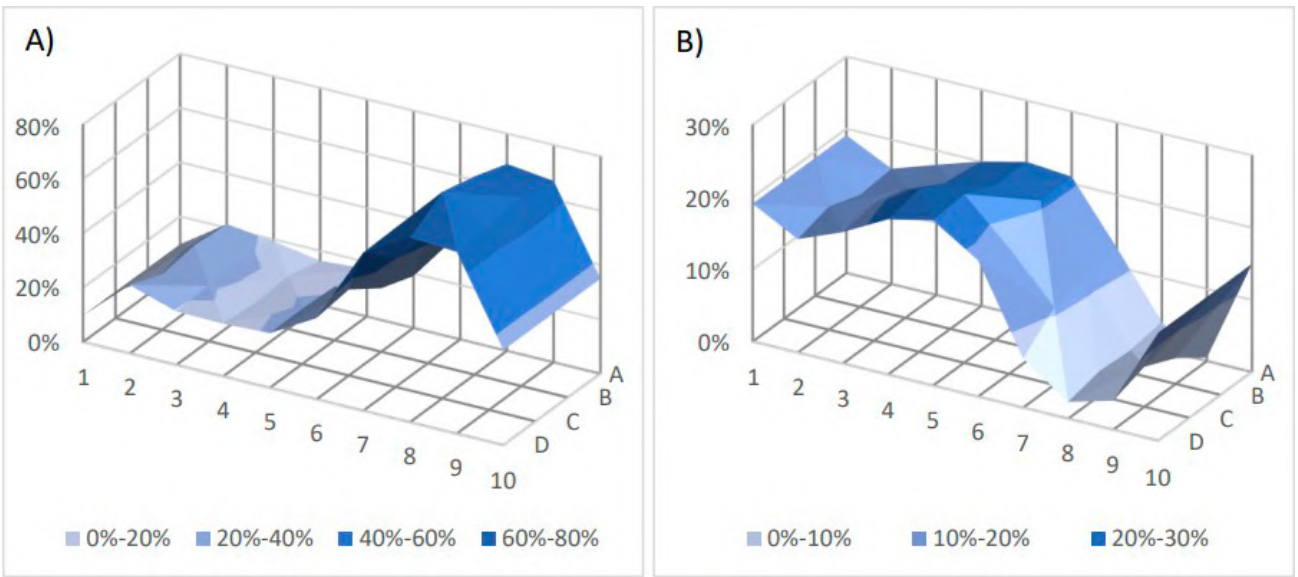
Development and application of a novel sampling funnel for continuous in-line

particle size measurement in dry granulation by roller compaction

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Keywords | Dry Granulation, Roller Compaction, In-line Measurement, Particle Size, Representative Sampling

In the dry granulation by roller compaction process two counter rotating rollers are used to compress a powder into a dense, compact ribbon. In a second, subsequent step, the ribbon is milled down using a sieve mill. This process is continuous by nature and produces granules with a defined particle size distribution (PSD), raising the need for in-line PSD monitoring to ensure continuous quality control.



This work aims to provide a method for representative in-line sampling and PSD measurementin dry granulation. A 4 by 10 testing tube grid was placed directly at the sieve of the milling unit of an Alexanderwerk WP 120 Pharma roller compactor. The PSD of each individual samplewas measured by sieve analysis. The measured sieve fractions were plotted in map diagramsaccording to their position under the mill, see Figure 1. It was found, that the PSD varies greatlydepending on the position along sieve length (row 1 to 10) but is constant along the width of the sieve mill (column A to D). A novel sampling funnel was developed which integrates the sample along the sieve length.

Figure 1: Dependency of different sieve fractions in a PSD on the position under the sieve mill in a roller compactor. A) sieve fraction <0.20 mm, B) sieve fraction 0.40 to 0.63 mm.

The novel sampling funnel was used to take a representative sample for in-line PSD measurement with an in-line particle probe using spatial filter velocimetry. It was possible to measure the PSD in-line and to track changes of the PSD with changing specific compaction force of the roller compactor. The representative in-line measurement was validated using at□line sieve analysis of the complete granule stream.

Characterizing the milling of nanomaterials using differing on / in / at line process analytical technologies

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Keywords | PAT, Nanoparticles, milling

Many of the materials we use are created by milling. Application areas are vast - food, pharmaceuticals, coatings and catalytic material are just a few examples. However milling process is vastly expensive in both energy and time, and in materials costs if material need reworking. Modern sustainable approaches need to consider making more efficient but the process owner needs to know when to stop and waiting for a laboratory to give a green light that a material is small enough is an expensive luxury, getting that information quickly and robustly

has never been more important. Characterisation techniques have been used at-line / on line for many years but mainly in the micron area, the nano material area has not seen the same attention. The EU funded Horizon 2020 PAT4Nano project has been undertaken to develop, compare and validate the performance of materials in this area.

This paper will discuss the use of on/at line material characterisation equipment on a mill showing the process can be tracked from small micron starter materials into the nanomaterial area in an automated manner. A laboratory scale mill was used, and samples were automatically sampled throughout the milling process. Principally laser diffraction and dynamic light scattering techniques were used in this study. Samples used in this study come from the end user companies involved in the consortia and include materials such as pharmaceuticals, catalytic materials and pigments. The results obtained with different technologies will be contrasted and the ability of such technologies to track the milling process and the comparability with traditional laboratory results will be demonstrated.

2D and 3D Structural Characterization of Coated Particles

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Keywords | dry particle coating, mechano-fusion, particle characterization

The process of dry particle coating is used to create composite particles also called hetero-aggregates with tailored characteristics and specialized functionality. While wet coating processes are now well understood and can be modified through a good knowledge of DLVO interactions between particles, there is still a lack in precisely describing prevailing forces and micro processes during dry particle coating, such as de-agglomeration of feed materials, re-arrangement or re- agglomeration. More than ever, the characterization of dry coating processes is important as new applications of dry coated particles arise (e.g. fabrication of functionalized battery materials (Zheng et al., 2019)).

The dry coating process analyzed in this study is called mechano-fusion (MF). Inside a cylindrical processing chamber, larger host particles (1-200 µm) are brought into contact with (sub-)micron guest particles by using of a rotor. The coating process itself takes place when the material is conveyed into the gap between chamber wall and a piston-like stator. There, high shear and compressive stress act onto the particles which finally leads to the guest particles becoming mechanically attached to the host particles' surfaces (Pfeffer et al., 2001).

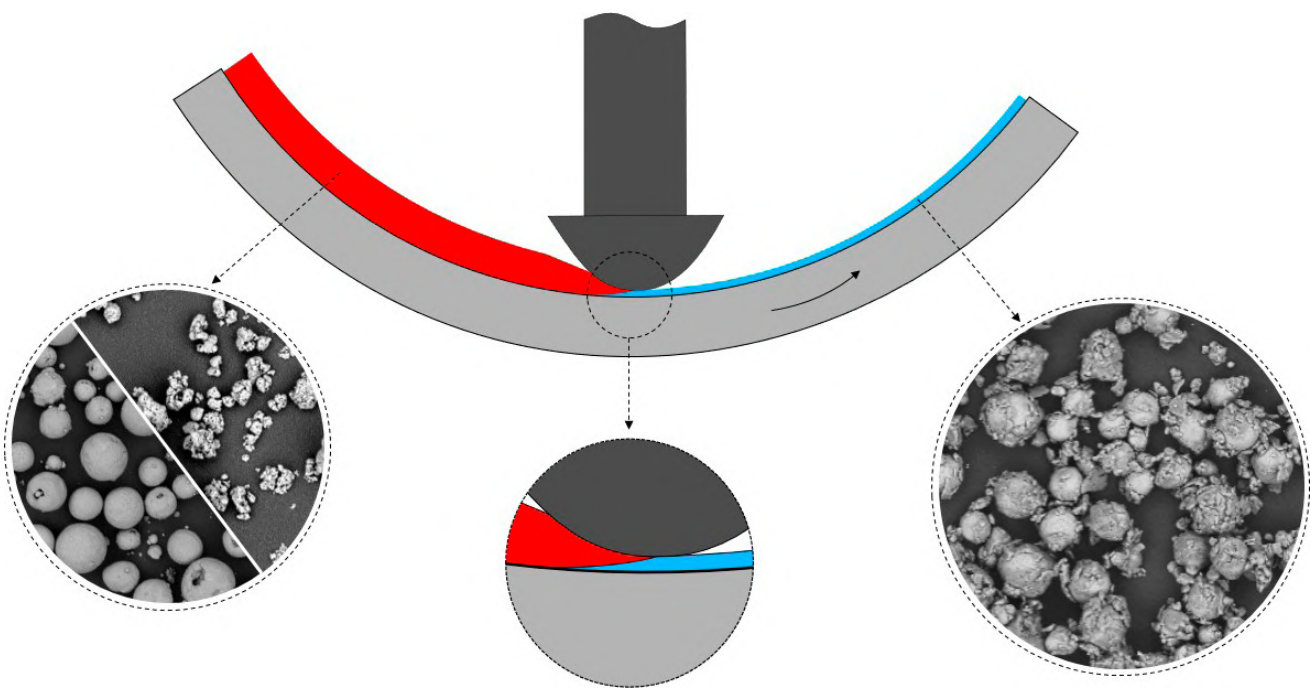


Fig. 1 Scheme of mechano-fusion process

During characterization of the coated particles it is important to find structural descriptors for single particles and even the entire particle system. This in turn allows feedback for the process and feed material parameters. 3D imaging using micro- or nano-computed tomography

(CT) offers the possibility of gaining an insight in the structures of coated particles without a stereological error (Ditscherlein et al., 2020). Due to the limitation in achievable voxel size of the CT, 2D imaging techniques are required for higher resolution analyses. First, scanning electron microscopy (SEM) images are taken of a sample after MF to get an impression of sample homogeneity and degree of coating. Then, particulate samples are embedded in a stable epoxy-based matrix to perform CT analysis. Polished sections from the embedded samples will additionally be analyzed using the Atomic Force Microscope (AFM).

In this regard, the presentation will showcase methodology and first results of the structural analysis of coated particles using the above-mentioned techniques.

Inline measurement of the fouling potential of reverse osmosis feed water using Dynamic Extinction Spectroscopy

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Keywords | Membrane fouling, Lambert-Beer, Fouling index, Clean water, Inline measurement

The demand for pure and drinking water is steadily increasing, which shifts the focus of the current research in water desalination to more energy efficient and economic solutions [1]. Although reverse osmosis (RO) desalination plants have a field leading position, membrane fouling remains one of the most challenging topics [2,3]. Colloidal foulants cause a loss in plant performance and RO-Module lifetime [3] since fine particles ranged between 100 nm and 2 µm aren't affected by hydrodynamic (crossflow) – or diffusive effects and deposit more likely on the membrane [4]. Colloidal particles are irreversible foulants and cause permanent membrane damage [5], even before permeate flux declines [2]. Since colloids pass broad sections of the pretreatment process [5], interest in feed water monitoring is justified.

Inline measurement

An optional method to characterize the fouling potential of RO feed waters is to measure the extinction of different monochromatic light rays. Preliminary studies at our institute showed dependencies between particle concentration, mean particle diameter and Silt-Density-Index (SDI) of nanoparticles below 1 µm. The particle concentration is evaluated by the Lambert-Beer law, while the mean particle diameter is calculated by Dynamic-Extinction-Spectroscopy (DES) as shown in the following figure. Particles below 1 µm show a specific extinction ratio with different wavelengths of monochromatic light.

Particles above 1 µm are detected by the analysis of the fluctuation of the extinction value by the Statistical-Extinction (SE-) Method [6].

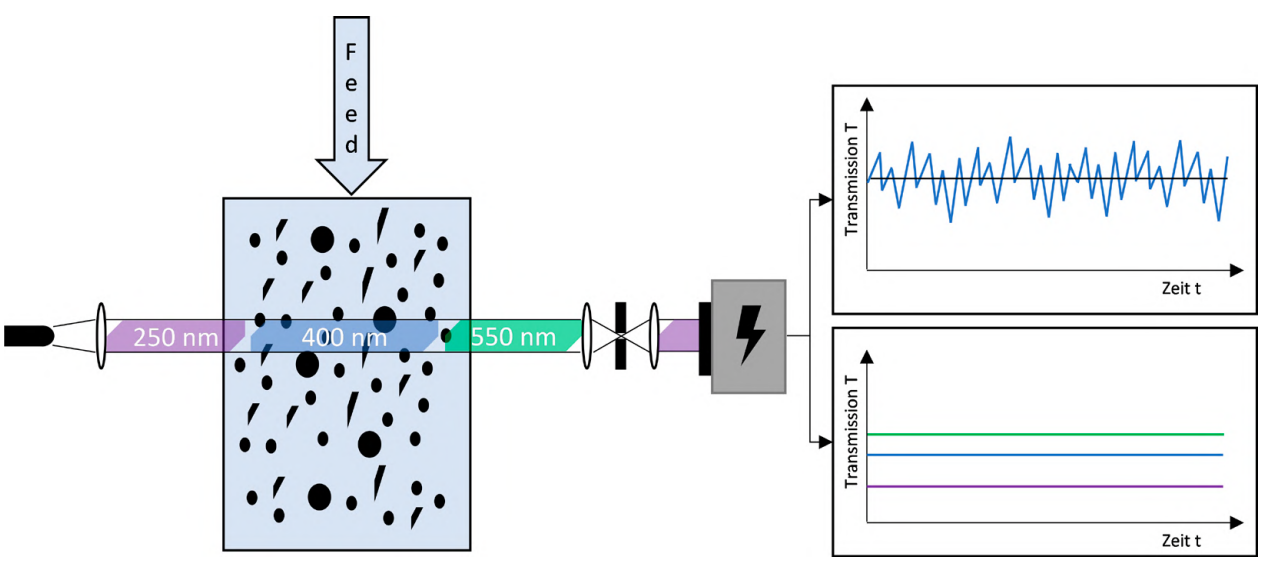


Figure: DES measurement principle

Results and possible advancements in RO-Desalination

A set of measurements for simultaneous reading of SDI/Modified Fouling Index (MFI) and DES measurements of feedwater suspensions with various concentrations and particle sizes will be presented.

The implementation of the presented inline fouling potential measurement with an advanced system control can trigger suitable precautions to prevent membrane damage at an early stage. These include for instance a temporary reduction of the filtrate flux, an increase of the membrane overflow velocity or the adjusted dosage of coagulants.

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Novel Approaches for Multidimensional Particle Property Characterization by Means of Analytical Ultracentrifugation

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Keywords | Multidimensional particle properties, Size, Shape, Density, Optical Properties, Colloids, Analytical Ultracentrifugation, Sedimentation

Multidimensional particle property characterization leading to detailed knowledge about the size, shape, composition, functionalization and optical properties of colloids is key for the understanding of complex multiphase products. We could demonstrate that analytical ultracentrifugation (AUC) equipped with multiwavelength extinction [1] and emission [2] detection is a powerful tool for the simultaneous determination of size, shape, composition and optical properties, one of the greatest challenges in particle science and technology. Our recent developments significantly extend the possibilities of AUC for multidimensional property characterization of complex colloidal systems, such as the size-bandgap relation of semiconductor quantum dots [2], the size and shape distribution of plasmonic nanorods [3] or the simultaneous characterization of size and composition of noble metal alloy nanoparticles as shown in Figure 1.

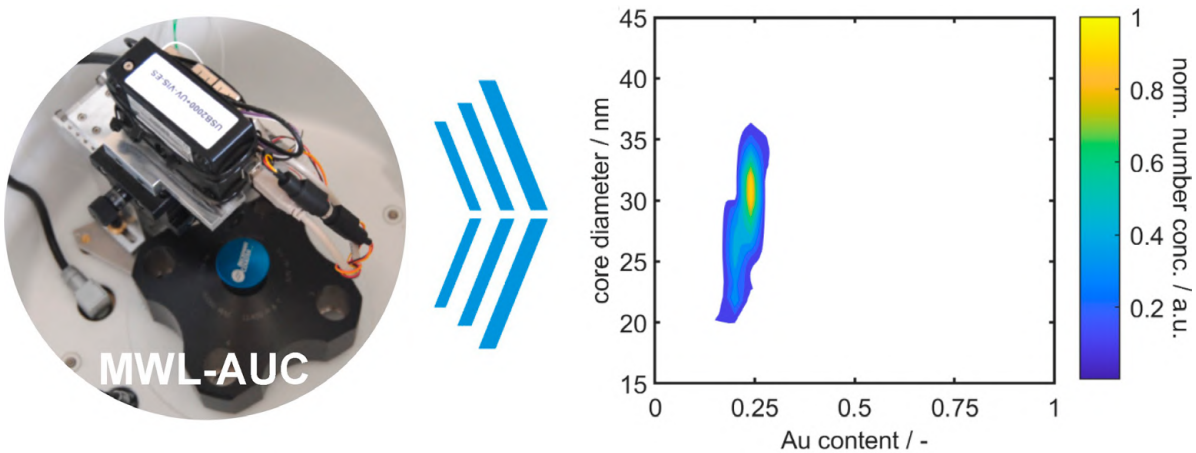


Figure 1: Schematic workflow for the application of AUC equipped with a multiwavelength extinction detector (MWL-AUC) to derive the 2-dimensional size-composition distribution of gold-silver noble metal nanoparticles (publication in preparation).

Moreover, AUC can be used to derive the thickness and coverage of silver metal patches coated onto silica core particles [4] or to study the dynamic interaction between polystyrene or silica cores and microgels [5]. Recently, we investigated the sedimentation non-ideality of silica and polystyrene particles as a function of the particle concentration and probed in this way the extent of electrostatic interactions.[6]

Our contribution will highlight the wide-ranging potential of sedimentation analytics for multidimensional characterization of complex particulate systems. The direct correlation of the size, shape, density and optical properties of particles is of manifold importance for particle technology as knowledge-driven product design comes into reach.

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FLASH COMMUNICATIONS

Mass transfer in fountain confined conical spouted beds: ascertaining the effect of operating conditions and geometric factors of the internal devices

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Keywords | Mass transfer, Fountain confiner, Conical spouted beds, Modelling and simulation

The design of industrial scale gas-solid contact dryers, as those based on spouted beds, requires ascertaining the hydrodynamics and heat and mass transfer mechanisms. Accordingly, several correlations have been proposed in the literature for calculating the minimum spouting velocity, operating pressure drop, particle cycle time and heat (Saldarriaga et al.; 2016; Yaman et al.; 2019) and mass transfer coefficients (Szafran and Kmiec; 2004). However, the correlations for estimating the mass transfer coefficient are not suitable for fountain confined conical spouted beds.

Thus, a previously validated model is used for calculating the mass transfer coefficient in batch drying of fine sand in a fountain confined conical spouted bed dryer (Pablos et al.; 2020). Drying runs have been carried out varying the inlet air velocity and temperature, and the geometric factors of the internal devices, namely, draft tube and fountain confiner. Fine sand with a Sauter mean diameter of 194 μm micrometre was used in the drying runs.

Overall, the mass transfer coefficient is sensitive to both the inlet air temperature and velocity. Thus, its value decreases as the inlet air temperature is increased and the air velocity is decreased. This is mainly explained by the turbulence attained in the dryer. Although the influence of the geometric factors of the internal devices is less significant than that of the operation conditions, it has also been analysed due to their impact on the system hydrodynamics and stable operation. The length of the fountain confiner is the most influential factor on the mass transfer coefficient. Accordingly, long fountain confiners enhance the gas-solid contact, as the gas residence time is increased. Furthermore, concerning the design of the draft tubes, the lowest value of the mass transfer coefficient is attained when operating with a non-porous draft tube, whereas the maximum value is attained when operating without draft tube. This is mainly explained by the role played by both particle circulation and spout diameter.

Therefore, both operating conditions and geometric factors of the internal devices should be considered for designing and scaling-up spouted bed dryers, as they significantly affect in the drying mass transfer mechanism.

Tracking the order of mixing effects in ternary adhesive mixtures using a coloured tracer

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Keywords | Powder mixing, adhesive mixtures, dry coating, order of addition, coloured tracer, Inverse gas chromatography

A quick and effective assessment of the quality of the mixture is of important to ensure content uniformity, powder flow etc., across several

powder processing industries. Recently, dry powder coating technology, comprising of guest-host particle interactions, has been explored to produce uniform adhesive mixtures (Karde & Ghoroi, 2014). Specific powder mixing conditions like the order of addition, energy input and particle properties (Khala et al., 2021; Shah et al., 2017) are critical to achieving these effects. The objective of this work is to use a coloured tracer for tracking the influence of the order of addition and analyse the mixing performance in the ternary powder blends comprising of guest and host components.

To investigate the order of mixing effects, blends were produced by mixing coloured tracer (iron oxide nanoparticle, IONP) and nanosilica as the guest components with lactose monohydrate as the host particle mixed at 330 rpm speed for 20 min in an overhead stirrer set-up. These powder blends were analysed for their mixing characteristics through visual imaging and colorimetric estimations. Several surface analytical techniques like scanning electron microscope (SEM) for surface area coverage (SAC) determination, energy dispersive X-ray spectroscopy (EDS) for surface elemental mapping and Finite Dilution Inverse Gas chromatography (FD-IGC) for surface energy heterogeneity characterisation were used.

The results show that for the same powder composition (96% w/w lactose, 2%w/w IONP, 2% w/w nanosilica), the sequence of addition of guest particle drastically influence the mixture appearance (colour) (fig.1a). This was corroborated using the surface energy heterogeneity results, where the surface energy trend moved towards the component added last (fig.1b.). In competitive scenario, where both the guest particles were added together, nanosilica was found to be the dominant component coating the lactose surface. Overall, from these results, the importance of the order of addition of individual powder components during mixing was highlighted through imaging, colorimetry measurements and FD-IGC analysis.

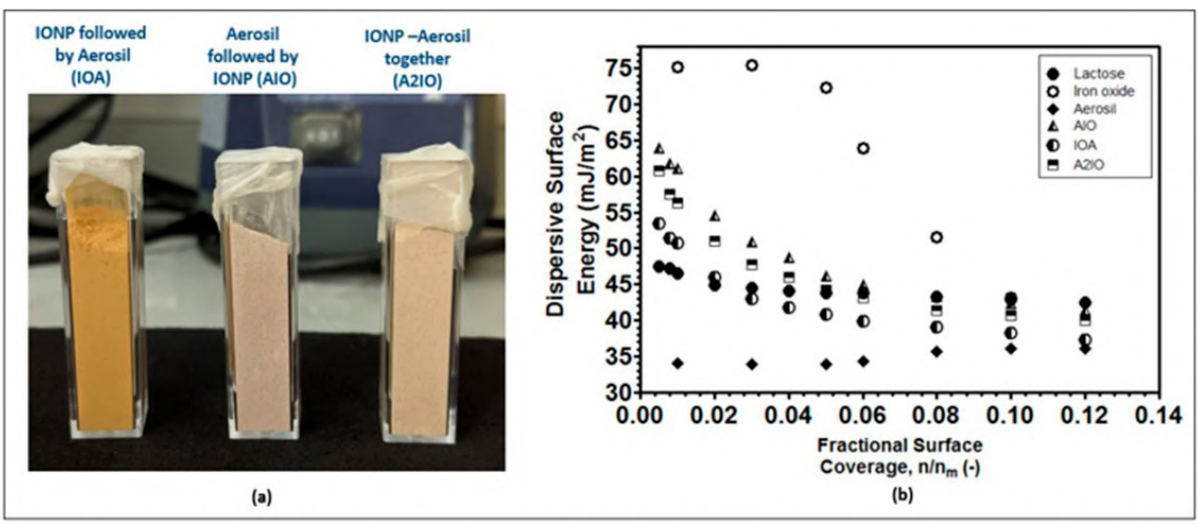


Fig. 1. Ternary powder blends showing (a) variation in appearance for the same composition and (b) surface energy heterogeneity of these mixtures along with the individual components

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Drug Particles in Polymeric Extrudates: Size and Orientation Studies via Scanning Raman-Microscopy

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Keywords | Scanning-Raman-Microscopy, Drug Particles, Composite, Hot-Melt-Extrusion

Holt melt extrusion (HME) is a commonly used production technique in pharmaceutical industry and research (Crowley et al., 2007). During the process, a thermoplastic carriers such as water-soluble polymers or lipids are compounded with an active pharmaceutical ingredient (API) either to form a molecular, amorphous solid solution or a dispersedly loaded composite. The API particles in such latter composites

can be challenging for the extrusion process itself (Witzleb et al., 2011) and downstream processes like 3D printing of individualized dosage forms (Tidau et al., 2019). Due to these challenges, it would be favorable to determine the particle size distribution and orientation inside of the filaments produced by HME. Unfortunately, APIs are not only typically partially soluble in the polymeric matrix, but also soluble in the solvents the polymers are soluble in, making a particle size analysis via redispersion impossible. Thus, we developed a novel method for determining both, particle size distribution and orientation simultaneously by applying scanning Raman-microscopy.

Therewith, we are able to inspect extruded filaments with different API loads.

For the measurements, a confocal Raman microscope (alpha300 R, WITec GmbH, Ulm, Germany) was applied in area scan mode with simultaneous surface tracking. The longitudinal- and crosscut sections of several filaments with different API loads were investigated and evaluated towards the Feretmax drug particle size distribution and orientation relative to the extrusion direction. In comparison to these data, the raw API particles were analyzed applying dynamic image analysis (QICPIC, Sympatec GmbH, Clausthal-Zellerfeld, Germany).

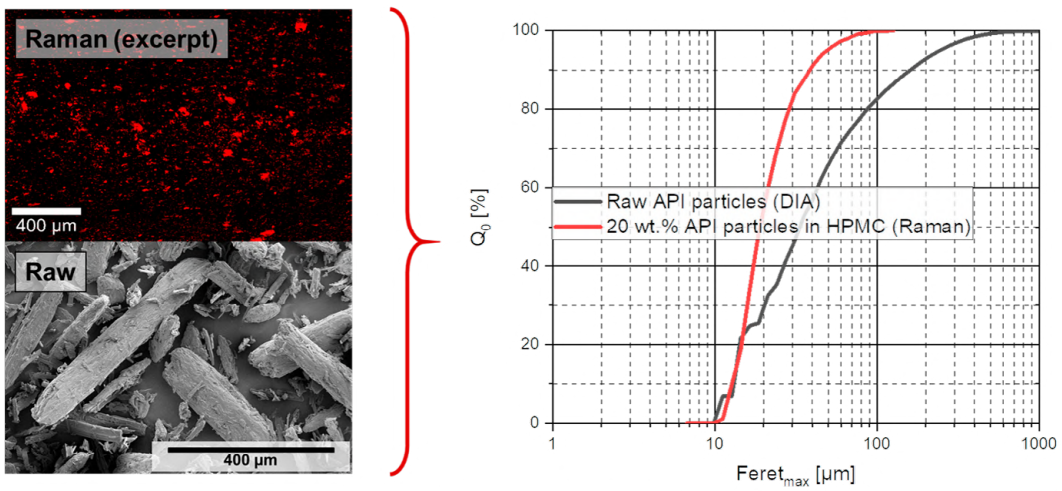


Figure 1: *Left: Raman heat map of API distribution in a longitudinal cut section of HME filament with 20 wt.% drug and SEM-Picture of raw drug particles; right: Particle size distributions of particles in the pictures on the left side*

Applying this method, different effects can be observed. For example, a significant reduction in particle size of needle- shaped Theophylline (e.g. Figure 1), the shape and their orientation of those particles towards the extrusion direction, and the effect of different particle loadings on those effects can be analyzed.

Therefore, accompanied with data from rheological studies and 3D prints, we gained a deeper process understanding.

Volume and number average sizes of Mg(OH)2 particles from concentrated Mg2+ containing solutions

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Keywords | Magnesium hydroxide, Nanoparticles, Mixing, Precipitation, Reactive crystallization, Mineral recovery

Magnesium hydroxide, Mg(OH)2, is a chemical compound widely employed in several industrial fields. In recent years, Mg(OH)2 has been successfully extracted via precipitation from highly concentrated Mg2+ sources, i.e. saltworks bitterns. The precipitation is characterized by different complex phenomena, e.g. extremely fast reactions, reactants mixing, particles nucleation and growth. Among the others, mixing plays a crucial role in the process: although the characteristics of precipitated particles strongly depend on mixing, insufficient information on this phenomenon is available in the literature.

The present work aims at filling this gap by reporting a detailed experimental investigation on Mg(OH)2 particles produced under different reacting conditions. Mg(OH)2 precipitation tests were carried out by mixing 1M MgCl2 artificial solutions with stoichiometric NaOH ones.

Two 2 and 3 mm diameter circular cross-sectional T-shaped mixers were employed to tune reactant mixing at Reynolds numbers ranging between 2000 and 38,000 corresponding to mixing times between 60 and 1.4 ms (Romano et al., 2021). Mg(OH)2 particles were characterized employing a static light scattering Mastersizer2000 analyser with and without ultrasounds treatment and poly(acrylic acid sodium salt).

The obtained characteristics volume (d43) and number (d10) average sizes reduce from ~5 μm to ~0.14 μm and from ~0.5 to ~0.07 μm, respectively. No particle size variation is observed for mixing time varying from 2 to 1.4 ms, implying that mixing is not affecting the precipitation process anymore.

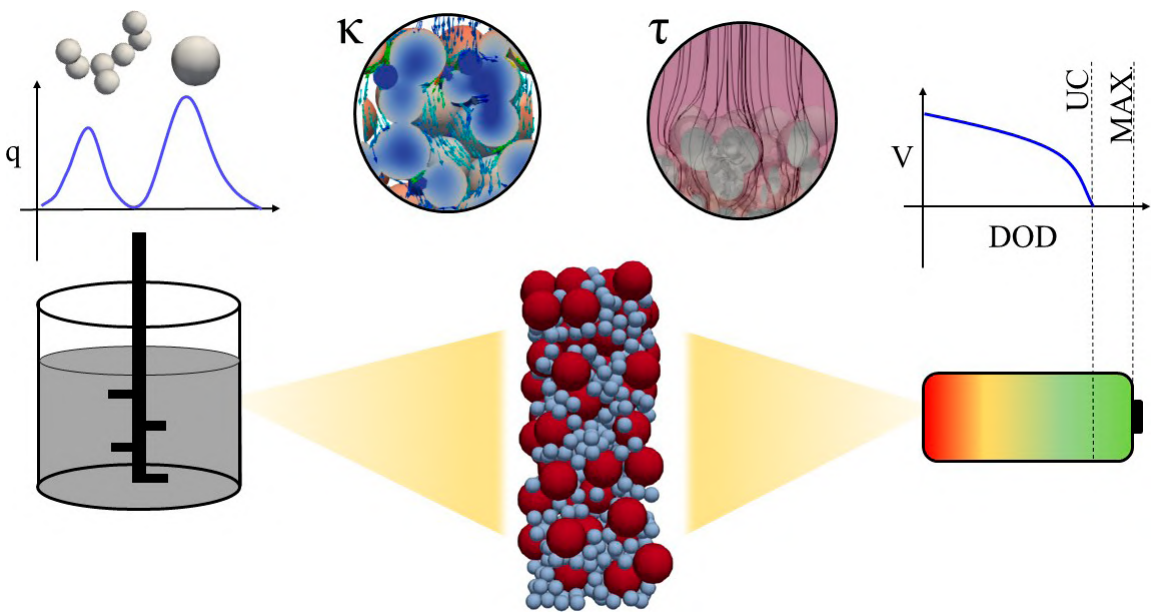
This project has received funding from the European Union's Horizon 2020 research and innovation programme under Grant Agreement No. 869467 (SEArcularMINE). This output reflects only the author's view. The European Health and Digital Executive Agency (HaDEA) and the European Commission cannot be held responsible for any use that may be made of the information contained therein.

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Influence of Carbon Binder Domain on the Performance of Lithium-Ion Batteries: Impact of Size and Fractal Dimension

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Keywords | carbon binder domain, cathode microstructure, cell performance, conductivity, distribution, particle size, fractal dimension



A lithium-ion battery (LIB) cathode comprises three major components: active material, electrical conductivity additive, and binder. The combination of binder and electrical conductivity additive leads to the formation of composite-clusters known as the carbon binder domain (CBD) clusters. Preparation of a LIB cathode strongly influences the dispersion of the above- mentioned constituents leading to the formation of distinct pore and electrical conduction networks. The resulting structure thus governs the performance of LIBs. The presence of CBD is essential for the structural integrity and sufficient electrical conductivity of the LIB cathode. However, CBD abundance in LIB cathodes leads to unfavourable gravimetrical and volumetrical consequences owing to its electrochemical inertness. Increasing CBD content adds to the weight of the LIBs, thus negatively impacting the energy density. Furthermore, increased electrical conductivity is won at a cost of ionic conductivity as CBD clusters breach the pore networks in the cathode microstructure.

The following study establishes a link between the various possibilities of CBD cluster size and fractal dimension that may eventualize

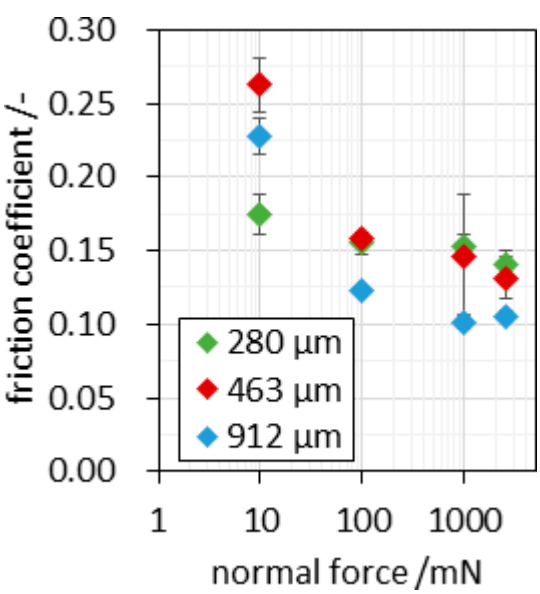
during slurry preparation to the resulting microstructural properties and hence to the performance of LIBs by means of idealized cathode geometries. Since the performance determining processes occur at the microstructural scale, which are often very tedious to study via experimental research, the study makes use of spatially resolving microstructural, numerical, simulations. The results demonstrate that the CBD cluster size has a strong influence on the cathode microstructure. The CBD cluster fractal dimension on the other hand displayed a minor influence on the structural properties of the cathode and the size of the primary particles was shown to be the dominant factor. Finally, performance evaluation simulations confirmed the trends seen in structural properties with changing cluster size and fractal dimension.

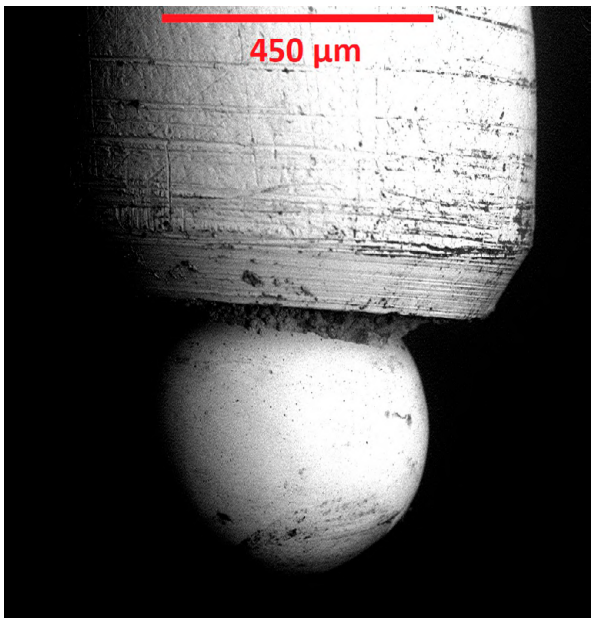
Sliding friction measurements of single particles at different contact forces

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(1) Technische Universität Kaiserslautern

The Discrete Element Method (DEM) is widely used to simulate granular processes to understand the influence of micro processes, such a friction, on the flow behaviour. The parameters of the contact models used in the DEM have to be calibrated with experiments to accurately describe the granular processes. Besides the coefficient of restitution, which can be determined by free fall tests, the sliding friction coefficient is an important parameter for these contact models. Typically, this parameter is derived from experiments in a shear cell. The resulted tangential forces are measured for given normal forces to calculate the friction coefficient. With this method, an average friction coefficient is calculated independent of the normal forces. However, the applied contact forces are often varying from forces in real processes and do not describe single particle - wall interactions correctly.
In this study the particle-wall friction coefficient for single micro particles at varying forces is investigated. Therefore, tribology measurements were performed with a triboindenter (TI Premier, Hysitron). The spherical single zirconium oxide particles with different diameters are glued on an indenter tip and sheared on a stainless-steel substrate. The friction coefficient was determined at normal forces between 10 mN and 2.5 N (Figure 1). Besides the variation of the contact force, the particle velocity was also varied to identify the influence of the velocity on the friction coefficient for single particles.

Additionally, oblique particle collisions between 20° and 70° were investigated with free fall experiments at low collision velocities (< 1 m/s) with the comparable range of forces occurring during the collision. The dynamic friction coefficient was calculated with these experiments by using the rigid body theory for collisions. The results of the different approaches show that the friction coefficient depends not only on the particle-wall materials but also on varying contact forces as a result of different particle velocities during particle collision as well as the roughness which was measured for the particles and substrate.





Influence of relative humidity on localised caking behaviour in food powders

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(1) Freeman Technology

Keywords | Caking, Powder Caking, Powder Testing

During extended storage of powders, particular and bulk properties can change, leading to downstream processing issues. Caking occurs when the powder bed gains strength as a result of sustained particle-particle interactions.

Caking can occur by one or more mechanism, the most common being the absorption and /or migration of moisture. In addition to capillary bond formation, moisture may activate chemical reactions between surface groups and /or plasticise the powder particles, leading to mechanical deformation. As moisture is typically absorbed at the powder-air interface, and then migrates, interactions may not occur uniformly throughout the powder bed, leading to non-homogenous caking.

In this study, the influence of relative humidity on three food mixtures was evaluated using a FT4 Powder Rheometer®, (Freeman Technology, Tewkesbury, UK). As the rheometer blade moves through the powder bed, flow energy is recorded as a function of bed height, quantifying localised variations in the powder bed. Samples were stored for between 1-4 days at 75%RH, and the flow energy was measured at 24 hours intervals. In order to simulate variations in storage conditions, the powders were also subjected to alternating humidity conditions, in which the samples were stored at 75%RH for 3 days and then return to ambient conditions for 3 days.

The results demonstrated clear progression of the caked region over time but distribution of the caked region could also be seen to vary between materials. Sample 1 presented clear demarcation between the caked non-caked region regions, whereas Sample 2 exhibited greater resistance to flow throughout the powder but with increased resistance at the powder- air interface. Alternating the humidity further demonstrated the complexity of caking behaviour, with samples exhibiting different responses to decreasing humidity, including one that continued to exhibited an increase in flow energy.

Overall, this study demonstrates that powders exhibit complex and varied caking behaviour and evaluating a powder's propensity to cake cannot be restricted to a simple test, rather a comprehensive methodology, capable of providing precise, localised measurements, is required.

Characterization of metal powder for additive manufacturing process

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An in-depth understanding of the bulk behavior of particles based on their individual properties is a vital step for the powder handling industries, a good example is the selection of appropriate powder material and their flow consistency in additive manufacturing process which would have significant effects on the quality of the final products. Identification of the most reliable method to characterise powder flow behaviour in correlation to the conditions of powder spreading is still challenging. Therefore, the objective of this study is to attain the most trustworthy characterisation method in correlation to the condition of spreading in additive manufacturing. For instance, the low consolidation state of the powders within the process requires a characterisation technique which is capable of measurement for such conditions. In this study, a few standard and advanced flowability techniques such as hall flowmeter, Hausner ratio, compressibility, shear cell, FT4, and ball indentation were used to measure the flowability and compared the outcomes with results of "Spreadability" with focused on different shapes and surface properties of Ti6Al4V material. The results have led to a better understanding of different available flowability techniques and correlation of the best technique with powder bed-based additive manufacturing.

The authors would like to acknowledge financial support from EPSRC-UK Future Manufacturing Hub in Manufacture using Advanced Powder Processes (MAPP) (EP/P006566/1, www.mapp.ac.uk).

Polyamide Powder Rejuvenation for 3D Printing

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Keywords | Additive Manufacturing, Powder Rejuvenation, Powder Recycling, Powder Flow

In Additive Manufacturing (AM) Selective Laser Sintering (SLS), actual printed parts typically account for 10-20% by volume of the powder deposited during the build process. The remaining 80%-90% loose powder must be recovered and reused in order to make the process economically viable. Continued reuse can affect both the chemical and physical properties of powder resulting in built parts that don't meet specification or require uneconomical virgin-to-used powder ratios.

A rejuvenation process can restore the functionality of used feedstocks, enabling direct reuse without addition of virgin material. However, it is not suitable for all feedstocks as it is dependent on the chemical and physical characterisation of the used powder

In this study , several Polyamide 12 (PA12) powders, consisting of both "as received" and rejuvenated powders were evaluated using an FT4 Powder Rheometer® (Freeman Technology Ltd, UK). The aim was to investigate the relationship between flow behaviour and rejuvenation process.

The results demonstrated that successful rejuvenation resulted in improved flowability, with lower Aerated Energy and Compressibility values. In contrast a failed application of rejuvenation resulted in an increase in AE.

Furthermove, the aeration behavour could be used to indentify suitble candiates for rejuvenation, with samples that are more sensitive to the effects of aeration, especially in combination with relatively low compressibility, exhbiting unsucessful rejuvenaton.

Overall the results demonstrate how multifaceted powder flow assessment can be used to determine suitable candidates for rejuvenation, as well as providing conformation of sucessful powder rejuvenaton without the the need for potentially expensive and time consuming print trials.

Investigating the polymeric powder spreading properties at various temperatures for selective laser sintering

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Keywords | Selective Laser Sintering, High Temperature, Powder flowability, Spreadability, Wavelet Transform

Selective laser sintering is one common method of additive manufacturing that allows the production of 3-dimensional objects by adding powder layers. The final product structure and properties strongly depend on the powder layers quality. Powder delivering and spreading steps in selective laser sintering (SLS) play an essential role in preparing a uniform powder bed in each process run.

The powder spreadability would be impacted by the particle size, the particle shape, the forces between the particles, the humidity, and the temperature. The temperature can more effectively control the residual stresses and the consequent formation of cracks in more focus. In addition, it can improve the final artifact density due to the better wettability of the fused phase with the neighboring particles. Furthermore, heating the camber of sintering can reduce the high-power laser demands and control the thermal gradient. However, in the worst-case scenario, powder preheating makes the powder flowability poor and negatively affects the powder spreadability.

In this study, the spreadability of four polymeric powders including Polyamide 6, Black-Polyamide 6, Polypropylene, and Thermoplastic polyurethane was investigated by the spreading apparatus designed at the Powder Technology laboratory of the University of Salerno at different temperatures. In particular, this apparatus can run the spreading step similar to the commercial machines utilizing a heating and temperature control system at any fixed temperature value. Furthermore, a method was proposed to evaluate powder layer quality from images taken from the powder bed at the desired temperature in the presence of surface grazing light. The procedure is based on Wavelet Transform. Moreover, this procedure can identify the characteristic size of surface roughness compared to the agglomerate size obtained through the analysis of the shear testing results.

Advances in Dynamic Light Scattering

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Keywords | Nanoparticles Characterization, Size Characterization, Nanoparticle Number Concentration, Dynamic Light Scattering, Multi-angle Dynamic Light Scattering, Adaptive Correlation, Capillary Dynamic Light Scattering

Nanoparticles can have exciting properties especially when compared to their bulk counterparts. Some types of nanoparticles are well known for their size-dependent optical properties, but their size can also impact their mechanical, electronic, or biological functions. So, as researchers reduce the material size scale to solve novel (or old) problems, so we become more reliant on more accurate particle sizing techniques.

Dynamic Light Scattering (DLS) is a ubiquitous and non-invasive measurement for the characterization of nano- and micro- scale particles in dispersion. It is a well-established size characterization tool and arguably the starting point for most nanoparticle-based formulation. However, there are some criticisms usually pointed at DLS instruments: the influence of dust (or any other much larger contaminants) on the data quality even when in minute quantities, the variability of results in the upper size scale limit, and the low resolution and angular dependency of DLS data which can lead to slightly different results when using different instruments.

In this talk, some of the latest advances in DLS will be explored with reference to varied application areas. More specifically:

1. Adaptive correlation presents a new statistical approach to assess the statistical relevance of every sub run collected during the measurement. (Malm et al., 2019)
2. Capillary DLS reduces convection current influence in the Brownian motion of particles, allowing for a more accurate size determination of slow-diffusion particles. (Ruseva et al., 2018)
3. Multi-angle DLS reduces the restraints imposed by single angle correlation, by providing a higher resolution multi- variate analysis. Additionally, the higher resolution allows for the calculation of nanoparticle number concentration. (Austin et al., 2020)

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3 PARTICLE PROCESSING

KEYNOTE LECTURES

Prediction of slurry-particle flow within a SAG mill using an efficient and scalable DEM-SPH fully-coupled approach

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Keywords | Keywords: SPH, DEM, multi-GPU, Slurry Mill

Most autogenous (AG) and semi-autogenous (SAG) mills operate as wet grinding processes with the addition of a reasonably constant ore-to-water ratio to improve the transport of fine material and suppress dust. As the comminution process takes place, overly fine fragments are generated and mixed with water to form a slurry, affecting not only the dynamics and grinding behavior of the mill, but ultimately impacting the mill performance and power draw.

Numerical modeling brings an interesting way to predict the behavior of slurry mills, adding to the understanding of the influence of the operational conditions and design variables on the grinding process. The Discrete Element Method (DEM) is a well established method to model breakage, particle-particle interactions, as well as particle-equipment interactions in comminution applications. The existence of the slurry phase, however, poses a complex challenge as the influence of the liquid phase on the coarse particle motion has to be included in the computational model. Tracking the slurry's complex free surface is very burdensome for Eulerian grid-based flow solvers that rely on the continuum approach, as they tend to fail to capture the correct interface profile. Moreover, slurry-particle interaction is usually accounted for by means of empiric correlations that lack robustness when dealing with complex particle shapes, and are also highly dependent on the CFD mesh cell size to particle size ratio, which makes it difficult to handle large particle size distributions.

Smoothed Particle Hydrodynamics (SPH) provides a particle-based method to model complex free-surface flows in mills with good accuracy, and its explicit formulation makes it suitable for massive parallelization on GPU-accelerated solvers. This study shows how the combination of DEM and SPH methods can be efficiently applied to accurately capture both particle and slurry flow dynamics in mills, accounting for the particle-fluid interactions. The coupled model can be used to assess the influence of the operational conditions as well as the mill and discharge design parameters on the charge flow and to help optimize designs for improved performance and wear life. The numerical results are compared with benchmark data, and the simulation performance and scalability are also discussed.

Modeling the Impact of Ball Size–Material on the Fineness of Cement Clinker Particles Prepared by Dry Ball Milling

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Keywords | Ball milling, Population Balance Modeling (PBM), Dry milling ball size, Cement industry

In cement industry, one possible way for cost reduction is to replace traditional steel grinding balls with different materials, including alumina ceramic, in dry ball milling. The density of a ceramic ball is less than half that of a steel ball, which will result in lower mill power as the mill power is directly proportional to the grinding ball density for a given operating condition. On the other hand, these different ball types could affect breakage kinetics of a cement clinker; hence, the choice of these balls must be assessed holistically. Here, we estimated the breakage parameters of a population balance model (PBM) via a global optimizer-based back-calculation method for a cement clinker milled in a tumbling ball mill and compared the performance of steel vs. ceramic grinding balls. The PBM parameters for each size (20, 30, and 40 mm) of the steel balls and the ceramic balls were then used to predict the timewise evolution of particle size distribution (PSD) for a mixture of different ball sizes (based on uniform mass of different ball sizes). Fig. 1 shows good agreement between the experimental and

predicted evolution of the PSD of the cement clinker when a ball mixture was used, proving good prediction capability of the PBM beyond its fitting capability. The back-calculated specific breakage rates for the individual balls and the mixtures were higher for the steel balls than for the ceramic balls when ball sizes were kept the same. Our Discrete element method (DEM) simulations of the ball mill indicate that for a given ball size, steel balls are associated with a higher collision frequency at the high collision energy levels based on dissipated energy ($1x10^{-3}$ J), while ceramic balls are associated with a much higher collision frequency at the low collision energy levels than the steel balls. Additionally, for a given ball material, collisions of larger balls generate higher collision energy levels than those of smaller balls. These findings explain the origin of faster breakage observed with the steel vs. ceramic balls as well as the higher breakage rates with the larger balls.

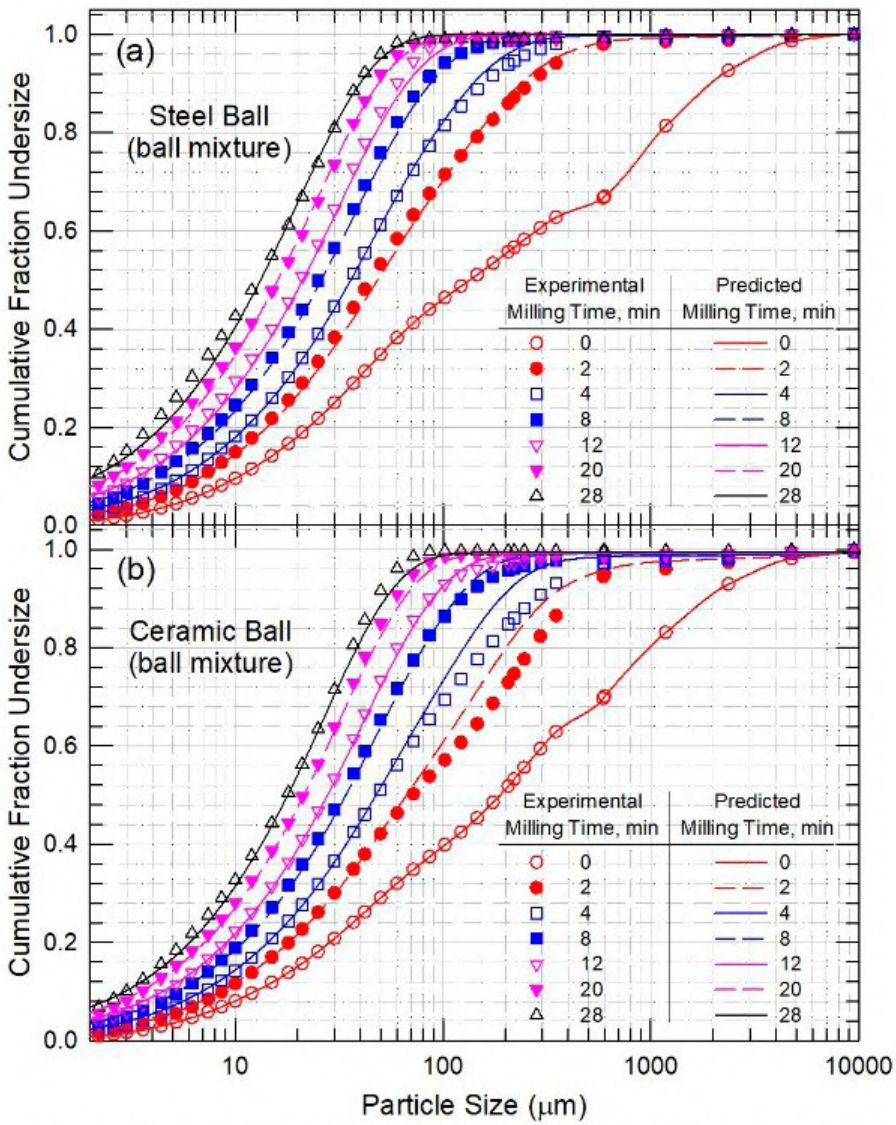


Fig. 1. Comparison of the experimental and predicted temporal evolution of PSDs using a mixture of 20, 30, and 40 mm balls made up of: (a) steel and (b) ceramic.

Challenges in Modelling and Understanding of Particle Formulation by Spray Granulation

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Fluidized bed spray granulation is the process of choice for producing highly functional, high value granular products. The interaction

between the spray liquid and particles are complex and depend on the drying or thermal conditions as well as the properties of the spray and the material system itself.

Due to the high-value and functional nature of fluidized bed spray granulation products, the conservation of product properties during scale-up is of the utmost importance. To be able to consider this, correlations are needed that estimate the influence of the deviations from the local drying conditions present in lab-scale granulators, in which product formulation trials are commonly conducted. In this work, we present a workflow for developing a multidimensional, linear correlation between process conditions and demonstrate its superiority over an one-dimensional, drying-potential approach. The surface roughness of particles obtained under varying conditions in a lab-scale granulator is correlated to the process conditions and the resulting multidimensional, linear correlation is rigorously analyzed for the importance and co-linearities of the individual process parameters using a principal component analysis. In addition, CFD-DEM simulations were performed for different apparatus geometries and process conditions showing the influence of these parameters on the liquid distribution in the system (Fig). These simulations are supplemented with simulations about the hydrodynamics and moisture distribution in a horizontal fluidized bed with four chambers allowing the simulation of a pilot-plant scale process. It was found that apparatus configurations have a significant influence on the dynamic behavior of the whole process. Recently, a novel control concept for bed mass and particle size distribution was integrated resulting in an improved process stability. Experimental and numerical results can be used for developing kernels for our in-house dynamic flowsheet simulation tool Dyssol allowing simulations of property-distributed parameters as occurring in fluidized bed spray granulation.

Figure: a) CFD-DEM simulation of different fluidized bed setups with liquid injection and b) exemplary different product morphologies dependent on the thermal and spraying conditions.

ORAL COMMUNICATIONS

Towards H2 production by pilot-scale preparation of nickel cobalt oxide anodes: Characterization and optimization

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Keywords | Ni-Co-O powder, Spray coating, Plasma treatment, Large-scale electrode, Catalytic performance

Recent years have witnessed a demand for new renewable energy, due to the rapid depletion of conventional sources. Hydrogen (H_2), most abundant element, can be obtained through water electrolysis (Vidales, Choi et al. 2018), a process currently undergoing rapid development (Mo, Kang et al. 2016). Many researchers focus on the development of stable and high-efficient anodes in alkaline electrolyzers (McCrory, Jung et al. 2015), however, the upscaling of these electrodes into pilot-scale fabrication enabling direct property-performance correlations in industrial settings is lacking.

We aim to bridge this gap starting from ink preparation using commercial Ni-Co-O powder, through electrode preparation by spray coating, to catalytic testing. As a first step, the commercial Ni-Co-O micropowders are characterized by transmission electron microscopy as well as energy-dispersive X-ray spectroscopy for size and composition. The well-characterized powder is then dispersed in different solvents, where the dispersions are systematically evaluated utilizing analytical centrifugation and Hansen parameter calculations for their stability. Afterward, selected dispersions will be sprayed on Ni plates/meshes where the effects of surface treatment such as plasma will also be investigated. Highly complementary techniques will be used to characterize the structure, composition, surface topography, and porosity of the electrodes. Finally, catalytic testing of these electrodes will be carried out and correlated to the physicochemical electrode properties. A substantial improvement in the particles could be achieved based on the gathered data on ink behavior, electrode structure, and performance level.

In conclusion, our results pave the way towards large-scale electrode depositions relevant for the chemical industry due to inherently

scalable technologies applied. Due to proper standards and reference processes, catalytic performance can be tuned by optimizing various material properties of the electrodes.

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Application of nanocellulosic materials as adsorbents to remove Ni2+ and Pb2+ from wastewater

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(1) Chemical and Materials Engineering Department (Complutense University of Madrid), (2) ETSIDI, Mechanical and Chemical Eng. And Industrial Design Dept. (Polytechnic University of Madrid)

Keywords | Wastewater treatment, nanocellulose, heavy metals, nickel, lead, adsorption, bacterial cellulose

Nowadays, there is a need of novel and sustainable adsorbents with low environmental impact like nanocelluloses with high selectivity to harmful pollutants such as heavy metals.

In this study, bacterial celluloses (BC), were evaluated as lead (Pb2+) and nickel (Ni2+) adsorbents. The tested concentrations were in the order of the typical levels in industrial effluents (0.1-50 mg/L), as indicated by Kumar et al (2008) and Parvathi et al (2007) for nickel and lead for industrial effluents, respectively. The experiments were developed with 25 mL samples of synthetic waters and different operating conditions were analysed: contact time, adsorbent dosage and pH. BC was synthetized from *K. sucrofermentans* and applied at 1% consistency (dried mass/total suspension mass) (Campano et al, 2018).

According to the obtained results, a high adsorption capacity of both materials was achieved when the BC was added up to equilibrium and the pH was close to neutral (pH 6). Nickel adsorption removal was found to be 57 mg/g, in the order of adsorption capacity found for commercial adsorbents, like activated carbons (Sivakumar et al., 2018). In the case of lead, the interaction between metal ions and the adsorbent surface was effective even when the dosage level was as low as 4 mg dried cellulose/L. The adsorption capacity of lead reached values close to 1300 mg/g. This result overpass most of the previously seen values of adsorption capacity and shows the high selectivity of this material to lead ions. Selectivity of BC to Pb2+ adsorption has been previously identified (Wan et al., 2019; Chen et al., 2008), as seen in table 1.

Table 1. Nickel and lead equilibrium adsorption capacities reported in bibliography by applying bacterial celluloses

Nanocellulose adsorbent	Heavy metal	Adsorption capacity [mg·g-1]	Ref.
BC	Ni^{2+}	57.52 [C_0=50 mg·L-1]	This work
	Pb^{2+}	1280 [C_0=50 mg·L-1]	
BC	Ni^{2+}	1.5 [C_0=50 mg·L-1]	Wan et al. (2019)
	Pb^{2+}	16 [C_0=50 mg·L-1]	
Bacteria-BC composite	Ni^{2+}	4 [C_0=50 mg·L-1]	
	Pb^{2+}	52 [C_0=50 mg·L-1]	
BC	Pb^{2+}	22.56 [C_0=100 mg·L-1]	Chen et al. (2008)
Carboxymethylated BC		60.42 [C_0=100 mg·L-1]	

The main differences which justify the varied results between the BC synthetized in this study and the rest are that this BC is just synthetized and sterilized, while Wan et al (2019) and Chen et al (2008) used non-sterilized and surface carboxymethylated BCs, respectively. These results are indicators of promising applications of these materials for industrial wastewater treatment. The authors wish to acknowledge the financial support from projects RETOPROSOST-2-CM S2018/EMT-4459 and CONFUTURO-ES PID2020-113850RB-C21.

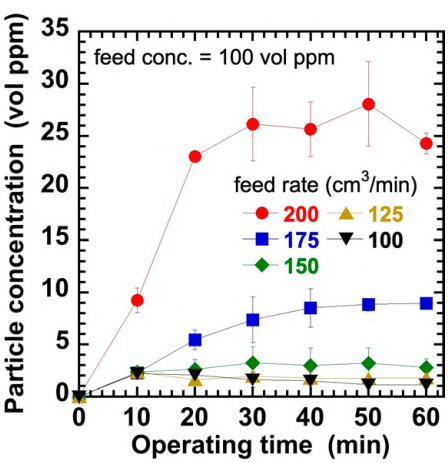
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Continuous particle aggregation and quick sedimentation for aqueous slurries by using DC electric field

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(1) Hosei University

It is desired to quickly correct fine particles in water without any flocculant for material recycling because the purity of the corrected particles becomes higher. Thus, we developed a novel particles aggregation method by applying DC electric field to aqueous slurries instead of adding flocculants [1]. Based on this knowledge, in this paper, we have developed a novel sedimentation separator as shown in Fig.1, in which multi-inclined electrodes (stainless steel plates) are set up in a slurry container. The concentrated slurry and the supernatant can be continuously corrected and their flow rate can be controlled. In this apparatus, the particles aggregation can be enhanced by applying DC electric field and sedimentation separation efficiency can be improved by Boycott effect. We tried to separate sub-micron alumina particles in water continuously. The effects of slurry flow rate and the applied DC voltage on the separation efficiency was investigated. Besides, the effect of the arrangement of multi-inclined electrodes on the separation efficiency was examined as well.



It was shown that the particles were continuously and quickly separated without any flocculant by applying DC electric field. Figure 2 shows the particle concentration in the sampled supernatant. From this figure, it was shown that the clear supernatant without non-aggregated particles could be obtained by tuning the applied DC voltage according to the flow rates of the slurry and the underflow. Furthermore, it was demonstrated that the direction of DC electric field should be arranged as the same direction of particles sedimentation, resulting in

much quicker and homogeneous sedimentation.

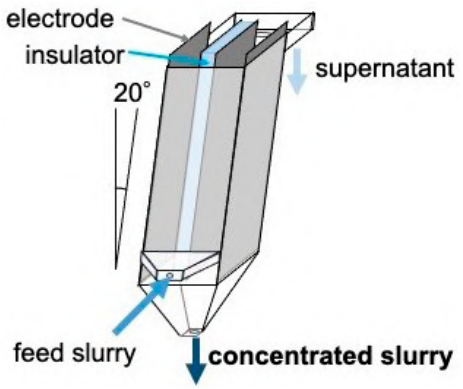


Fig.1 Experimental apparatus Fig.2 Particle concentration of the supernatant

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[1] Mori T., Nagashima H., Ito Y., Era Y., Tsubaki J., 2019, Agglomeration of Fine Particles in Water Upon Application of DC Electric Field, Miner. Eng., 133, 119-126.

Systematic screening of binder functionality for agglomeration of skim milk powder

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Keywords | Agglomeration, Food powders, Binders

The application of fine powders as generated by standard spray drying usually goes along with slow rehydration, poor flowa- bility and dust formation. Improvement over primary particles regarding powder handling is achieved by agglomeration. Fluidized bed spray agglomeration is a well-established size enlargement process. Binders are sprayed on the primary particles and dried after successful collision with further particles to solid bridges. Using binders beyond water can be motivated by a better energetic utilization, larger agglomerates, modified surface tension or a specific functionality (Bück & Tsotsas, 2016).

In this study, the influence of functional binders on agglomeration processes is investigated using spray-dried skim milk powder as a model system for the transition of Geldart classes C and A. With particle sizes of 20 µm (D50.3), skim milk powder is considered to be difficult to fluidize and the particle size should be enhanced by factor two to four using agglomeration. The long-term objective is to systematically recommend or select a binder that is suited for a given application. Preliminary studies have indicated that agglomerate properties generally differ between polymers (whey proteins, hydroxypropyl methylcellulose) and short-chain carbohydrates (lactose, maltodextrin). These binders were selected regarding their varying functional properties, i.e. glass transition, wettability, viscosity and drying kinetics (via water vapor sorption). With these binders a wide dry substance concentration range can be covered (0.04 to 40 %).

The influence of binder type on process as well as property function is investigated. First, the agglomeration kinetics for different functional binders are determined. Secondly, the influence of concentration for each binder is evaluated regarding agglomerate properties at defined particle size. The agglomerates are produced via top-spray agglomeration in a fluidized bed dryer with a constant average drop size, which was determined offline by Malvern Spraytec. Powder properties, i.e. rehydration behavior (wettability, dispersibility and disintegration), as well as stability, are evaluated (Ali et al., 2015). Special focus was laid on the simultaneous effects on stability and rehydratability. The underlying hypothesis is that agglomeration efficiency and product structure are correlated, which drives powder properties.

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Light scattering from nanoparticle agglomerates

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Keywords | light scattering, nanoparticles, agglomerates, discrete element model, discrete dipole approximation

Modern fire (smoke) sensors detect the light scattered from carbonaceous particles (soot) emitted during open fires. Such particles have fractal-like structure formed by agglomeration and surface growth (Kelesidis et al., 2017b). Yet, their optical properties are calculated typically by Mie or Rayleigh Debye Gans (RDG) theories impeding their selective sensing among other airborne particles. So, fire detectors are not selective enough for such particles producing false alarms costing up to 1 billion £/y in United Kingdom alone. Here, carbonaceous agglomerate structure and light scattering are measured from premixed ethylene flames (Kelesidis et al., 2020) and simulated by coupling Discrete Element Modeling (DEM) for surface growth and agglomeration (Kelesidis et al., 2017a) with the Discrete Dipole Approximation (DDA) (Kelesidis & Pratsinis, 2019). Using the classic RDG theory (Fig. 1: broken lines) results in 50 % smaller differential light scattering cross-sections than those measured in premixed ethylene flames (symbols). In contrast, the DEM-derived particle structure and light scattering (solid lines) are in excellent agreement with those measured here in premixed flames. Thus, the DEM-DDA can be used to optimize the selective sensing of fire detectors.

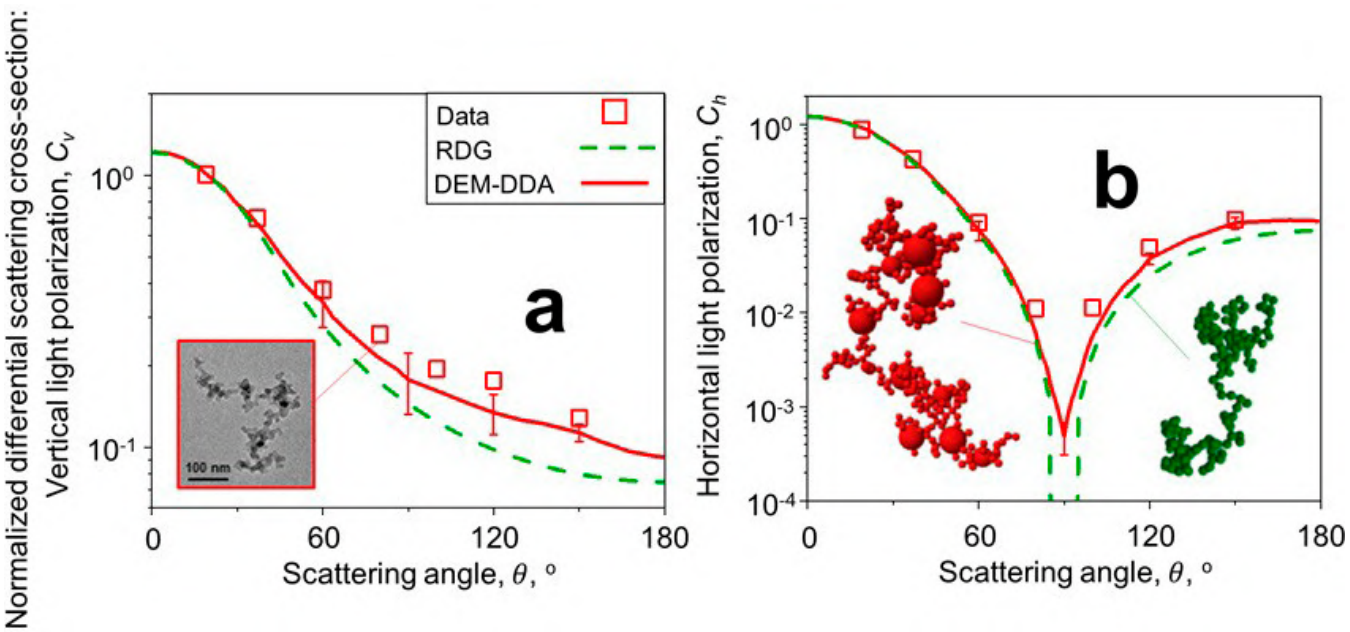


Figure 1. Distribution of normalized differential scattering cross-sections for verical, (a) *Cv*, and horizontal, (b) *Ch*, light polarization as function of scattering angle, θ, of measured (symbols) and DEM-derived soot agglomerates (solid lines) from a premixed ethylene flame are compared to the RDG theory (broken lines).

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Influence of the specific energy input of different slurry- devices on the properties of Si-Anodes

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(1) Institut für Partikeltechnik

Keywords | extrusion, scalability, continuous battery production, silicon anode material, active material stress, battery slurry, specific energy input

Lithium-ion batteries are the leading technology for energy storage. Intensive efforts have been made to improve the energy density of lithium-ion batteries, especially through the use of active materials containing high capacity silicon (Chae et al., 2017). But the type of processing with its specific energy input (Em) into material is also crucial for the performance of the battery. Therefore, in the future, the focus will be on the continuous production of battery slurrys (extrusion) in which higher stress intensities can be applied (Haarmann et al., 2020).

In order to produce an electrode of the highest possible quality, an efficient dispersion step is necessary. The success of the dispersion process depends largely on the specific energy input, the stress intensity and frequency. Thereby high Em's can result in a reduction of the carbon black particle size and can lead to an improvement of the conductivity of the electrode (Schilde et al., 2010). However, too high specific energy inputs can also lead to possible damages to the active material, deagglomeration of carbon black particles and to resulting capacity losses during battery operation. For this reason, the main objective of this study is to compare the Em's in a discontinuous high-intensity mixing process with those in a continuous extrusion process and to apply a modeling approach to describe the relationship between particle sizes and Em. To this effect, slurries are prepared in discontinuous and continuous processes and investigated with respect to their particle morphology and rheological properties. To have a closer look on the stressed slurries, the physical properties of resulting electrodes are analyzed. The electrochemical performance of the manufactured Si-Anodes are represented with an area capacity of 8 mAh/cm2 in full cells.

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Stirrer design for improved fluidization of cohesive microsilica

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(1) TU Delft

Fluidization can be used for the processing of powders or as a reactor type for chemical reactions. When it comes to powders with a small particles size, fluidization becomes hard due to strong attractive inter-particle forces. This results in channel formation and agglomeration, both of which undermine fluidization. Throughout the years, several techniques have been developed, often referred to as assistance methods, which allow for improved fluidization of cohesive powders. Mechanical vibration, electric fields, pulsed flow and acoustic vibration have all been shown to improve the quality of fluidization. When it comes to scaling up the reactors, utilizing these techniques, with exception of pulsed flow, becomes increasingly hard. In literature, only a couple of studies were found on the effect of stirring, or mechanical agitation, on the fluidization quality of cohesive powders. And no literature on stirrer design was found at all.

In our study, we look at the effect on fluidization quality of several stirrer parameters. Blade diameter, blade thickness, blade angle, blade orientation and the use of a helix design were all studied. The stirrers were 3D printed and tested in a 5cm column where bed height and pressure drop were measured. As cohesive powder, we used 10µm silica particles.

Results have shown a significant improvement of fluidization quality when stirrers are used. When no assistance is utilized at all, channels are formed and no fluidization takes place. Using a stirrer at a low height disrupts the channel formation and allows the powder to fluidize with a normalized pressure drop of up to 0.9 and bed height expansion ratios of around 1.5, which is comparable to our earlier study on the effect of vibration.

Stability/aggregation of binary colloidal dispersions in solvent mixtures

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Keywords | Stability, selective agglomeration, binary colloid, surface interaction

Stability/aggregation in multicomponent colloidal dispersions plays a key role in many technological processes and real- world applications. In this case, selective agglomeration of colloidal dispersions is an important example where a second liquid is titrated into a stable colloidal dispersion of nanoparticles for selectively agglomerating one component for separation. Selective agglomeration is a purely interface-controlled method that needs a clear understanding and control of the nanoparticle surface chemistry and molecular interactions to achieve the highest separation yield. To the best of our knowledge, though a wide range of fundamental studies of colloidal dispersions exists, the understanding of binary dispersion in solvent mixtures yet is missing. In this work, the stability/aggregation behaviour of a binary colloidal dispersion of quantum confined semiconductors and noble metal nanoparticles is experimentally investigated. Thioglycerol capped ZnS QDs (< 10 nm) and citrate-stabilized Au nanoparticles (< 20 nm), synthesized based on previously developed chemical methods [Turkevich et al., 1951, Komada et al., 2012], were used to generate the binary colloid with water as the continuous phase. The role of the chemistry and bonding strength of the surface ligand, the composition of the binary continues phase, and the presence of unwashed ions in the stability/aggregation in the binary dispersion is studied. We combine various characterization techniques including UV-Vis spectroscopy, dynamic light scattering, transmission electron microscopy, nuclear magnetic resonance spectroscopy, and fourier transform IR spectroscopy to comprehensively understand the aggregation mechanism based on surface interactions at the molecular level.

In conclusion, this study leverages our understanding of colloidal interfaces and complex interactions of binary colloids in solvent mixtures. This is so far not addressed but urgently needed for designing high-quality particulate products and efficient recycling.

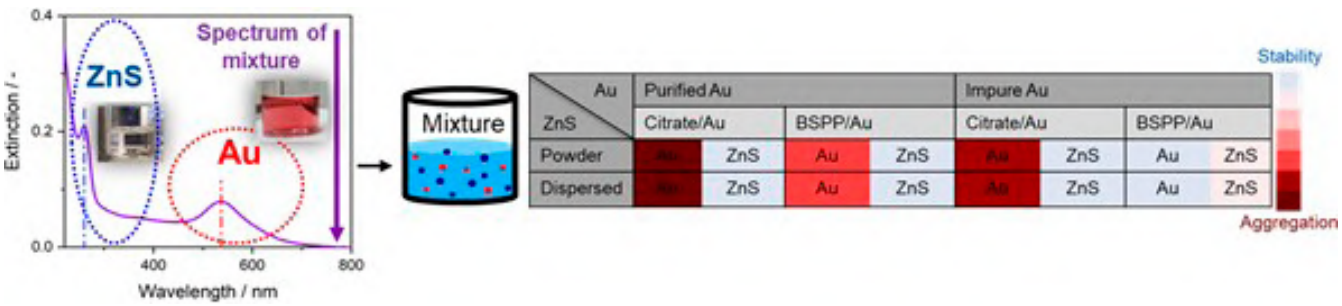


FIGURE 1. UV-Vis spectra and developed stability map of the binary colloid: Au and ZnS nanoparticles

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Systematic study of comminution of carbon carrier material for fuel cell application using wet impact mill

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Keywords | Polymer electrolyte membrane fuel cell, wet impact milling, analytical centrifugation, comminution, performance test

In the polymer electrolyte membrane fuel cell (PEMFC), carbon black with a high specific surface area is used as a support material for the electrocatalyst nanoparticles. Commercial catalysts offer little control over the particle size of the support material, making it difficult to tailor porosity in the final electrode and to distribute active sites on demand.

Thus, it is essential to overcome this challenge by developing a methodology for the systematic crushing of carbon aggregates via continuous

wet impact milling. This enables to optimize the carrier particles independently in terms of their size for targeted porosity and platinum loading.

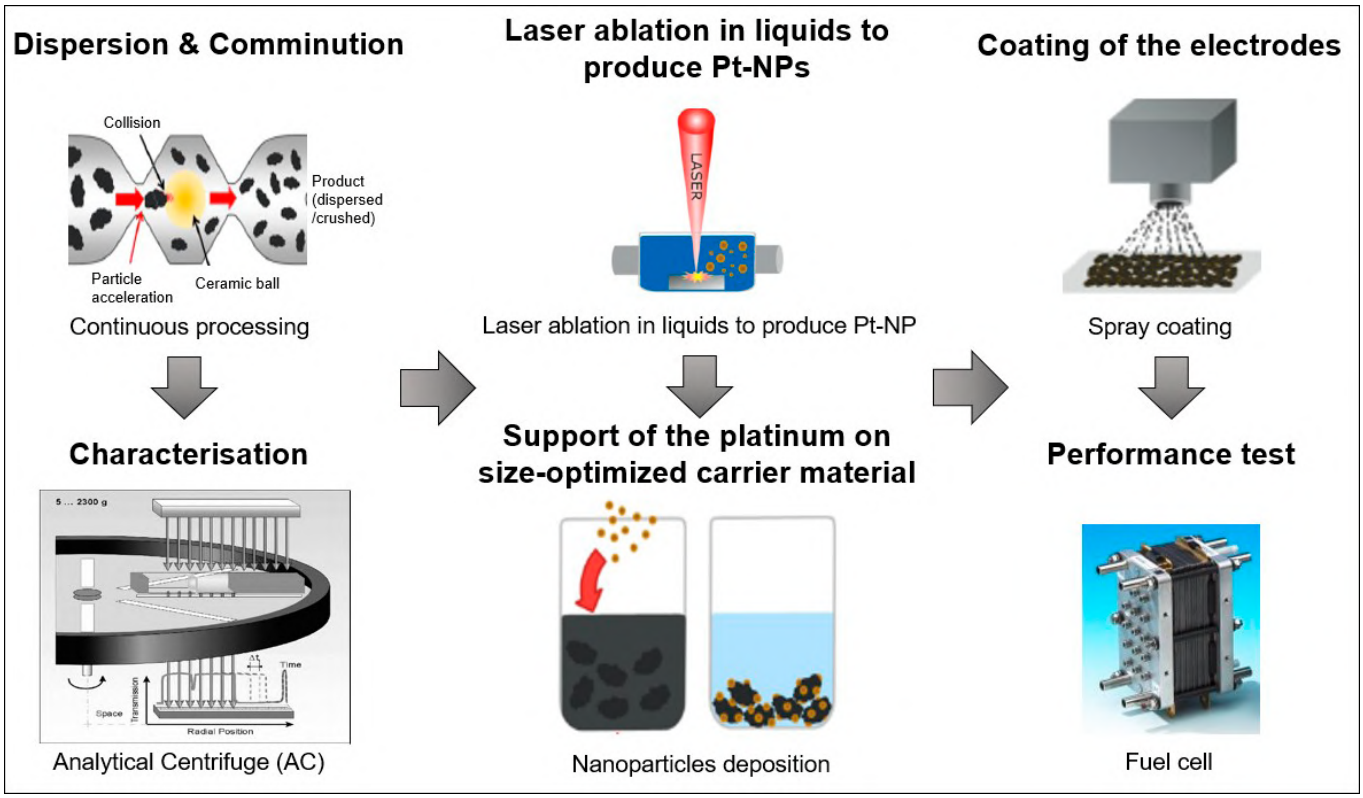


FIGURE 1: Schematic description of the study and interaction of the partners.

Continuous wet impact milling (Sugino Corp, Japan) offers two adjusting screws to control the comminution process, the hydrostatic pressure that can be achieved in the high-pressure chamber (1000 - 2450 bar) and the number of passes in the high-pressure chamber. The particle size distribution was determined by (LUMisizer, LUM GmbH). As carbon carrier material Vulcan XC72R was applied (Cabot GmbH, Germany), which is typically used in commercial catalysts for PEMFC (FIG. 1).

We found that dispersing the ink by wet impact milling leads to more stable formulations and significantly better fuel cell performance. It turned out that the change of the hydraulic pressure did not have a significant effect on the resulting particle sizes while we found that the mean size of the carbon can be reduced from 250 nm to 150 nm by changing the number of passes. Crucial for the comminution and homogenization of the carrier material is continuous energy input and thus the number of cycles.

After adjusting carrier particle size, platinum was supported with the project partners and the composites' electrochemical properties were tested in technically relevant membrane electrode assemblies (FIG. 1).

In summary, the interdependencies between the individual process steps can be deciphered and used for a knowledge- based and efficient production of PEMFC electrodes, while at the same time improving the environmental balance by reducing the required amount of precious metals such as platinum.

Selective agglomeration of submicron particles from wet fine grinding with potential for wear separation

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Keywords | stirred media milling; selective agglomeration; wear; interfacial separation

In the field of pharmaceutical/life-science products and for optical applications, suspensions with particle sizes in the submicron/nanometer

range with high quality standards are required. Within the top-down synthesis in stirred media mills, wear (abrasion) of the mill components, primarily the grinding media, is a major challenge. Due to an increasing contamination of suspensions with wear particles during the grinding process, the products suffer a loss of quality and can often be used only to a limited extent. A direct separation of the wear particles during/ after the grinding process cannot be accomplished easily, as these usually have particle sizes similar to the product components. In this regard, especially for very small particles (<1 µm), interfacial particle properties and the resulting particle interactions become strongly relevant, causing common mechanical separation principles to face their limits and to lose efficiency as well as selectivity.

Within this framework, a novel concept is demonstrated to separate grinding media wear particles from product suspensions achived by wet stirred media milling. The basis of the approach relies on the integration of the selective agglomeration into the milling step, allowing only the wear component to be transferred to a coarser particle size range, which enables a material-selective separation through known mechanical principles. For this, the success of a selective agglomeration strongly depends on the targeted control of the material-related, electrostatic particle-particle interaction and the corresponding agglomeration kinetics between the respective particle species. The investigation was carried out using the organic compound anthraquinone as an exemplary product material. In a first step, the fundamental mechanism of the selective agglomeration is presented on the basis of binary, defined sub-mircon mixtures with a model wear component (ZrO2). In fact, ZrO2 agglomerates with a size of up to 30 µm can be obtained applying suitable conditions, proving to be easily separable and thus enabling almost complete separation of the mixture. Within the further course of the study, the integration of the selective agglomeration into the milling step is considered, in which the focus is set on the identification of suitable stress conditions for the agglomeration step.

CFD simulation of highly loaded large-scale cyclones including particle agglomeration modeling

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Keywords | dense dispersed gas-solid flow, Dense Discrete Phase Model (DDPM), agglomeration modeling, sub-grid drag model, industrial-scale cyclone, coarse-grid, coarse-grained

The hydrodynamics of multiphase flow in highly loaded cyclones is complex because of having highly turbulent flow, particle- particle interactions, and particle agglomeration. This makes CFD simulation of such a case challenging especially for industrial cases where the use of coarse grids and coarse grains is inevitable.

In order to simulate industrial-scale cyclones with high solid loading, a hybrid multiphase model (Dense Discrete Phase Model based on Kinetic Theory of Granular Flow KTGF with particle tracking capability) was combined with an agglomeration model and a sub-grid drag model. In industrial cyclones with high particle load, the agglomeration of fine particles can affect the PSD and consequently the separation efficiency and pressure drop as the two most important performance parameters of cyclones. In this work, particle agglomeration was included to the CFD model by a computationally efficient stochastic model.

The model was validated by the experimental data from a pilot-scale cyclone with a diameter of 1.6m. Reasonable agreement with experimental pressure drops and separation efficiencies was obtained. Furthermore, key trends such as improvement in separation efficiency and reduction in pressure drop of cyclone due to an increase in particle load are well captured by the model. The study concludes that including the agglomeration model for fine particles (d < 15 µm) is crucial for the accurate prediction of pressure drop and separation efficiency.

A sensitivity analysis was performed on sub-models, model parameters, and numerical parameters to assess their significance on the model predictions. Different options for modeling of particle-particle interaction (based on KTGF), turbulence, and drag (five homogeneous drag models and four heterogeneous drag models) were examined. Additionally, parameters related to particle-particle restitution (that affects particle agglomeration), and particle-wall rebound were studied. Furthermore, a number of numerical parameters were investigated in this work, including the discretization method, grain-to-particle size ratio, grid-to-parcel size ratio, and time-step size. In addition, a number of operating conditions, such as solid loading and particle size distribution, were examined.

Agglomeration of wet solid particles in a fluidized bed

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Keywords | Fluidization of wet particles, CFD-DEM simulations, Agglomeration regime maps,

We have performed CFD-DEM simulations in three-dimensional periodic domains to investigate the effects of the Bond number (Bo), the capillary number (Ca), the liquid loading (Ψ), and the solid volume fraction (φ) on the dynamics of agglomeration in dilute suspensions of wet particles fluidized by a gas.

In these simulations, the averaged equation of motion for the gas phase was solved using OpenFOAM, and the particle motion was evolved using DEM implemented in LIGGGHTS, which were coupled together using CFDEM (Girardi, Radl and Sundaresan 2016), (Wu, Khinast and Radl 2018). Models for liquid bridges force and liquid transfer rate (Mohan, et al. 2014) are included in the simulations. The evolution of agglomerate size distribution (ASD) and mean quantities such as its Dagg were extracted from the simulation results through a computational geometry algorithm. The simulation revealed both the initial growth phase and the subsequent steady state.

Based on the simulation results obtained using a wide range of the relevant dimensionless parameters, we have constructed maps demarcating agglomerating and non-agglomerating regimes under steady-state conditions (Figure 1). The agglomerating regime contains systems in which Dagg/dp ≥ 2, while the non-agglomerating regime encompasses those in which Dagg/dp < 2. This talk will present the simulation methodology, sample results, and regime maps.

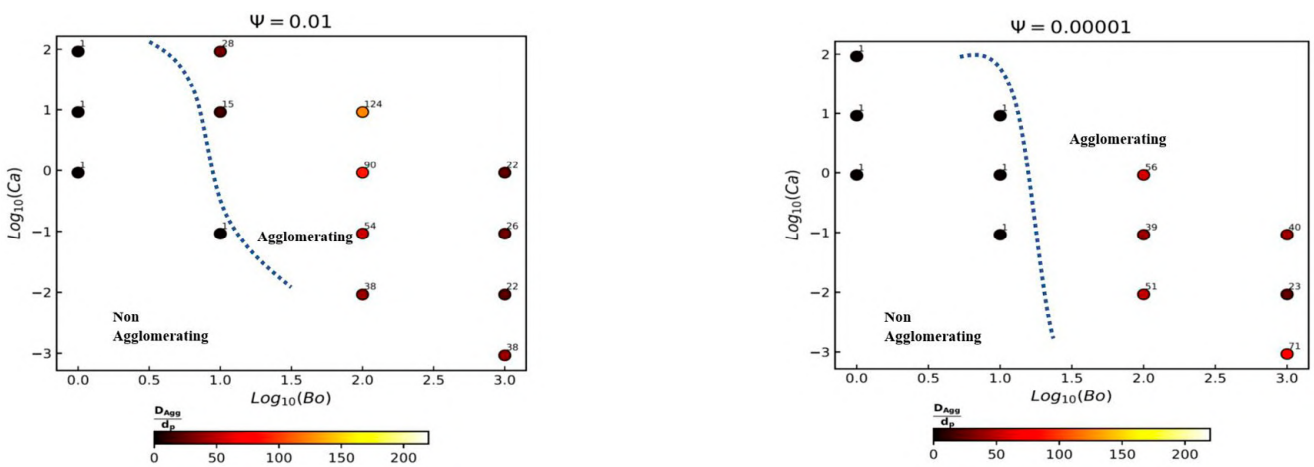


Figure 1: Agglomeration regime maps of cohesive systems in the Bo-Ca space for two liquid loadings (Ψ) and a solid volume fraction of 0.05. The color of the icons indicates the ratio of the mean diameter of the agglomerate and the primary particle diameter (Dagg/dp). The dashed line: Dagg/dp is ~ 2.

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A Widely Applicable Method to Stabilize Nanoparticles Comprising Oxygen-Rich Functional Groups

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Keywords | nanoparticles, stability, surface, functional devices, nanoparticulate inks

Colloidal stability of nanoparticle suspensions is a fundamental pre-requisite for the applications of nanomaterials in functional devices and for the development of new tools and methodologies in nanoscience. Herein, a versatile method is described to achieve dispersion and colloidal stability of various organic and inorganic solids with technological applications in the field of pharmaceutical industry, batteries and fuel cells, dye sensitized solar cells, pigments and coatings.[1] The method is based on the use of Zr(IV) salts solubilized in water to form hydroxo-complexes able to interact with the carboxylic- and hydroxo- functional groups at the solid-liquid interfaces of nanoparticles. The colloidal stability is assessed via sedimentation analysis, Zeta potential measurements, ultraviolet-visible spectroscopy (UV-Vis), attenuated total reflection-Fourier transform infrared spectroscopy (ATR-FTIR) in liquid phase and isothermal titration calorimetry (ITC). The effect of the anion and the interplay between pH and Zr(IV) hydroxo-complexes generated in-situ are studied in depth.

Finally, our method enables liquid phase length and diameter characterization of rod-like goethite (a-FeOOH) particles without relying on tedious counting the particles from electron micrographs and stabilizes also drug nanoparticles, Pt-coated carbon electrode materials and various oxide nanoparticles.

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Planetary roller processing – The new level of continuous granulation

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Keywords | Continuous manufacturing, Melt granulation, Planetary roller processing, Planetary roller extruder

Granulation is a standard unit operation to modify the particle size for improved material handling and dosing properties in powder processing [Serno et al. 2007]. Here a research focus in recent years has been on continuous operation. For this type of manufacturing, a promising alternative is the processing with a planetary roller granulator, which is classified as multiple screw machine. In addition to the modular design of the typically applied twin screw machines [Djuric et al. 2008], the surface-to-volume ratio and consequently the temperature control is dramatically enhanced. This is related to the unique process concept of planetary roller machines (Figure 1), where planetary spindles orbit a heated central spindle within a heated barrel. The ribbed helix surface of these elements and their interlocking results in an axial material transport and enlarged contact area between machine surface and processed material. This is especially valuable for melt granulation, where a binder is melted to build-up solid bridges.

The objective of this study is the fundamental investigation of planetary roller melt granulation in two steps. The characterization of the process space and then the systematic exploration of a design space. In both cases, the machine configuration (spindle elements, number of planetary spindles, barrel) was kept constant and main critical direct process parameters varied. As a result, the process space of continuous melt granulation defined by the feed rate and screw speed is determined for different parameter sets of barrel and central spindle temperature (Figure 2). The findings include basic limitations related to the used equipment (motor, feeder) as well as the product quality. In addition, the impact of the direct process parameters on the properties of the granule product (size distribution, flowability, density) is characterized and correlated to indirect process parameters (feed load, specific mechanical energy, specific thermal energy).

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Single Drop Granulation of Pharmaceutical Binary Mixtures

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Keywords | Wet Granulation, Spreading, Tunneling, Granule Morphology, Granule Porosity, Granule Uniformity

Many industries, including pharmaceuticals, food, agricultural chemicals, detergents, catalysts, and consumer products, manufacture a variety of products from particulate materials. Product or intermediate specifications may include size, size distribution, shape, strength, porosity,

and content uniformity of particles or of particulate-based delivery forms. A common particulate unit operation is wet granulation, whereby a liquid is added to fine particles which then agglomerate together to form larger granules with improved flow properties. In single drop granulation, one drop forms one granule, and the granule size can be controlled by the drop size. Two main categories of granule formation mechanisms, namely Spreading/Crater Formation and Tunneling, have been identified (H.N. Emady et al., 2011) for single-component inorganic powders, and they can be predicted via a regime map that incorporates dimensionless groups involving formulation properties and process parameters (H.N. Emady et al., 2013).

In this work, single drop granule formation on a static powder bed of pharmaceutical mixtures of acetaminophen and microcrystalline cellulose was studied to investigate the granule formation mechanisms and resulting granule properties (size, morphology, pore structure, and content uniformity). The granule formation mechanism was characterized via high- speed video imaging; granule properties were characterized via image analysis with a prism setup (size and morphology), X- ray micro-CT (pore structure), and UV-vis spectrometry (content uniformity). It was found that the primary particle size, the arrangement of particles in the bed, and the wettability of the powder bed strongly affect the formation mechanism, which in turn determine the end granule attributes. A regime map was also attempted to predict the granule formation mechanism and resulting granule properties.

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Monitoring microcapsules formation using laser diffraction spectroscopy

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Keywords | online monitoring, microencapsulation by solvent evaporation, laser diffraction spectroscopy

Microencapsulation by solvent evaporation is a simple technique to produce microcapsules in a stirred tank. First, the core material and the encapsulant material (usually a polymer) are dissolved in a solvent; next, this liquid mixture is emulsified in a second liquid phase; then, solvent evaporation is promoted leading to polymer precipitation and transformation of the emulsion into a suspension of solid microcapsules; finally, microcapsules are recovered by filtration and drying. Besides final product characterization, real time monitoring of particles formation and evolution may provide valuable information to optimal product/process design. Amongst other techniques, FBRM (Focused Beam Reflectance Measurement) has been used to inline monitoring of several particulate processes, including microencapsulation by solvent evaporation (Silva et al., 2013; Muhaimin et al., 2021), but has significant drawbacks, namely reduced accuracy for small particles and dependence on particles's optical properties. On the other hand, LDS (Laser Diffraction Spectroscopy) is a well-accepted reference method to measure particle size distribution (PSD), being increasingly used online, namely in crystallization and spray drying (Ma et al., 2001; Medenport et al., 2015).

Here, LDS is applied for the first time to online monitoring of microencapsulation by solvent evaporation through a simple but representative case (encapsulation of ibuprofen in ethylcellulose, solvent: dichloromethane; continuous phase: water; surfactant: Tween 80; process: 0.75 L of mixture, 35 mm Rushton impeller; recirculation of 0.6 L/min through Materziser 2000 measurement cell; offline measurement of solvent concentration in the continuous phase using a refractometer; evaporation at 35 ºC during 3 h). The attached figure shows measured PSD along time, for a base case. Two other cases were studied: one with a higher concentration of dispersed phase and another one with higher impeller speed. Reproducible results were obtained in all cases, providing valuable process insights. In particular, the set of PSD and solvent concentration data along time offer a rich picture of how the particulate system evolves, including the initial fast emulsification followed by an increasingly slower solvent extraction from particles until completely solid particles are formed, which can be of industrial importance to optimize microencapsulation formulations and processes.

The role of nozzles number and inclination angle in the spiral jet milling of pharmaceutical substances

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Keywords | Classification; comminution; spiral jet milling; geometrical optimization

Spiral jet milling is a very specialized process in which high-energy particle-particle and particle-wall collisions are used to micronize powders of various substances. Among the different technology sectors in which it is employed, the processing of active ingredients for the pharmaceutical industry constitutes one of the most common applications.

In this framework, the capability of reproducing the same particle size distribution (PSD),defined in terms of D10, D50 and D90, is very important for a successful process transfer from different milling sites. For some specific pharmaceutical applications, like Dry Powder Inhaler (DPI), the relation between D10-D50-D90 is very critical to define the physical properties of the micronized powder. This relation is properly described by the SPAN formula (SPAN=D90-D10/D50), which is needed, more than other analytical properties, to capture the effect of the different jet mills specific geometry, instead of the size of the apparatus or the applied process parameters.

In this work we studied the two major geometry factors affecting the PSD, consisting in the nozzles number and inclination angle. Indeed, while the specific energy (SE) can be easily reproduced by adjusting the main process parameters,i.e.,the grinding pressure and the powder feed rate; the classification effects due to the nozzles topology cannot be described by the sole operating conditions but depends on the brand-related specific geometric characteristics.

We therefore performed experiments micronizing two different input products in the same spiral jet mill and we investigated the effect produced by the different number of nozzles and the different nozzles angle on the PSD of the resulting product. We identified and we analyse the relation intercurring between span and specific energy.

Finally, a simple guide to correct the geometry is proposed to achieve a proper technology transfer of the micronization process.

Three-dimensional microstructure analysis and breaking mechanisms in heterogeneous structures

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Keywords | breakage mechanism, in-situ particle breakage, XRM, micro-CT, compression, fracture, microstructure analysis

The challenges we face today are based on how our society can contribute to a sustainable and efficient management of natural and limited resources. Nowadays, many recycling methods are available to recover materials that represent the main components of the waste. Most of the lower concentrated elements which have limited availability and are crucial for modern technology dissipate in the core product mass flux. To achieve a closed and consistent recycling circuit, it is also necessary to recover those critical and mainly rare elements such as lithium in batteries or tantalum in capacitors.

For this purpose, the priority program 2315 was created to develop a new solution to recover those elements with a geo- metallurgical approach. The idea is to use the slag phase as a carrier for the elements where they react with e.g. oxygen or sulphur and crystallize at a low cooling rate. This step imitates the natural rock formation process and, therefore, the product are so-called engineered artificial minerals (EnAM).

The presented part of the project deals with the liberation of the EnAM. Slag particles with EnAM phases are used to determine the breaking mechanisms. Gaudin (1939) and King et al. (1998) postulated that the breaking behaviour of single particles is directly correlated with the particle microstructure. For a non-destructive and three-dimensional investigation, X- ray microscopy (XRM) is suitable for correlative high-resolution analysis of the microscopic structure and the already existing fractures prior to the breakage and for in-situ compression experiments of the slag particles.

First results have shown that is possible to segment fracture planes from the XRM data with machine learning algorithms. Fracture width distribution and fracture lengths are determined (Figure 1 (left)). The workflow is transferred onto the crushed particle data. In-situ compression measurements can be conducted with a Deben tensile load cell CT5000 and the XRM data can be correlated with force-strain curves (Figure 1 (right)).

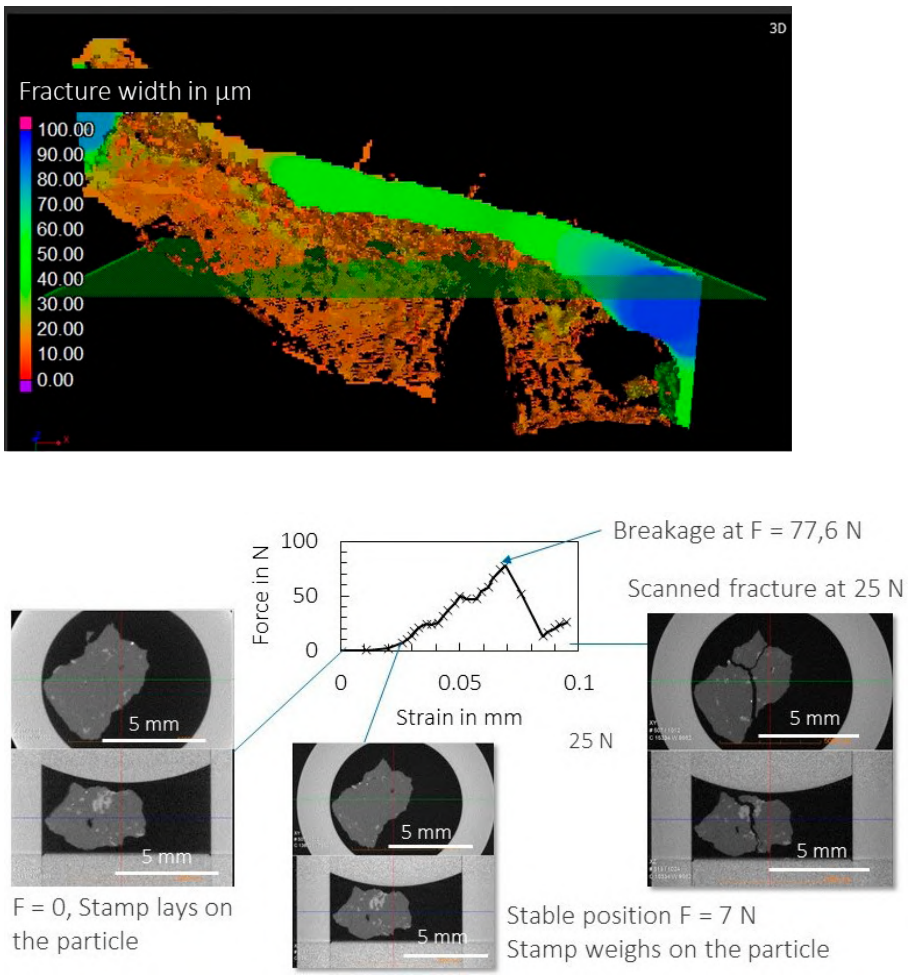


FIGURE 1: Fracture width distribution of a fracture plane (left), in-situ measurement with a model particle in the compression load cell, and data acquisition with XRM (right).

Optimization of particle breakage during combined crystallization-wet milling processes – A Mechanistic Modelling Approach

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Keywords | wet milling, crystallization, breakage, growth, optimization

The pharmaceutical industry is starting to adopt continuous active pharmaceutical ingredient (API) manufacturing in order to reduce production costs, improve manufacturing flexibility, reduce infrastructure costs, reduce manufacturing lead time (from typically 6 months to 10 days) and to improve sustainability. To facilitate the transition, digital design and modelling are being adopted to aid in exploring the design space and study the optimal performance of important unit operations, such as crystallization and wet milling. The combination of crystallization and wet milling processes is an increasingly studied area of pharmaceutical manufacturing and presents several benefits for industrial application (1, 2). However, given the complex interplay of crystallization and wet milling mechanisms, there is a need for mechanistic understanding and modelling to control the particle attributes through identifying the critical process parameters.

The focus of the work reported here aims to decouple and better understand the mechanisms involved in a combined cooling crystallization-wet milling process. Carefully designed lab-scale experiments were performed in stages to isolate the rate processes. This informed the development of a population balance model. In detail, this work focuses on the development of a mechanistic model-based workflow for

the optimization of an integrated crystallization and wet milling model, with a view to parametrize the breakage kinetics as well as optimize the milling performance. Results from model validation showed that with the dataset used for modelling, the model was able to effectively estimate the crystallization and wet milling breakage performance as shown below. The calibrated kinetics were subsequently utilized to explore the design space and optimize the process.

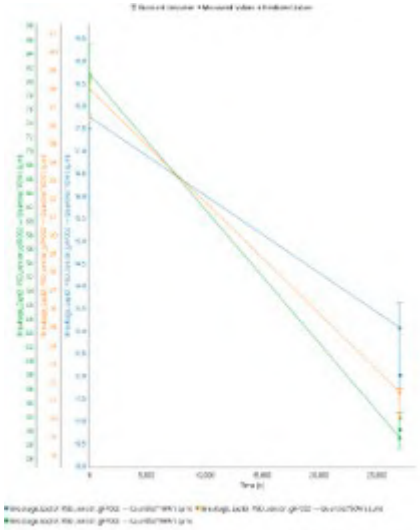


Figure 1: Predicted PSD quantiles for a wet-milling process

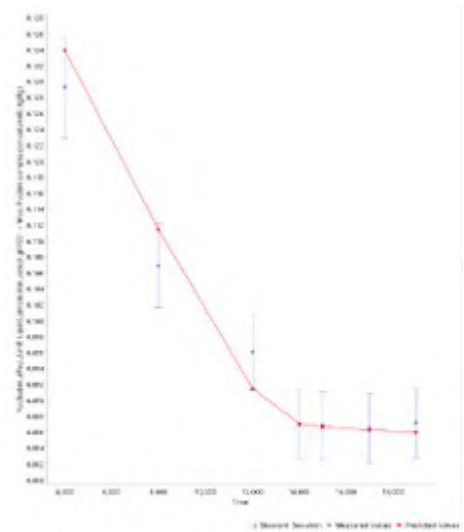


Figure 2: Experimental vs Predicted fit for an integrated crystallization and wet milling dataset

These kinetic estimations were used to understand the design space and optimize the process.

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Wet nanomilling of drug materials using a novel stabilization mechanism via Zirconium complexation

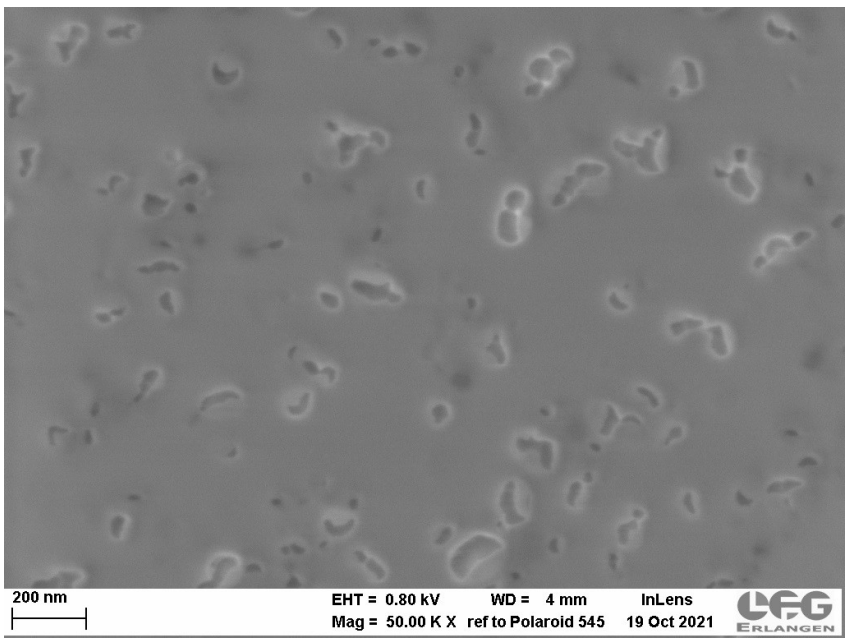
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Keywords | stirred media milling, naproxen, grinding limit, solubility, colloidal stability

Size reduction of drug particles down to the nm-range by wet milling is an effective and scalable method to achieve enhanced bioavailability due to a higher dissolution rate (Peltonen 2018, Konnerth et al. 2017). Traditionally, surfactants, polymers or combinations of both are used as milling additives in drug formulations. Via steric stabilization, an apparent grinding limit of several 100 nm is typically observed for drug particles (Bitterlich et al. 2015, Kumar et al. 2014).

Using a novel electrostatic stabilization mechanism that utilizes complexation of Zr(IV) species by carboxylic acid groups at the particle surface, long-term stable monocrystalline naproxen nanoparticles of 30 nm are prepared by stirred media nanomilling. However, such small particles cannot be stabilized against agglomeration with the conventional polymeric stabilizer polyvinylpyrrolidone. We demonstrate that the Zr(IV)/drug concentration ratio must be in an optimum range to provide excellent electrostatic stabilization and to avoid agglomeration and high drug solubility. X-ray diffraction reveals that the crystallite size of naproxen does not decrease during grinding, implying that particle breakage occurs along the grain boundaries.

By varying systematically the size of the milling beads and therefore the stress energy (SEmb) and the number of stress events (SN), we found that small milling beads are favorable for product fineness and nanoparticle yield. The threshold SEmb must be above a critical value to achieve significant fracture. In further studies, the nanoparticle yield of so far 40 % due to particle growth will be further increased, for instance by coupling the milling with a classification step. Analysis of the solvent phase after milling and the particle geometry shows that mechanical stress leads to enhanced solubility of the particles, which in return promotes re-crystallization. The solubility can be reduced by lowering the process temperature and stabilizer concentration. In contrast to inorganic materials (Knieke et al. 2009), which were almost free of lattice imperfections at the true grinding limit, we find indications of microstrain in the stressed nanoparticles. With this contribution, we extend the possibilities of nanomilling beyond inorganic materials (Stenger et al. 2005, Knieke et al. 2011) for the production of pharmaceutical nanoparticles.



Autogenous grinding of silicon in wet-operated stirred media mills: Investigation of process and product parameters

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Keywords | silicon, lithium-ion-battery, comminution, stirred media mill, autogenous grinding, nanoparticles

Due to its outstanding electrochemical properties, the interest in silicon nanoparticles as anode material in lithium-ion- batteries is increasing rapidly. With that, high capacity production methods of high-quality silicon nanoparticles are being investigated intensively. One possible solution is autogenous grinding in stirred media mills. Here, grinding media and feed particles are made of the same material. As a result, grinding media wear becomes the product, and thus, contamination is reduced to a minimum. Kwade applied autogenous grinding in stirred media mills and ground limestone down below a few micrometres (Kwade, 1997).

In this study, parameters and their impact on the energetic performance of the autogenous grinding of silicon is demonstrated. The autogenous grinding is based on a conventional nano-grinding of silicon in ethanol (Nöske et al., 2019). The goal is a direct comparison of the energetic performance of the conventional and autogenous process.

Process parameters such as stirrer tip speed, autogenous grinding media (aGM) filling degree, and aGM size influence the specific energy input. As the stirrer tip speed increases, the wear rate of the aGM increases. Thus, the product mass increases resulting in a better efficiency as long as the slurry viscosity and particle number does not impede the energy transfer of the grinding media. Same effects were observed with an increasing solid content. For autogenous grinding, the grinding efficiency at different filling degrees is a function of product particle size. This effect is pronounced when the feed material is coarse. The stirrer contains a deflector wheel that holds back particles above a process specific cut size. Larger particles need higher stress energies to be ground by the same grinding media size. Therefore, a aGM filling degree of 50 % is more effective due to increasing aGM velocities. On the contrary, product sizes in nanometre range profit from an increasing number of contacts at 80 % filling degree. Furthermore, aGM size of 0.65 mm show a better efficiency than 0.25 mm when feed particles with a median particle size of 2 µm are used. However, the smaller aGM size generates narrower product particle size distributions compared to the larger size.

Influence of different drying profiles and calendering loads on moisture content and structural properties of particulate cathode layers for Lithium-ion-batteries

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Keywords | particulate cathode layers, drying, calendering, adsorption, moisture content, structural properties, Lithium-ion- batteries, NCM622 cathodes

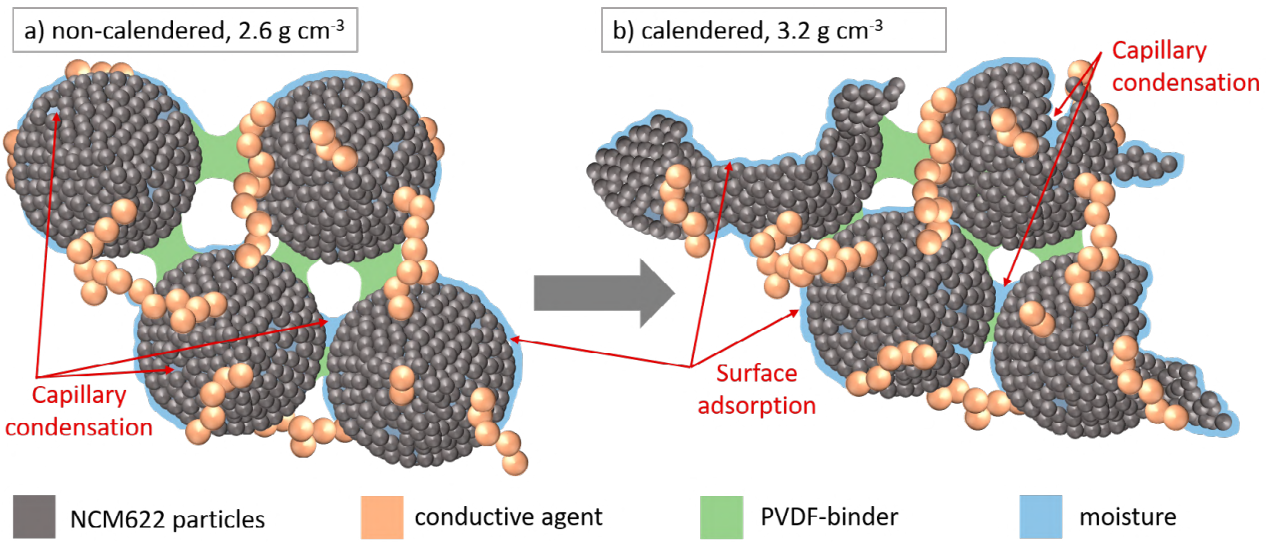
Electrodes for Lithium-ion-batteries (LIBs) are produced by coating a solvent-based paste onto a current-collector, followed by convective, continuous drying. The design of the drying profile significantly influences the formation of the particulate, porous coating and consequently its microstructural properties and moisture. Drying implies a high energy saving potential, but too high drying rates impair the electrode properties, particularly due to binder segregation. After drying, the electrodes are calendered, i.e. compressed, which also strongly influences their particulate properties and moisture. Moisture represents a contamination in LIBs as it impairs electrochemical performance and safety, which is why electrodes are post- dried before cell assembly. Currently, the individual process steps are considered separately with regard to moisture, without taking interactions into account. Consequently, potentials for energy saving or quality improvement cannot be identified.

In this work, the influence of different drying profiles and calendering loads on microstructural properties and related moisture content of NCM622-based cathodes was investigated. Moisture was analyzed as function of electrode microstructure at all process steps including drying, storing, calendering and post-drying. For a further evaluation of the formed particulate structures, the pore size distribution of the entire coating and the conductive network was determined via mercury intrusion. In addition, adhesion strength and electrical resistance was measured and SEM- and EDX-analysis was performed.

The results showed that the energy consumption during drying can be reduced to 40% while maintaining an adequate mechanical stability of the coating. Moisture decreased when drying with higher temperatures, but lower drying time. However, during handling the electrodes underwent a high resorption, which was further increased by calendering. The latter was in accordance with previous results [1], which showed a higher moisture uptake after calendering due to an increased specific surface area, caused by particle breakage. Altogether, if

resorption after drying could be prevented by directly transferring the electrodes into a dry room, post-drying could be saved and the total energy consumption would be remarkably reduced.

[1] Huttner et al.: Increased Moisture Uptake of NCM622 Cathodes after Calendering due to Particle Breakage, J. Electrochem. Soc. 168 090539 (2021), doi.org/10.1149/1945-7111/ac24bb, CC-BY 4.0



Schematic representation of moisture sorption at particle surfaces in a NCM622-based cathode coating in (a) the non-calendered state and (b) after compression to a density of 3.2 g cm^{-3}. Due to the enlarged surface area after calendering (b), a higher amount of moisture is adsorbed. [F. Huttner, A. Diener, T. Heckmann, J. C. Eser, T. Abali, J. K. Mayer, P. Scharfer, W. Schabel, A. Kwade, *J. Electrochem. Soc.* **168** 090539 **(2021),** doi.org/10.1149/1945-7111/ac24bb, CC BY 4.0]

Towards a Higher Energy Density for Lithium-Ion Battery Anodes via Hierarchically Structured C/Si Agglomerates Using Spray-Drying

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Keywords | agglomeration, spray drying, silicon anodes, high energy density anodes

Silicon has a high potential to replace commercial graphite anodes. It can help to achieve excellent energy density Lithium- ion batteries (LIB) because of high theoretical specific capacity it offers (Chan et al., 2008). However, major challenges such as rapid capacity fading and low coulombic efficiency due to the huge volume change (~300 %) during cycling have seriously hindered its commercialization (Wu et al., 2013). Although nanostructuring has been successful in minimizing volume expansion issues, the electrochemical performance of nanosized silicon is still limited due to unstable solid- electrolyte interphase, low tap density and overall poor electrical properties due to the higher interparticle resistance (Liu et al., 2014). To tackle those challenges, we introduce a new concept of post-synthetic spray-drying to produce hierarchically structured microagglomerates of carbon/silicon (C/Si) composite nanoaggregates synthesized in the gas phase.

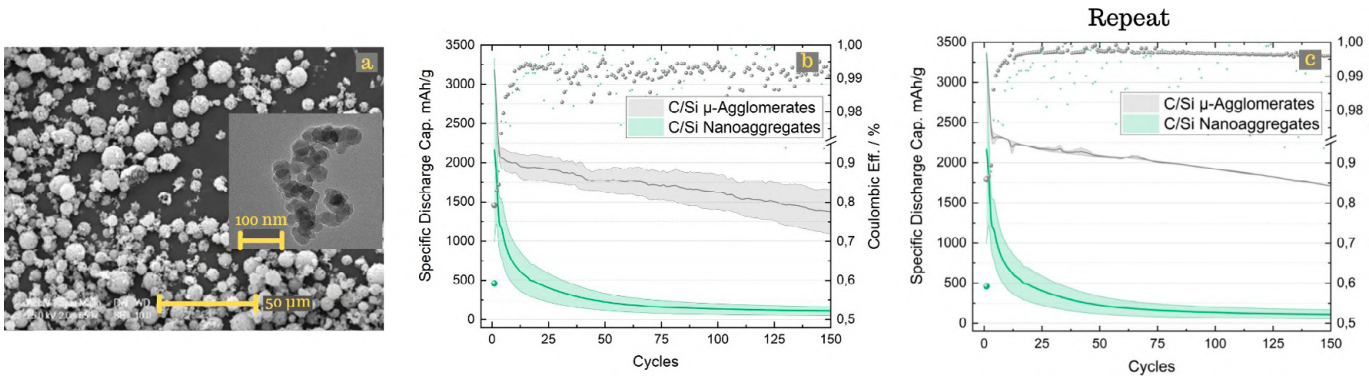


Fig.1.a) Agglomeration of C/Si nanoaggregates (inset) using spray-drying. b) Specific discharge capacity and coulombic efficiency with cycling of electrodes made from C/Si nanoaggreagtes (green) and µ-agglomerates (grey). c) Repeat to evidence reproducibility.

The resulting agglomerates were characterized i) on the powder level by scanning electron microscopy and [illegible] sorption, ii) on the dispersion level by rheometry and analytical centrifugation, iii) on the electrode level by atomic force microscopy for structure and iv) via electrochemical testing on half-cells for electrochemcal performance (Fig. 1b and c). The results show that the electrodes from C/Si agglomerates with highest redispersion stability exhibit excellent specific discharge capacity as compared to nano C/Si powder.

To conclude, our investigation suggests how an established industrial process (nanoparticle synthesis in a hot-wall reactor) can be combined with scalable one-step spray-drying to get highly dense active materials for LIB. This enables to make full use of the new materials' potential by the right packaging into optimum electrode structures with high performance and longevity.

References

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Wu et al., Nat. communications 2013, 4. pp. 1943.

Experimental Study on Drying Bamboo Chips with the Drying Device that Reproduces the Demonstration Facility

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(1) Fukuoka University

Keywords | Drying, Biomass Energy, Renewable Energy, Bamboo, Lower Calorific Value

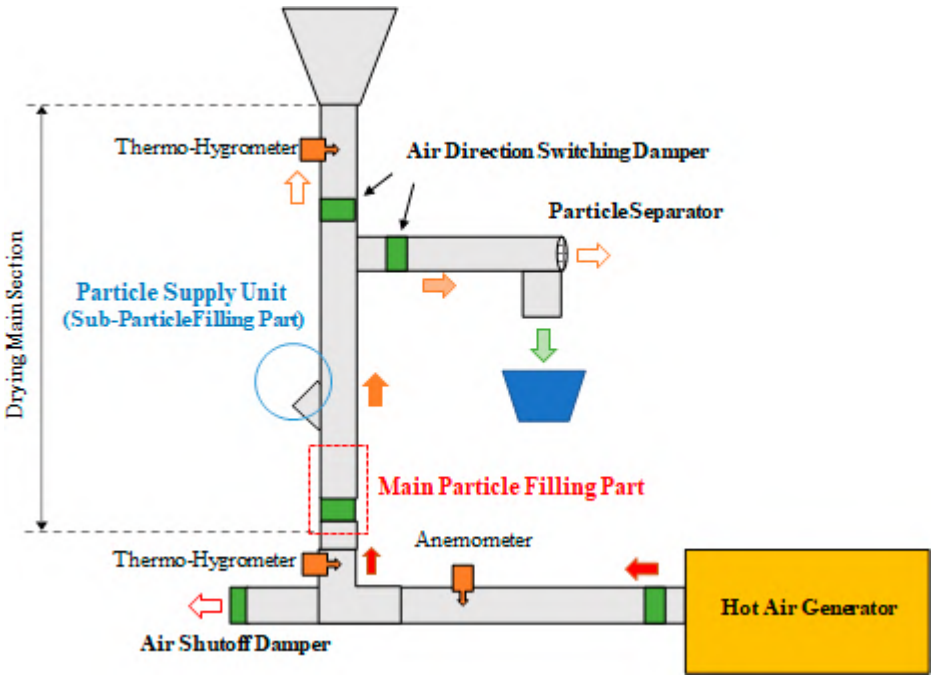
"Carbon Neutral" is becoming a trending keyword, with growing interest in climate change worldwide. Expansion and promotion of renewable energy use is required with the aim of breaking away from fossil fuels. Biomass energy is one of energy-creations that can achieve substantial "Carbon Neutral".

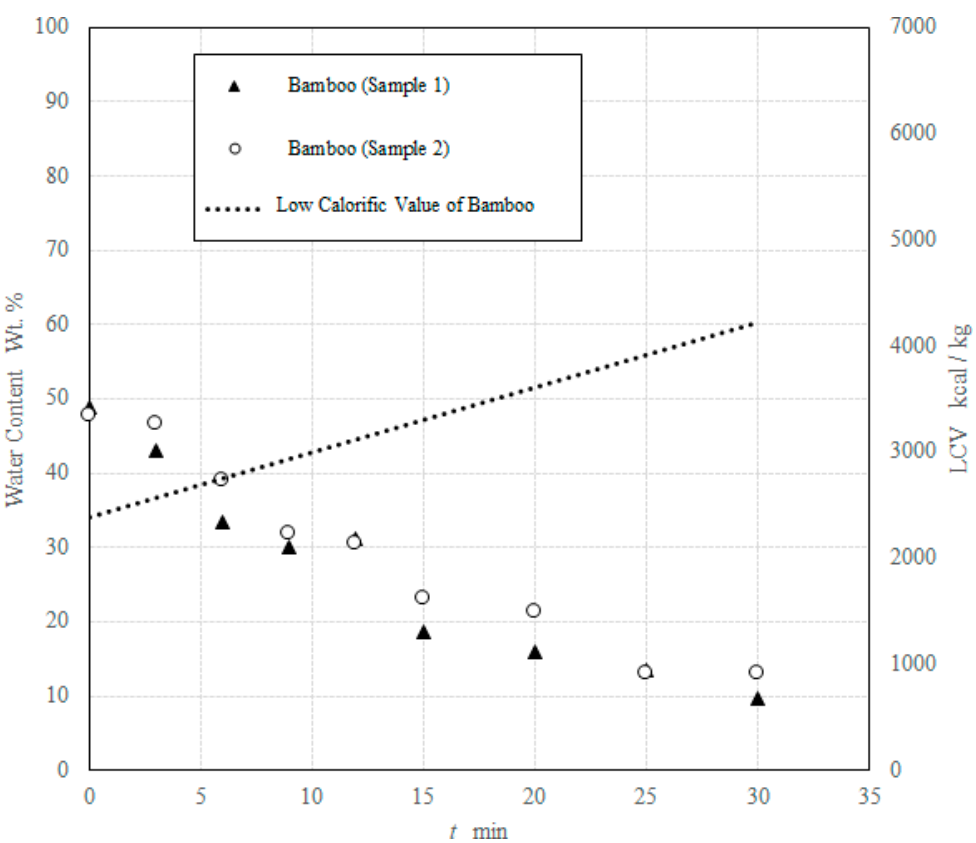
In Japan, it has been exploring the possibility of using "moso-bamboo" as a fuel for biomass boilers, which has a particularly strong fertility and has regarded as a troublesome of forestry workers. In this research, regarding "high water content", which is one of the biggest problems in biomass fuel, bamboo chips are dried by a simple drying device assuming that a boiler is installed, and the water content changes with time and accompanies it. We investigated the increase and decrease in low calorific value.

Zinc-coated air conditioning ducts are used for piping throughout the device. A hot air generator was used instead of the actual waste heat from the boiler. The inner diameter of the pipe is 125 mm, which is the same as in the demonstration experiment. The three-axis geometric mean of the particles is *Davg.* = 6.83 mm. Bamboo chips is filled in sub-Particle filling port and the mean flow velocity through pipe is u = 5.2 m/s. The average temperature of the air at the inlet of the dry section was Ti = 67.8 °C, and the average humidity was 7.5% RH. The filling amount of chips per experiment is 120 g.

It is well known that the low calorific value of wood and bamboo is significantly reduced when the water content is high. From this research results, it was confirmed that the water content of 30% (Wt. %) or less, which is one of the indicators as a fuel for biomass boilers, can be

achieved in only about 15 minutes from the start of drying. By using this drying technique, the significant decrease in low calorific value due to the higher water content of bamboo can be significantly recovered in a relatively short time. In the near future, we will also show the relative power consumption of the dryer.





Effect of rotation speed to thermal dehydration characteristics of waste gypsum particles in a constant volume rotary vessel

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(1) National Institute of Technology, Oita College, (2) National Defense Academy

Keywords | Calcium sulfate dihydrate, Calcium sulfate hemihydrate, Heating, Thermal dehydration, Rotation speed, Pressure, Temperature

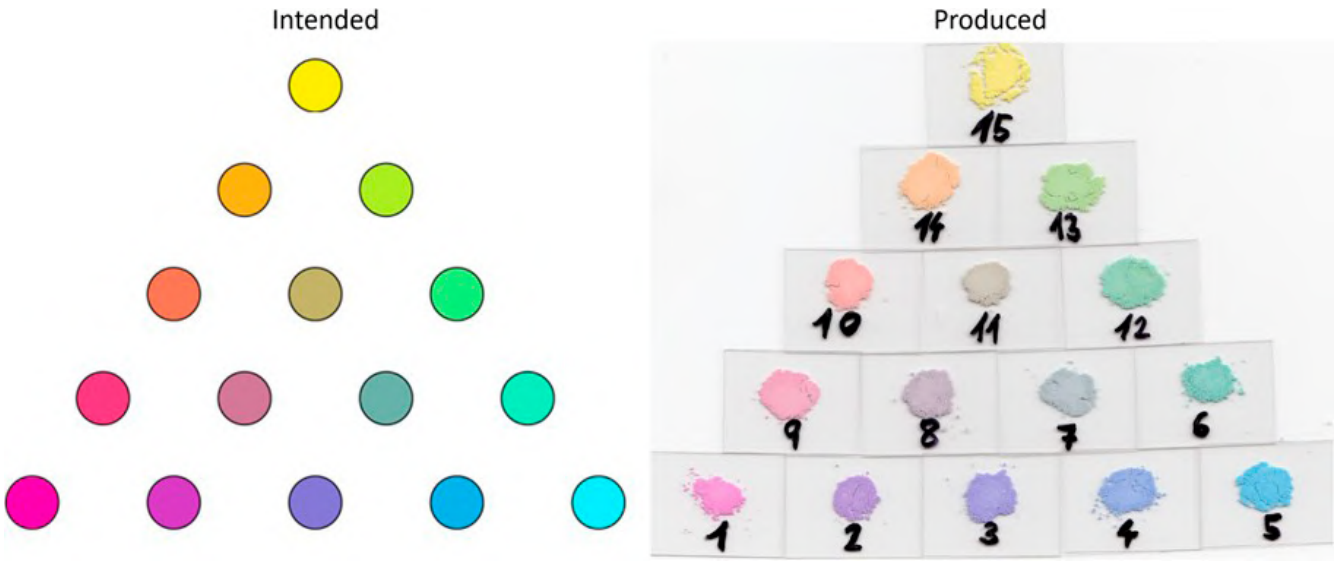
Gypsum board is widely used as an interior material for buildings with excellent fire resistance, sound insulation, and workability. On the other hand, the emissions of waste gypsum boards are risen by demolishing old buildings. Therefore, various efforts for recycling waste gypsum are required. One of them is expected that calcium sulfate hemihydrate (CaSO4.0.5H2O) is applied as the ground improvement material. Calcium sulfate hemihydrate is generated usually by the heating of calcium sulfate dihydrate (CaSO4.2H2O). However, the thermal dehydration characteristics of gypsum have not been clarified well when the state of temperature and pressure were varied. Therefore, this study has experimentally investigated the state change characteristics of calcium sulfate in a constant volume rotary vessel. The particle used was calcium sulfate dihydrate which was obtained from the crushed waste gypsum board. The particle size range is 850-2000 /mu m, and the particle density is 2623 kg/m3. Figure 1 shows a schematic diagram of a constant volume rotary vessel for heating the calcium sulfate dihydrate particles of 100 g. Temperature and pressure inside the vessel were measured simultaneously using the thermocouple and the pressure sensor, as shown in Fig.2. The rotation speed of 1 and 35 rpm, and the vessel temperature was heated up to 180°C. The result indicates that the growth rate of temperature against the heating time was almost the same for both rotation speeds. However, the periodical temperature change has occurred at the low rotation speed of 1 rpm. In this case, the particles inside a vessel were collapsed periodically toward the downward direction. This particle behaviour was related to the temperature profile of the rotation speed of 1 rpm. Additionally, the pressure difference occurred at different rotation speeds, as shown in Fig.2. This trend indicates the desorption of the crystal water of calcium sulfate dihydrate by the increasing temperature in the case of high rotation speed. It means the rotation speed influences the heat conduction and the heat transfer of the particles in a constant rotary vessel.

Continuous high-throughput screening of spray-dried formulations for bioavailability enhancement

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(1) UCT Prague

Keywords | Spray drying, automation, amorphization, high throughput screening

In pharmaceutical industry, the last decade saw a rise of high throughput screening and combinatorial chemistry – two powerful techniques, which significantly accelerated the rate of discovery of the new active pharmaceutical ingredients (APIs). However, due to the nature of these processes, a majority of these newly discovered molecules are practically insoluble in water. Since drug intake into human body is controlled by the ability of the drug to dissolve in water, this poses a challenge for formulation scientists. One of the solutions to this problem is drug amorphization, which can be achieved in different ways, however recent literature review (Edueng et al., 2017) reveals spray drying as the most popular technique nowadays. However, spray drying is a batch unit operation, which means that before product with a different composition can be produced, the process has to be stopped and the device has to be cleaned, which significantly increases the time needed for product development. In this work we introduce a new, highly efficient approach to combinatorial production of spray-dried samples, which is often needed for the design of new drug forms. Our device consists of a series of pumps connected with a mixer, which is able to produce variable inlet and an autosampler capable of collecting multiple particle fractions. In our model experiments, we saw 1.1% deviation from intended ratios while producing 15 different particle fractions, while reducing time needed to produce these significantly. After validating the device using a model ternary system (Fig. 1), the methodology was applied to the screening of several multi-component amorphous solid dispersions consisting of a poorly water-soluble API combined with several polymeric excipients. The high-throughput screening methodology was shown to enable rapid identification of the optimum composition of spray-dried ternary amorphous solid dispersions in terms of stability and dissolution rate enhancement.



References

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How to predict tablet properties: the endmost pharma challenge ?

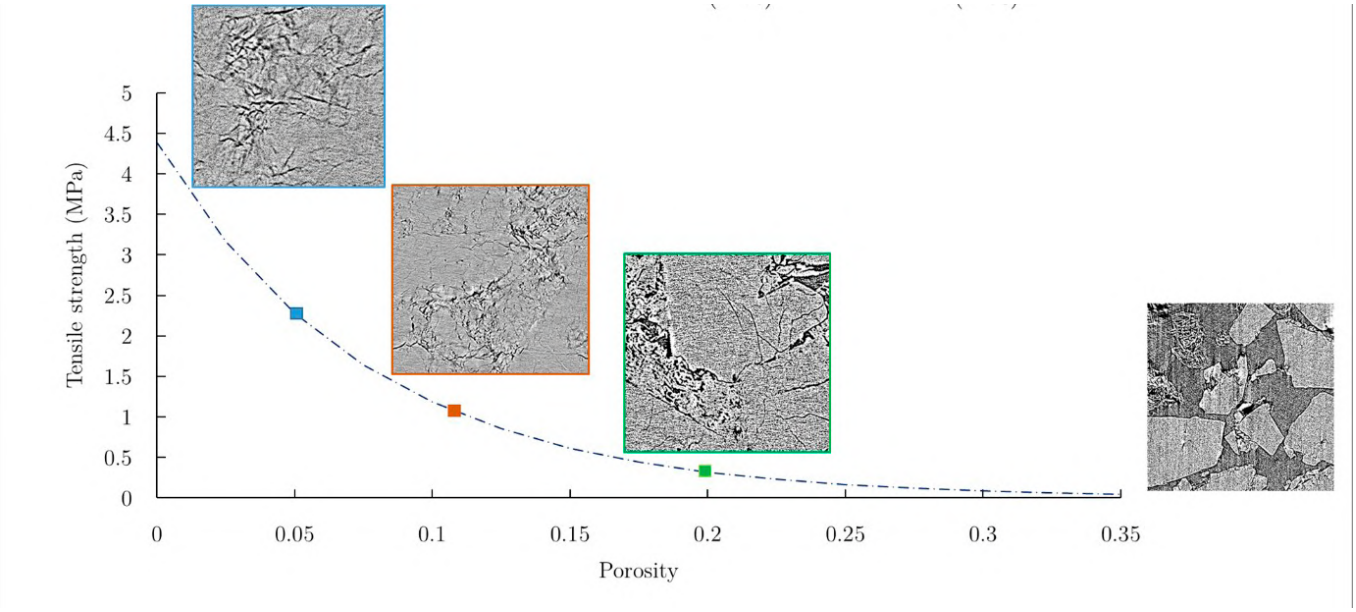
Mendez Torrecillas, Carlota (1); Robertson, John (1); Bordos, Ecaterina (2)
(1) University of Strathclyde, (2) University of Strathlcyde

Keywords | Direct compression, primary particles, machine learning,

Compression is one of the most widely used production techniques inside the pharmaceutical industry where tablets are the most preferred administration route for patients. Moreover, the recent focus on continuous direct compression lines, personalized medicines and advanced treatments, all at lower costs have converted direct compression into one of the larger researched topics of the field. However, this level of interest has not translated yet into a full understanding of how to predict the properties of a table.

The objective of this work is to tackle this challenge by combining statistical approaches such as machine learning to inform mechanistic models and help us to understand imaging results of real tablets obtained with X-ray Computed Tomography. Using this multi-level combined approach has already started producing relevant findings and furthered the understanding of the mechanisms of tablet production. This approach is taken by combining analytical techniques such as particle size characterisation, powder densities or flowability properties; producing tablets, analysing their properties including tomographic images and using available commercial software such as MATLAB and Orange for Machine Learning.

One of the main equations used in tablet compression is the Ryshkewitch-Duckworth equation which describes the evolution of the tensile strength which is inversely proportional to the porosity of the sample. This equation was originally developed for magnesia-stabilised zirconia and extrapolates mathematically a value at zero porosity. However, for common pharmaceutical materials is very difficult to reach low porosity experimental points which make this extrapolation very unreliable. On tomographic pictures, it was observed that zero porosity is obtained far before this point and breakage of the primary particles already happened (Figure 1). Nevertheless, the machine learning results showed particle size as one of the main properties influencing the tablet strength and when a smaller particle size of the same material was tabletted, breakage of primary particles was not observed. Currently, some of the compaction classifications used the material as a reference for compaction behaviour (Leane et al., 2015). However, our initial results show that a further investigation is required.



Use of Jump-tests for the characterization of the viscoplastic behavior of pharmaceutical powders during compaction

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(1) Université de Bordeaux

Keywords | Tabletting Pharmaceutical powders Viscoplasticity Compaction

Strain rate sensitivity (SRS) corresponds to the influence of the compaction kinematic on the powder behavior during compaction and on the final tablet properties. SRS is of particular importance during the scale-up at the industrial level as the production presses have often a kinematic very different from the one of the small scale presses used during development. Several physical phenomena can explain the SRS like viscoelasticity or viscoplasticity. This work focuses on the case of viscoplasticity.

Several experiments were performed using a compaction simulator (Styl'one Evolution, Medelpharm) in order to better characterize the viscoplastic behavior (figure 1). Three different pharmaceutical excipients were studied. They were chosen because they are known to be not viscoelastic and as such made it possible to focus on the viscoplastic behavior. First, monotonic compressions at different speeds were performed as usually done in the literature. They demonstrated that viscoplasticity was not constant during the whole compaction. Then, Jump tests were developed in order to study the influence of speed change during compression. Thanks to these tests, it was possible to show, for some of the products, the existence of transient phenomena during speed change that are similar to phenomena described in the literature as dynamic strain aging. Finally, introduction of long dwell-times during jump tests also indicated that all the products presented a stress relaxation even when the compaction was performed at a very slow speed. This was confirmed on long relaxation experiments (10h).

All these results showed that the viscoplastic behavior of pharmaceutical powders was more complex than described in the literature. Moreover, for all the products studied, even compactions performed at very slow speed must be considered as dynamic.

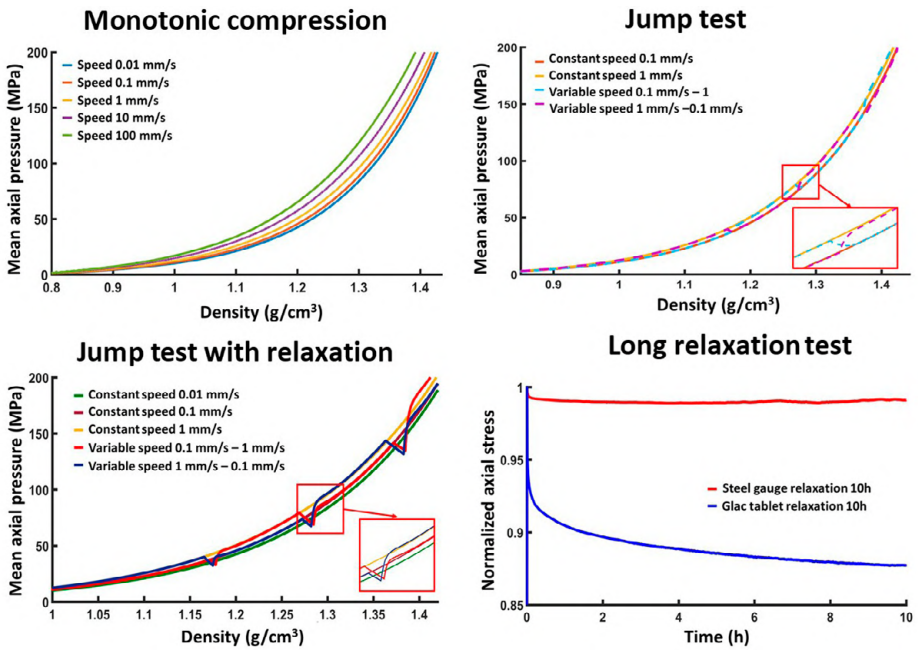


Figure 1: examples of results of the different tests performed on Lactose monohydrate

Modeling of compactibility of lubricated tablet formulations

Puckhaber, Daniel (1); Finke, Jan Henrik (1); David, Sarah (2); Serratoni, Mauro (2); Zafar, Umair (2); John, Edgar (2); Juhnke, Michael (2); Kwade, Arno (1)
(1) TU Braunschweig, (2) Novartis Pharma AG

Keywords | Tableting, Modeling, Lubrication

The compaction of pharmaceutical powders usually requires the addition of lubricants to reduce the ejection force occurring to remove tablets from the die. Applied lubricant particles adhere to the inner die wall and reduce the friction between tablet and die due to their comparably small shear strength. However, the internal addition of lubricants is also accompanied by a reduced tensile strength, especially when plastically deforming diluents are applied. Although some underlying mechanisms have already been qualitatively investigated, a quantitative prediction of the influence of lubricants on tablet properties has so far only been possible to a limited extent. In order to close this gap, binary mixtures of two commonly applied diluents (microcrystalline cellulose (MCC) and lactose (LAC)) and lubricants (magnesium stearate (MgSt) and sodium stearyl fumarate (SSF)) with systematically varied lubricant concentration were compacted and their compactibility investigated.

The results were used to develop an easily applicable empirical model which is able to precisely described the impact of lubricant concentration on compactibility of binary mixtures requiring only on a minimum of experimental data. Based on the equation of Ryshkewitch-Duckworth, a mathematical relationship between derived fit parameters and specific surface ratios of applied lubricant/diluent particles was established [1]. It could be shown that the knowledge of the compactibility profiles of the raw material and the highest lubricant concentration are sufficient to interpolate the compacibility profiles of unknown lubricant concentrations (Figure 1).

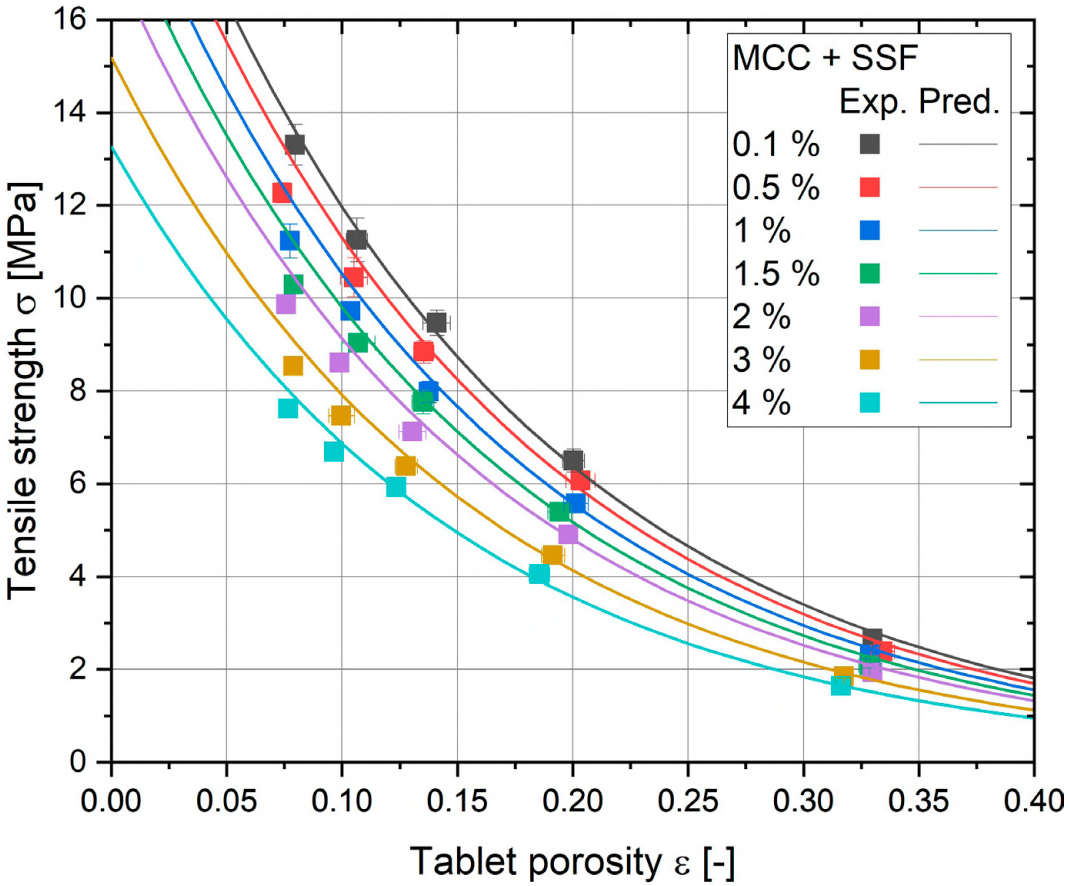


Figure 1: Comparison of predicted (pred.) and experimental (exp.) compactibility profiles of binary mixtures of MCC and SSF

Additionally, it could be verified that plastically deforming materials are more prone to lose mechanical strength due to the addition of lubricant due to their limited generation of unlubricated surfaces during compaction. In summary, the developed methodology displays a valuable tool for the development of new tablet formulations and should be further investigated for more complex powder mixtures to save time- and cost-intensive experiments.

[1] Puckhaber D., Finke J. H., David S., Serratoni M., Zafar U., John E., Juhnke M., Kwade A., 2022, Prediction of the impact of lubrication on tablet compactibility, International Journal of Pharmaceutics, Volume 617

Simple prediction method of tablet capping by continuous die wall stress measurement

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Keywords | Tableting, Rotary tableting machine, Capping

In the pharmaceutical industry, tablets are manufactured using rotary tableting machines. In this process, capping might occur depending on the operation condition and formulation. However, the detailed mechanism of capping remains unclear, causing that capping is a serious problem in industrial production. The objective of this study was to establish a method for predicting the occurrence of capping in rotary tableting machines. Microcrystalline cellulose (MCC), lactose (Lac), mixed powder of Lac and MCC (Lac+MCC), and lactose hydrate (DiLac) were used as model powders. The model powders were compressed by a rotary tableting machine with a compression interval of 2.0–1.3 mm. Uniaxial tensile tests of the obtained tablets determined the occurrence of capping. In this study, capping was assumed to occur when residual stress in the die exceeded the tablet strength in the die after unloading. To evaluate the residual die wall stress, we established a continuous measurement method of die wall stress in a rotary tableting machine by using a wireless digital telemeter (Fig. 1). The tablet

strength inside die was estimated using Rumpf's equation. Here, the tablet density inside die was determined by the geometrical calculation of the tablet height from the drawing of the rotary tableting machine. The tensile strength was estimated from the uniaxial tensile test of the resultant tablet. The tablet strength and residual stress inside the die were converted to von Mises stress to compare them. Fig. 2 shows the relationship between residual stress and tablet strength in the die for different powder materials. Based on the experiments, closed symbol indicated tablets without capping, while open symbol indicated tablets with capping. The proposed method could easily estimate the capping although there were some plots were not accurate. For this low accuracy, the appropriate correction of residual stress in the die would be necessary. We believe that these results will contribute greatly to the rapid detection of capping.

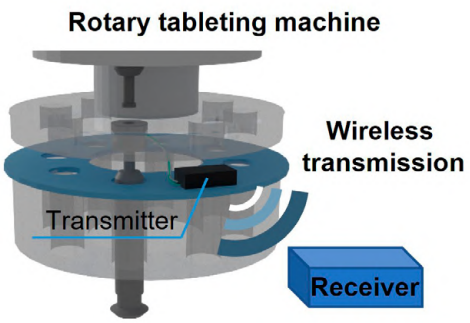


Fig. 1. Schematic diagram of measurement environment of die wall pressure.

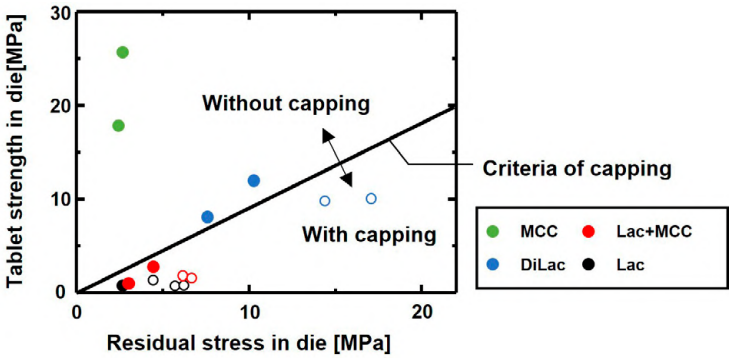


Fig. 2. Relationship between residual stress and tablet strength in die for estimation of capping.

Production and analysis of polymer-ceramic composite materials by a miniaturized fluidized bed granulation process

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Keywords | Fluidized bed granulation Composite material

Production and analysis of polymer-ceramic composite materials by a miniaturized fluidized bed granulation process
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The development and production of composite materials with desired properties is an important goal in the field of materials science and engineering. However, conventional processes are very labor-intensive, require high operating temperatures and pressures, and rely on the use of chemicals, some of which are harmful to the environment. Nature provides excellent inspiration for the development and production of functional materials. Biocomposites are characterized by exceptional mechanical, thermal, and optical properties while exhibiting multi-functionality, including functions such as adaptability and self-repair. To investigate the fluidized bed process as a potentially scaable process for the production of complex composites that mimic the biocomposite structure, a miniaturized apparatus with a conical fluidization gap was used to coat particles with a polymer layer. The miniaturized scale offers the possibility to use smaller amounts of potentially valuable bed material and the use of an organic solvent to process more complex polymers. As illustrated in Figure 1, the primary particles are fluidized in the plant and simultaneously coated by spray granulation. The granules are then hot compacted into a dense composite pellet to be tested for mechanical properties in the further process.

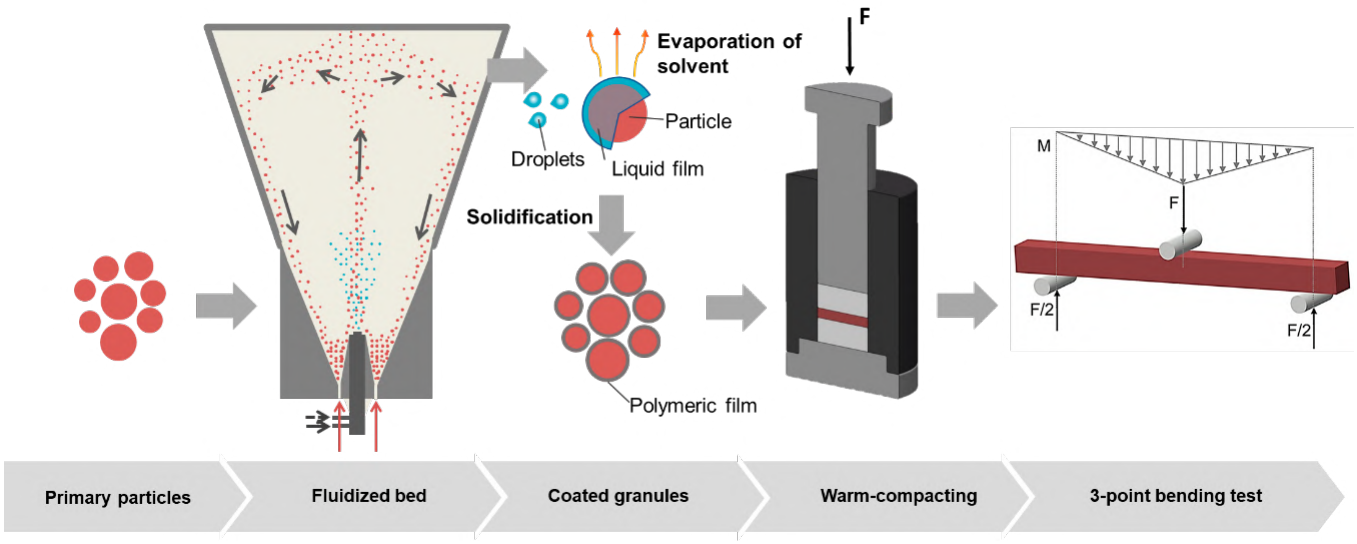


Fig. 1: Process route for the production of composites by fluidized bed granulation and warm-compaction.

In this work, the fluidization limitations of this new apparatus were determined (in terms of particle size and density), Al2O3- particles were successfully coated with a PVB-polymer and the mechanical properties of those produced composites were investigated. Ideal process parameters for the coating process and the optimum composite composition, in terms of polymer content, were identified to enable optimum mechanical properties of the composite pellets.

We gratefully acknowledge financial support from the German Research Foundation (DFG) via the collaborative research center SFB986 (project number 192346071).

Dependence of Layer Build-up on Spray Parameters in Fluidized Bed Spray Granulation

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Keywords | fluidized bed spray granulation, coating, spray parameters, surface structure

In various industries, such as pharmaceutical, food or chemical industry, fluidized bed granulation or coating is applied for the production of high-quality, tailor-made particles. By changing the overall process conditions and therefore influencing the solid-liquid interaction between the fluidized particles and the injected liquid, defined particle structures can be produced. An important set of influencing factors consists of all parameters related to the spray, including liquid properties, process parameters like spray rate and spray air pressure, and the configuration of the nozzle.

To investigate the influence of different spray parameters on particle structure and to integrate the solid-liquid interaction into simulation cases, coating experiments are performed in a lab-scale fluidized bed (Glatt, Germany) with different nozzle configurations and varied spray parameters. In order to investigate the growth of the particles and structure of the coating layers, samples are taken from the process chamber over the course of the experiments. The coated particles are analyzed regarding their surface structure and coating layer thickness via optical measurements, including confocal laser scanning and scanning electron microscopy. When spraying a sodium benzoate solution onto Cellets®500 particles, an increasing surface roughness can be observed over time. This indicates that the phenomena involved in the solid-liquid interaction between particle surface and solution droplets during the coating process, such as droplet spreading and crystallization of the sodium benzoate, cause certain deviations from a perfectly smooth coating layer surface. For a better understanding of how the droplets are distributed in the process chamber and interact with the solid particle surface, the properties of the spray solution and the spray cone angle are analyzed, with the latter showing a strong dependence on certain spray parameters, such as spray air pressure and liquid spray rate.

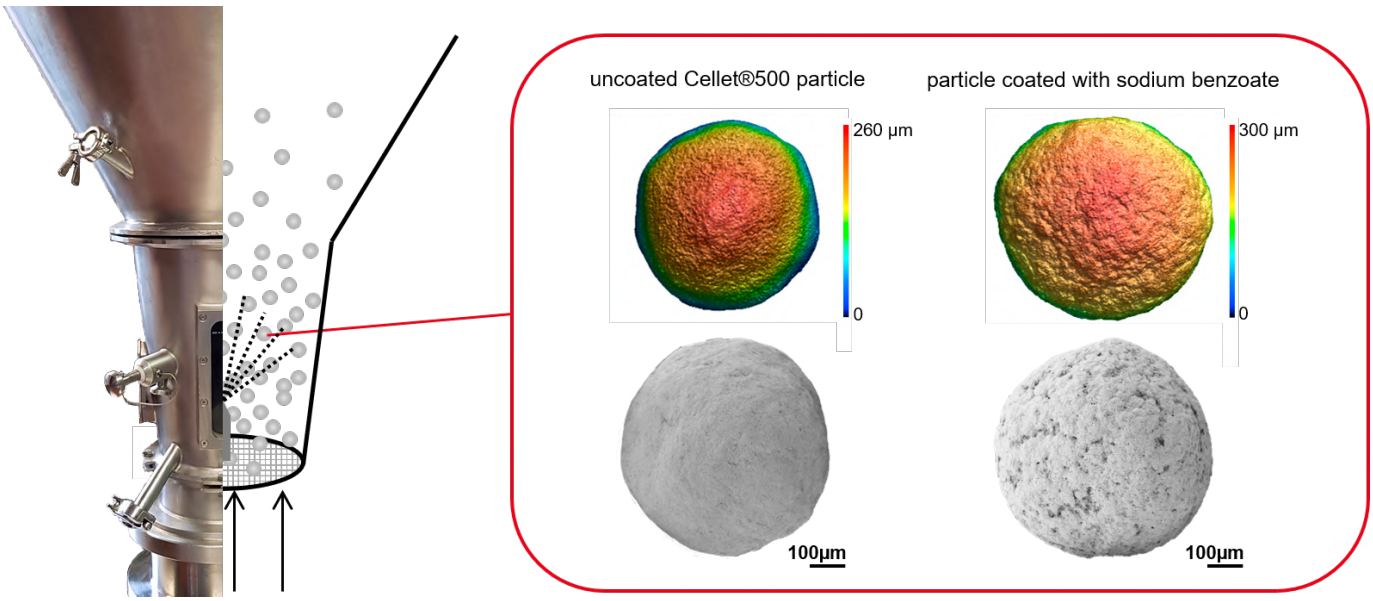


Fig. 1: Fluidized bed set-up and surface structure of uncoated and coated particle.

We gratefully acknowledge financial support from the German Research Foundation (DFG) via the graduate school GRK 2462 (project no. 390794421).

Development of uniform ceramic thin films by planetary spin coating method

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(1) National Institute of Technology Oita College

Keywords | Ceramics film, Spin-coating, Waviness, Piezoelectric elements, Fuel cells

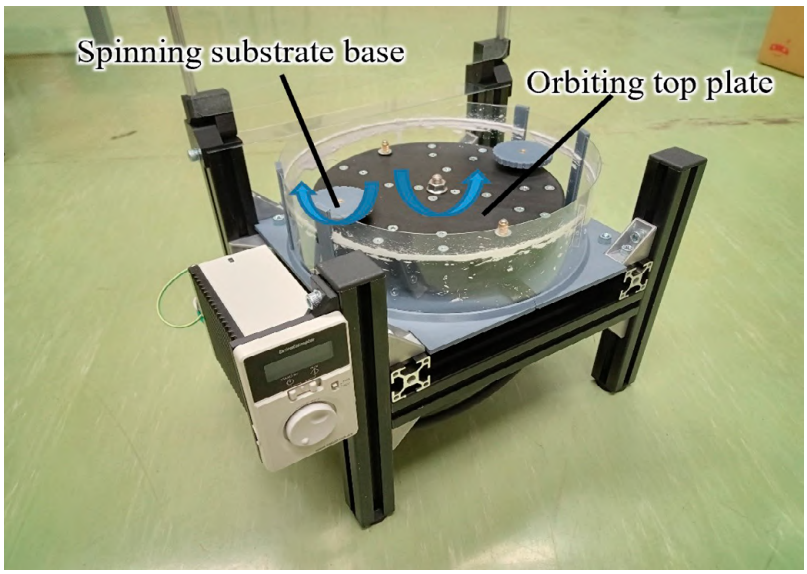
In recent years, the demand for small sensors for IoT, fuel cells, and all solid-state batteries has been increasing due to the SDGs. We have been developing high performance ceramic thin film fabrication technology for the above products by applying micro-patterning technology. However, it has been a challenge to improve the efficiency of thin-film fabrication, and we have attempted to introduce spin-coating into ceramic thin-film fabrication. Spin-coating is a technique for producing thin films on a substrate by rotating the substrate coated with a material liquid at high speed. Since only centrifugal force is used as a processing force, it is difficult to form a thin film directly from a high-viscosity slurry material such as ceramic slurry.

To solve this problem, we develop a planetary spin-coating technique, which is a thin-film forming technique for high- viscosity slurry materials, using the shear force control by the rotation speed of the orbiting and spinning.

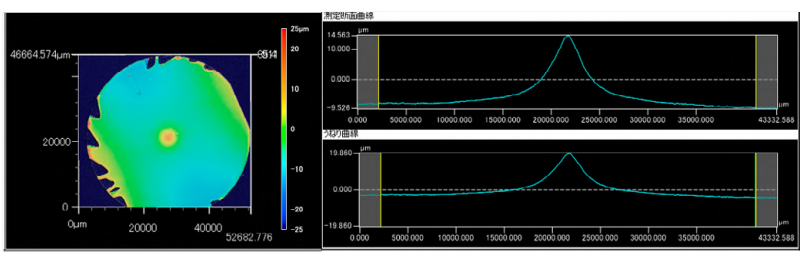
Figure 1 shows a photograph of the planetary spin coater developed in this study. The planetary spin coater mainly consists of an orbiting top plate and a spinning substrate base, and the centrifugal force on the substrate can be controlled by changing the rotation speed of the orbiting and spinning.

Figures 2(a) and 2(b) show the results of 3D scanning of ceramic films prepared by conventional spin coating and planetary spin coating, respectively. The ceramic film was prepared by coating a water-based slurry of alumina(Al2O3) and polyvinyl alcohol on a glass substrate, and the average particle size of the alumina used was 2.25 µm. The ceramic film produced by conventional spin coating has a central convexity, while the film produced by planetary spin coating has a uniform thickness. The arithmetic mean waviness Wa is 3.646 µm for conventional spin-coating and 0.152 µm for planetary spin-coating, which means that the waviness is reduced to about 4%.

The results show that planetary spin-coating is a very effective method, and it is expected to make a significant contribution to the performance of ceramic products in the future.

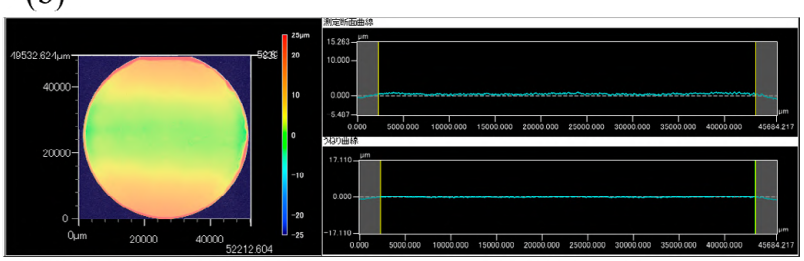


(a)



Height contour map Waviness

(b)



Height contour map Waviness

A DEM - Monte Carlo Approach for Optimization of Inter- and Intra-Tablet Coating Variability in a Lab-Scale Tablet Coating Process

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Keywords | DEM, Tablet coating, inter CoV, Intra CoV, Monte Carlo simulation

Tablets are the most common form of drug products. They are coated for various reasons, e.g., to mask the taste of the active pharmaceutical ingredient (API), protect against the environment, control the release of the API or add the API (active- coating). For functional and active coatings, coating uniformity is essential. For active coatings, the inter-tablet coating variability (the variance in the coating mass between

tablets in a batch) is important whereas both the inter- and intra-tablet coating variability (the variance in the coating thickness on a single tablet) can be important for functional coatings.

Although several experimental studies have related tablet coating process parameters to the final product quality, e.g., the dissolution rate (Radtke, Wiedey, and Kleinebudde 2019; Barimani, Šibanc, and Kleinebudde 2018), the effect of process parameters on the variability in the coating thickness on individual tablets has not been considered. Discrete Element Method (DEM) simulations have shown to be a very valuable tool (Boehling et al. 2016; Kumar, Freireich, and Wassgren 2015; Freireich et al. 2015) in better understanding coating processes, though they are very time-consuming for long coating times and large batch sizes.

In this work, we address the shortcomings mentioned above by developing a fast simulation workflow to predict the inter- and intra-tablet coating variability based on DEM simulations. To this end, we performed a computational DoE on a lab scale tablet coater to investigate the effect of process parameters on the intra- and inter-tablet coating variability. From each DEM simulation, we extracted distributions related to the time spent in and outside the spray zone as well as the amount of deposited solid on each tablet and its location on the tablet. These distributions are then used in a fast Monte Carlo-based algorithm to simulate the evolution of the inter- and intra-tablet coating variability.

Besides verification of the workflow, our simulations shed light on the intra- and inter-tablet coating variability and their dependence on process parameters. We find that the pan speed has the biggest effect on the intra-tablet coating variability while the spray rate has the biggest effect on the inter-tablet coating variability.

A mathematical model of contact spreading

Freireich, Ben (1)
(1) Origin Materials

Keywords | Particle Coating, Population Balance Modeling, Contact Spreading

In many particulate processes a liquid is added to the surface of particles, e.g. tablet coating, granulation, coking. In these applications a variability in coating content can occur. Interparticle coating variability occurs when particles have a different coating amount than others in population. Likewise, intraparticle coating variability occurs when the thickness of coating varies point-to-point on the surface of an individual particle.

Both inter- and intraparticle coating variability occur for many different reasons. When the coating is added via a spray, interparticle coating variability results from the varied time particles spend in the spray region and between visits to the spray region (Freireich & Li, 2013). Intraparticle coating variability can occur due to preferred orientation of particles as they pass through the spray (Freireich et al., 2015). Both have similar analogs in systems without spray coating.

In this work we study an additional mechanism of coating variability: contact spreading (Yusof et al., 2019). If coating does not dry, solidify, react, or adsorb immediately, some can be transferred when particles contact. Spreading of coating due to contacts is called contact spreading.

In this presentation we derive a several mathematical models of contact spreading under simplifying assumptions of varying complexity. Our hope is to develop a conceptual framework for discussing contact spreading, as well as a theoretical basis for distinguishing dominant mechanisms based on empirical data.

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Yusof, N., Green, J., Pitt, K., Arjmandi-Tash, O., Campbell, A. I., Ahmadian, H., Tantawy, H., & Smith, R. (2019). A novel method for the analysis of particle coating behaviour via contact spreading in a tumbling drum: Effect of coating liquid viscosity. Powder Technology, 351, 102-114. https://doi.org/10.1016/j.powtec.2019.03.044

Multi-layer particle coatings using Wurster fluidised bed for personalised medicine

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(1) University of Chemistry and Technology Prague, (2) Zentiva, k.s.

Keywords | Coating, Wurster process, Multi-layer, Personalised medicine

The current challenges in pharmaceutical development like proper dosing, personalization, dissolution control, and multi-API systems can be solved with coated particles using the Wurster fluid bed technology. The vast choice of the carrier, API and excipient combinations offers great variability in the material and release characteristics of the product but implies extensive testing for suitable process parameter setup. The process variability allows for the coating of APIs and excipients in the form of nanosuspensions or true solutions to produce multi-layer particles including active, protective and insulating layers to reach environmental endurance and overall product stability.

In this study, we test multiple polymers (HPMC, Eudragids, PVA, PVP) in combination with an immediately soluble API in a form of nanosuspension or salt. Therefore the product dissolution characteristics are driven primarily by the excipient choice and the layer properties. The coating carriers were chosen concerning the API chemical stability. These carriers of different input size 100–700 µm were coated using the MiniGlatt coater with Wurster insert by Glatt. Coatings were observed and analyzed by bulk dissolution, UV imaging, microtomography, and scanning electron microscopy for particle cuts. Evaluated parameters were homogeneity, thickness, porosity and layer adherence.

Final products were composed of two or more layers with modified release profiles. Both immediate and delayed release, including pH sensitivity, was achieved. Such effects were possible thanks to the high homogeneity of the layers that did not allow layer mixing during the coating process. Protective layers mitigate the undesired API diffusion in the dissolution process. The API nanosuspensions have shown good re-dispersibility upon release from the coatings. Utilizing the nanosuspension increased dissolution rate given by the particle size results in the release readily modified by the excipient and subsequent retardant layers.

Exploring the electrochemical behavior of Bi-based cathodes for the electrocatalytic reduction of CO2 to formate

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(1) Universidad de Cantabria, (2) Universidad de Alicante

Keywords | CO2 reduction; Formate; continuous operation; residence time; CO2 concentration; industrial implementation

Carbon dioxide (CO2) electrocatalytic reduction to formic acid (HCOOH) or formate (HCOO-) is currently considered as one of the most promising strategies to obtain value-added products from captured CO2. In this sense, both reaction products can be used as raw materials for several industries and they are gaining much importance as reactants in low-temperature fuel cells and as hydrogen carrier molecules (Irabien et al., 2018). In the last years, a lot of efforts have focused their attention on the study of new electrode configurations, electrocatalytic materials, and conformations of the electrochemical reactor (Díaz-Sainz et al., 2020). However, the influence of the residence time of reactants through the electrochemical and the CO2 concentration in the input stream, key variables in this technology, are rarely assessed in this field. In this context, the main goal of this work is to analyze the influence of these variables employing Bi-carbon supported nanoparticles in the form of Gas Diffusion Electrodes for the continuous CO2 electroreduction to HCOO- in terms of different key metrics.

Firstly, the influence of residence time, studied by the effect of the flow rate in the CO2 input stream d in the range from 50 (residence time of 12.6 s-1) to 400 mL·min-1 (1.6 s-1), as depicted in Figure 1a. In this way, the best result in terms of figures of merit was reached for a residence time of 4.2 s-1, which corresponds to a CO2 flow rate of 150 mL·min-1 in the input stream, reaching a Faradaic Efficiency for HCOO- of up to 96 %.

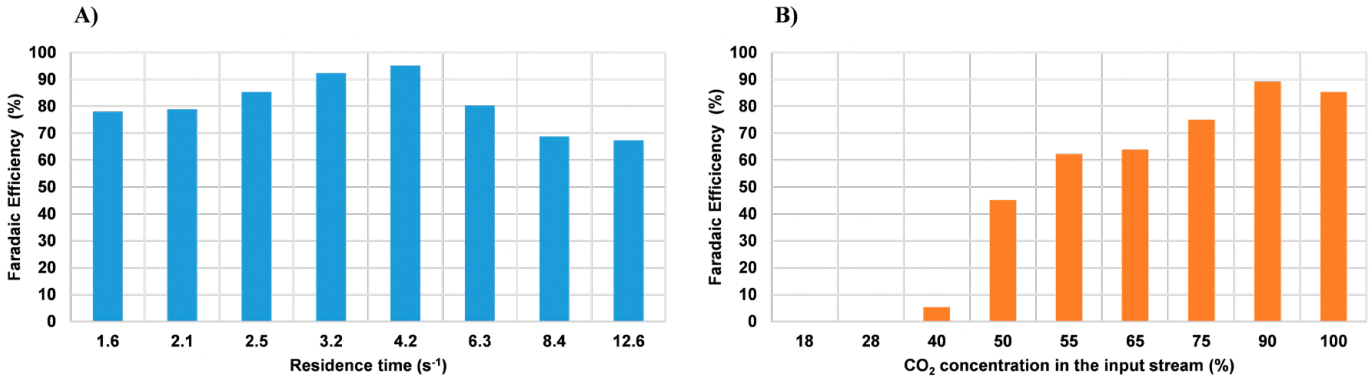


Figure 1. Influence of (a) the residence time and (b) the CO2 concentration in the Faradaic Efficiency.

Subsequently, the influence of the CO2 concentration in the input stream is also analyzed through this work. As can be shown in Figure 1b, similar results are obtained when the concentration of CO2 in the input stream is higher than 75 %. In contrast, further research is still needed to collect more data about the effect of potential contaminants from flue gas emissions for the future implementation of this electrochemical process at an industrial scale.

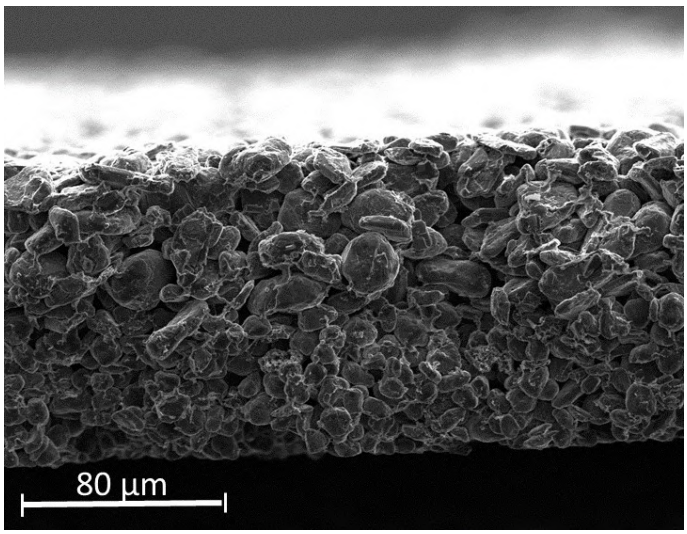
Different particle sizes of graphite in multilayer anodes for lithium-ion batteries

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(1) Technical University Braunschweig, Institute for Particle Technology

Keywords | multilayer-anodes, lithium-ion batteries, tortuosity, ionic resistance

Lithium-ion battery cells with high specific capacity and energy density are one of the current research priorities in industry and science. The challenges of these so-called “thick” electrodes are transport limitations within the electrode: lithium ions cannot reach the deeper layers of the electrode coating, which leads to a drop in performance during charging and discharging [1,2]. This phenomenon is a major challenge in the further development of future high-capacity electrodes. One possible solution is to use multilayer electrodes with different layer properties. In this way, pathways for the Li-ions can be created that reduce the ionic resistance of the electrode and ensure an increase in fast charging capability.

This work focuses on two-layer anodes with two different graphite particle sizes (x50 = 10 µm; x50 = 16 µm). First, both single-layer references and different configurations of two-layer anodes were processed. For the double-layer anodes, the two types of graphite were combined and their amount in the different layers of the anode was varied. The mechanical, electrical and electrochemical layer properties were investigated. However, special attention was paid to the characterization of the structural layer properties. Here, the electrodes were examined for their ionic conductivity by means of impedance spectroscopy and, thus, conclusions were drawn about the pore network and the tortuosity of the electrodes. It could be shown, that the combination of the two graphite particle sizes in the lower layer resulted in a significant change of the anode properties. It was observed that the two-layer anodes with a different mixture of both graphite types in the lower layer and only the coarser particles in the upper layer showed a significant decrease in ionic resistance compared to the single-layer reference anodes. One assumption is that by a combination of the different particles sizes a specific number of pores could be created, resulting in an improved pore network and reduced tortuosity.



Investigation and evaluation of the effect of different compressions on blend anodes containing Graphite and Hard Carbon

Strzelczyk, Nanny (1); Kwade, Arno (1); Gottschalk, Laura (1)
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Lithium-ion batteries are the leading technology for energy storage devices such as hybrid, plug-in and fully electric vehicles. Intensive efforts have been made to improve the energy density of lithium-ion batteries (Kwade et al., 2018). The development and application of carbon-containing active material mixtures are promising prospects for use in high-energy anodes. The motivation of using blend anodes containing different active materials is based on the combination of the advantageous properties of these materials. Graphite shows an ordered structure of graphene layers whereas Hard Carbon has a disordered turbostratic structure leading to a higher specific surface area. Due to the different microstructure of Graphite and Hard Carbon particles, major differences in the structural properties of the electrode layer and the cell performance can be observed (Chen et al., 2021).

In this study active material blend anodes containing Graphite (90 wt-%) and Hard Carbon (10 wt-%) are produced and calendered to different densities to investigate the availability of the interporous structure of the Hard Carbon in the electrode layer depending on the calendering compressions. In order to have a closer look on the effect of the different particle morphologies in the blend anodes, the electrodes are analyzed with regard to their mechanical and electrical properties and the cell performance. Furthermore, the pore size distribution and the tortuosity are analysed to get an insight into the pore network of the electrode.

This investigation reveals that the addition of Hard Carbon leads to an improvement of the electrical conductivity as well as the adhesive strength of the anode. Moreover, it could be shown that the beneficial effect of the Hard Carbon in blend anodes is dependent on the degree of compression of the anode. A lower compression is not only enhancing the adhesive strength but reduces the tortuosity which is crucial for the electrochemical performance.

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Kwade, A. et al. 2018. Current status and challenges for automotive battery production technologies. Nat Energy. 3 290–300.

FLASH COMMUNICATIONS

Improving the ionic conductivity of hybrid solid polymer electrolytes by surface modified filler particles

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(1) Technische Universität Braunschweig, Institute of particle technology

Keywords | all-solid-state batteries, hybrid solid polymer electrolyte, silane-based particle modification

According to Kato et al. all-solid-state batteries are considered as next-generation battery as they offer the potential to further increase the possible energy density due to the use of a lithium anode. Furthermore, no flammable liquid electrolyte is used, which improves the battery safety. As presented by Winie et al., solid-polymer electrolytes offer a great potential as they are of low costs, can be efficiently processed and are stable against lithium. So far, they are not commercially used in large scale due to their low ionic conductivity at room temperature (RT) and low transference number. Therefore, it is a current topic of research to increase the ionic conductivity by the incorporation of inorganic filler materials in the polymer matrix.

In this contribution, we present a solvent-based manufacturing route for hybrid solid polymer electrolytes (HSE) with active and passive inorganic filler materials. The influence of the filler particle properties like particle size or, in case of active materials, intrinsic ionic conductivity as well as filler content on the resulting HSE ionic conductivity were studied. Next to that the effect of a silane-based surface modification of the filler particles on the interphase resistance between the filler particles and the polymer matrix was investigated. The silane modification stabilized the filler particles and a homogeneous distribution and thereby enhanced transport properties could be achieved. A maximum ionic conductivity of 10-4 S cm-1 at RT was obtained for a HSE with silane modified Li6PS5Cl filler particles for a filler content of 2.5 vol.-%. Different filler types and filler contents were further investigated in cathodes and the effect of the filler addition was studied via electrochemical impedance spectroscopy. In addition, cathode properties like the electrical conductivity, adhesive strength and electrochemical performance at various cycling temperatures were studied.

References

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Modelling the twin screw granulator as mixing aggregate - On the inherent balancing of feeder fluctuations

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Keywords | Wet granulation, Continuous manufacturing, Twin screw granulation, Model development

Granulation is a standard unit operation to modify the particle size for improved material handling and dosing properties in powder processing [Serno et al. 2007]. For pharmaceutical technology, the method with the highest practical impact is wet granulation, while the research focus over the last decades has been on continuous manufacturing with twin screw granulators [Djuric et al. 2008] . A major advantage is the combination of different unit operations including homogenization. Thereby feeder fluctuations are balanced inherently during processings. However, since this property is not well understood, typically pre-mixes are used to secure product homogeneity. This requires an additional process step. The aim of this study is to model the mixing capacity of twin screw granulation in order to facilitate a direct wet granulation process.

Model development is based on the convolution of the feeder mass flow with the residence time density distribution of the twin-screw granulator. Hereby the width of the residence time defines the axial distributive mixing capacity of the granulation process. Preliminary, the determination of the residence time distribution function has been identified as crucial aspect for the experimental validation of the developed model. Furthermore, the basic fluctuation of the granulation process is limiting the maximum potential to balance feeding disturbances.

The characterization of this phenomenon in dependency on the process parameters is a central objective of the presented experimental investigations. Based on the results, the model for prediction the balancing potential of the twin screw granulation process is extended. Furthermore, an online optical system has been implemented as process analytical technology for the residence time determination. Thereby an accurate determination of characteristic parameters (Bodentstein-number Bo, mean residence time) for the description of the

residence time distribution was feasible. Finally, the improve experimental set-up was utilized to verify the extended balance model for feeder fluctuations during continuous twin screw granulation.

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[Serno et al. 2007] Serno, P., Kleinebude, P. and Knop, K., 2007. Granulieren: Grundlagen, Verfahren, Formulierungen, ECV Editio-Cantor-Verlag, Aulendorf.

Fractionation with respect to size and density by means of a classifying aerodynamic lens

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Keywords | aerodynamic lense, separation, aerosol mixtures, classification, multi-dimensional characterization, differential aerodynamic particle sizer

Aerosol mixtures of various species were fractionated using a classifying aerodynamic lens setup. Classifying aerodynamic lenses (CALs) separate particles based on their relaxation time, while showing a differential transfer characteristic. This principle was already applied for measuring aerodynamic particle size (Babick et al., 2018), but was, to our knowledge, not yet considered for application in material separation processes.

In this work, particles species of, e.g., Copper and Silicon, are mixed before being aerosolized in a dry disperser. From the feed aerosol a sample is taken via filtration, this sample is compared with a second sample which is taken at the product stream at the exit of the CAL. Subsequently, the separation efficiency is measured using off-line methods such as gravimetry, scanning electron microscopy and energy-dispersive X-ray spectroscopy. This process is depicted in Figure 1.

First results showed enrichment of silicon in the copper-silicon-mixture, while also reducing the geometric standard deviation of the size distribution.

Our results indicate that a separation process like this could be applied during the recycling of composite-materials.

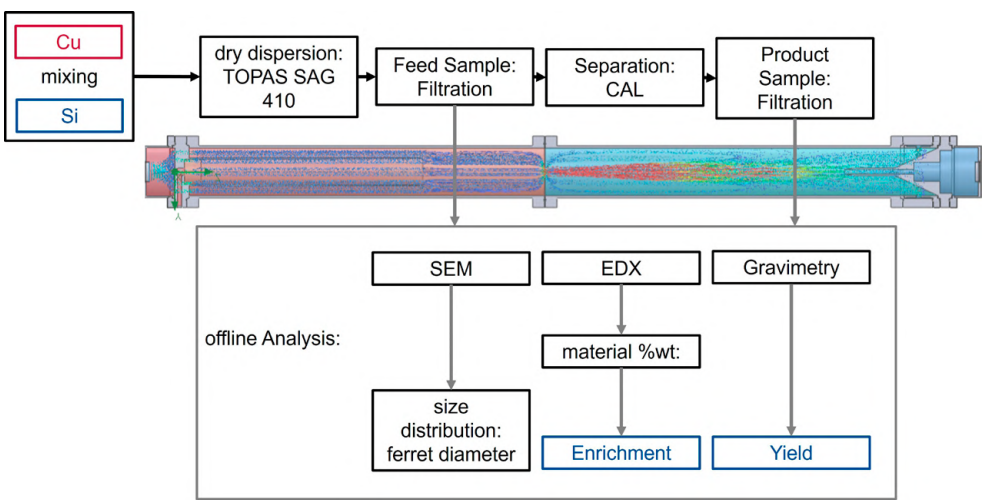


Figure 1 Principle for the offline determination of enrichment of silicon in a Si-Cu-aerosol mixture

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Acknowledgement

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Predictive simulations of wear in SAG mills using Rocky DEM

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(1) ESSS, (2) Anglo American

Keywords | Discrete element method (DEM), semi-autogenous grinding (SAG) mill, wear, grinding, erosion.

In semi-autogenous grinding (SAG) mill operations, one major consideration is the wear that occurs on the internal wear components, the lifter bars, shell plates, and grates. The significant costs associated with this maintenance, as well as its impact on equipment availability are the main reason why this field draws great attention from researchers and engineers, who are constantly trying to find ways to improve performance, minimize wear and increase the life of the wear components while minimizing the potential for catastrophic damage to the lining system. Wear is caused due to the collisions and abrasion between the balls and the granular material (ore) and surfaces of these wear components and can be a function of various parameters, including the operating conditions, the wear liner material, or the size distribution and abrasiveness of the granular material. In this work, the aim is to show how the milling process is modeled using the Discrete Element Method (DEM) approach. Simulations are carried out using Rocky DEM's technology, which allows the representation of surface modification due to wear throughout simulation time. Balls and ore are modeled using spheres and sphero-polygon particles in the DEM solver, respectively. The Archard model is used to compute the volume that will be removed from equipment's surfaces, where the amount of volume to be extracted is a function of the shear work applied by particles. The simulation times are only a few hundred seconds, once it would have been prohibitive to run simulations for the entire duration of the wear components life. Experimental data collected from laser scans are used to identify regions where wear is the greatest, to evaluate how wear progresses over time, as well as to serve as references to calibrate the DEM model. Once the calibrated model is obtained, simulations of different shell liner profiles are performed using the same conditions from the base case scenario. Results enable finding the shell liner profiles that present the greatest wear life for the lifter bars and plates while minimizing the impact on mill performance.

Influence of the drug load on pharmaceutical blends processability

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(1) Granutools

Keywords | Packing, flowability, tableting

The tableting process of pharmaceutical powders involves different steps, the most important is the filling of the die and the compaction of the powder. Good flowing properties of the powders are essential to guarantee a consistent flow through the die. Indeed, the filling of the die is of great importance to obtain low variability of the tablet mass and guarantee an uniform drug load to the patient.

The drug powder has usually a flowability too low to be directly processed, which requires the addition of specifically designed excipients to improve the flowability. A better understanding of the influence of the drug load on the blend behaviour is thus a key parameter to help developing formulation with higher tableting performance.

We investigated the influence of the drug load on the packing properties and flowability of pharmaceutical powders. Blends of Acetaminophen (APAP) and silicified microcrystalline cellulose (Prosolv SMCC90) have been produced at different drug loads of APAP. First, the influence of the fraction of APAP on the packing dynamics and flowability of the blends is investigated with respectively the tapped density and the rotating drum methods. Then, tablets are produced in a rotary press (Natoli® NP-RD30) to evaluate the effect of modification of the powder behaviour on the mechanical properties of the tablets, as illustrated in Figure 1.

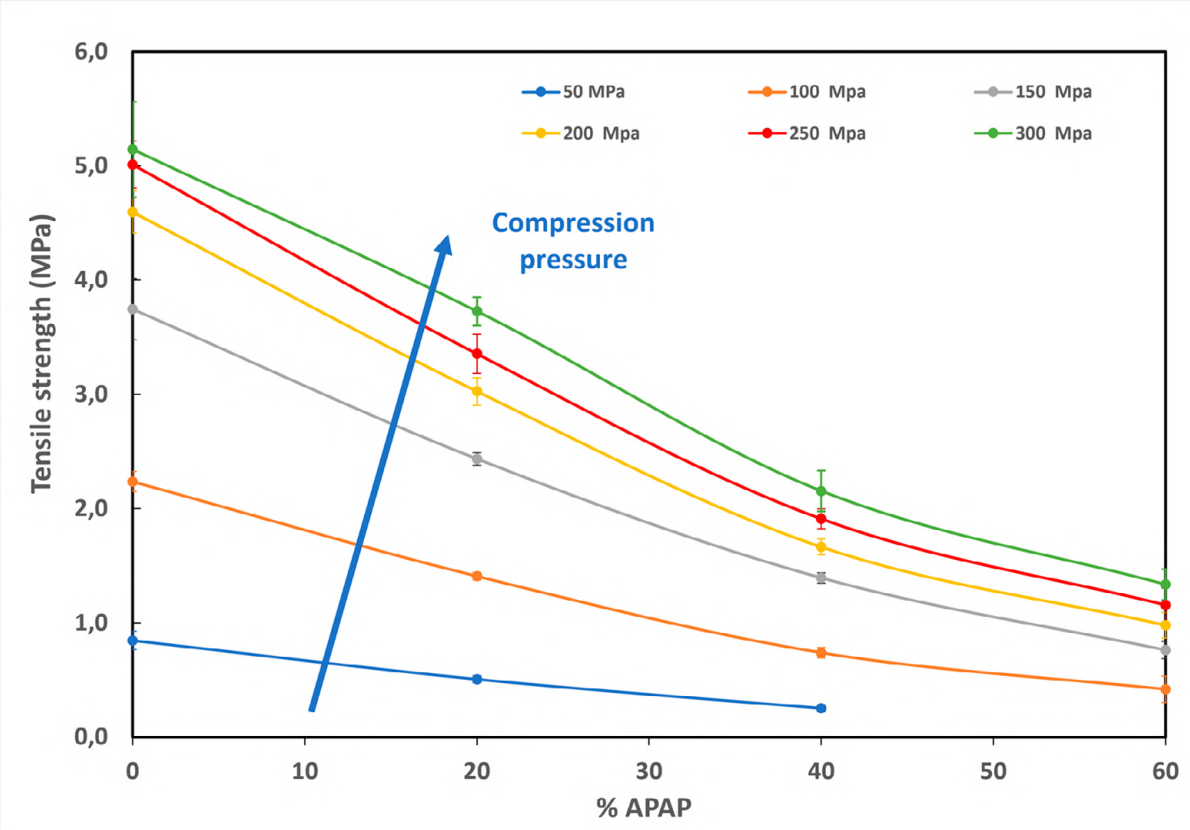


FIGURE 1: Tablet tensile strength obtained at different compression pressures as a function of the drug load (%APAP).

With these results, we highlight the importance of a good powder characterization. Indeed, assessing beforehand the packing and flowing behaviour of the blends will allow selecting suitable formulations to produce tablets meeting the quality criteria.

Investigating Microstructure and Dissolution Behavior of Granulated Urea Fertilizers

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(1) Purdue University

Impact of unloading kinematics on the occurrence of capping during the compaction of pharmaceutical powders

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(1) Université de Bordeaux, (2) Roche

Keywords | tableting, pharmaceutical powders, capping, defects, kinematics

Capping is a common defect that can occur during the manufacturing of pharmaceutical tablets (figure 1). Several studies showed that decreasing the unloading speed of the manufacturing cycle plays a role in the occurrence of such defects.

Following this idea, we study in this work the influence of the unloading step on capping.

Experiments were performed using a compaction simulator (Styl'one Evolution, Medelpharm) and model formulations presenting capping. First, measuring the die wall pressure made it possible to detect precisely that tablets capped just after the end of the unloading part of the cycle (i.e. some milliseconds only after the axial pressure came back to zero). Then it was confirmed that, by changing only the speed on the unloading part of the compaction cycle it was possible to stop capping (figure 2) and that this effect was not due to a dwell-time

increase. To go further and understand exactly this influence, we developed a two-step unloading phase where it is possible to slow down only the last part of the unloading.

Thanks to this new cycle, we showed that to mitigate capping it is not necessary to slow down the whole unloading step but that it is sufficient to decrease only the speed at which the contact between the punches and the tablet is lost. Capping seemed due to dynamical effects related to the release of the axial pressure. The modification of the unloading step promotes in some cases changes in the tablet global structure but its role in capping mitigation does not seems to be related to the change in the tablet macroscopic properties.

This work made it possible to improve the understanding of capping. Moreover, the two-step unloading cycle gave a new idea for possible modifications that could be done on rotary presses in order to mitigate capping.



Figure 1: Example of capped tablet

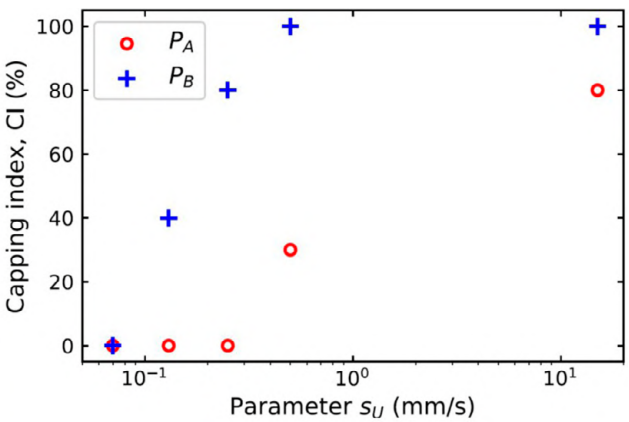


Figure 2: influence of the unloading speed (Su) on the capping index (% of capped tablets among the produced tablets using these conditions) for 2 different main compaction pressure

Feeding and Compaction – Differentiation of Damaging Influences on Coated Pellets in Tableting Machines

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Multiple Unit Pellet Systems (MUPS) are frequently applied to deliver drugs in small entities (pellets) with modified drug release. To achieve this, drug-containing pellets are coated with functional polymer formulations that delay or sustain the drug release. Enteric coatings which do not dissolve under gastric conditions, protecting the drug from the harsh conditions or the stomach from the impact drug, are frequently applied. In the intestine, the coatings dissolves and release the drug immediately. However, to protect the core, the coating layer around the pellet must be perfectly intact when it is applied to the patient to prevent any premature drug leakage. To form oral dosage forms of highest convenience, pellets are frequently blended with (excipient) powders and compressed to tablets. The necessary steps in this production process chain, in turn, exert stresses to the coated pellets and may infringe their coating integrity.

In this study, the influence of different sub-processes in rotary tablet presses, namely feeding and compaction, on pellets coating integrity was systematically elucidated. On the formulation side, different pellet coating approaches, including the application of outer cushioning layers, and different deformation behaviors of the blended powders were studied. The compaction sub-process showed the greatest influence on coating integrity by the main compression stress. Here, an additional (non-enteric) cushioning coating could shield the enteric coating from mechanical stresses and improve the integrity of pellets also at high compaction stresses.

In the feeding process, a forced paddle feeder was investigated as it is commonly applied on industrial rotary presses. Here, more shear stresses are applied to the pellets. The shape of the spokes of the paddle wheel as well as small changes in characteristic geometric measures

within the feed frame showed a distinct influence on the damage of the pellet coating.

Coating damage, measured as premature model drug release, was shown to increase linearly with residence time in the stressing volume of the paddle feeder. Further, the additivity of the damaging effects of these subsequent sub-processes, feeding and compaction, are studied.

Effect of Moisture on Physical Properties of Food Ingredients and the Resulting Strength of Compacts

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Keywords | compaction - moisture impact - formulation parameters - process parameters

The compacting of powders is a process used to convert powdery materials into a suitable form for industry and customer needs. Mechanical properties of resulting food compacts depend on the complex interplay of material, formulation, conditioning and process parameters. The high number of material and possible property variations make a general classification system of mechanical characteristics necessary. Moisture plays a major role in the alteration of powder and compact properties in the food industry, leading to changing flow behavior and compact strength. A general understanding of the susceptibility of materials toward moisture is to be established to improve and accelerate the whole development and production process, especially in case of formulation adaption.

In this work, the influence of significant components on the final compact properties are investigated, especially taking the effect of varying moisture content into account. In addition to the dominating product property tensile strength, glass transition temperatures (Tg) of formulations containing crystalline and amorphous components are determined by differential scanning calorimetry (DSC) to elucidate their explanatory power towards their effects on the compaction processes and the compact strength.

The results pronounce the different extents of the influence of moisture content on the deformation behavior and resulting strength of the compacts of different component classes. The systematic comparison of compaction results of the individual components with those of specific formulations enable the derivation of essential rules of mutual influence. DSC measurements support the direct effect of Tg on the binding power of amorphous components and, by that, on the compact strength. Additionally, blend behavior can by explained based on the mixing ratios of crystalline and amorphous components.

Effect of multilayer sintering on porosity and electrical resistivity of copper films prepared from nanoparticles synthesized from arc discharge

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Keywords | Thin film, conductive inks, copper nanoparticles, film porosity

Copper nanoparticles (NPs) are considered as an alternative for gold and silver NPs due to their low electrical resistivity and lower cost. A direct current arc discharge method has been developed to produce high-purity metal NPs with high production rates (Stein et al., 2018). In our previous study, we applied copper NPs synthesized from arc discharge to prepare conductive copper inks as well as films. Copper films with low resistivity (three times of the bulk copper) have been achieved after sintering in a reducing gas flow, where the oxides on the surface of copper NPs were removed (Fu et al., 2020).

Apart from the oxidation problem of copper NPs, film porosity is also a primary factor impacting the electrical resistivity. In this work, a multilayer-sintering method, where the coating and thermal sintering processes are repeated up to four times, will be presented. We studied the relationship between porosity and electrical resistivity of sintered copper films using scanning electron microscopy. Besides, the corresponding film porosities were evaluated by the image analysis software.

Our results indicated that the porosity of copper films could be effectively reduced from 33.6 % after one-layer sintering to .7 % after four-layer sintering, as displayed in figure 1. The porosity reduction of sintered copper films is in good agreement with the electrical resistivity decrease, revealing that this sintering method is efficient for addressing the porosity issue.

DEM simulation of a tablet-coating process and optimization of PAT placement

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(1) RCPE GmbH

Keywords | process analytical technology, tablet coating, coating thickness, coating homogeneity, discrete element method, optical coherence tomography

Coating is an important unit operation in the manufacturing of tablets. The uniformity of the coating across tablets and over the surface of an individual tablet is crucial for the tablet's appearance and, in the case of functional or active coatings, for the drug release. The coating quality can be monitored in-line in a coating process using process analytical technology (PAT) tools such as optical coherence tomography (OCT). OCT allows measuring the coating thickness of tablets by producing images of the coated tablets in the coater, which are then automatically evaluated. Finding the optimal position and settings for a given PAT tool can be a tedious trial-and-error process. In addition, it is not a priori known if the sampling is free of bias.

During a coating run of biconvex tablets in a lab-scale drum coater, the coating thickness was measured in-line using OCT. The same coating process was modeled using a discrete element method (DEM) simulation. Individual spray droplets were applied to the tablets using a cube mapping algorithm. The sampling process of the OCT was then mimicked in the DEM post-processing using a ray-tracing approach to obtain individual measurements of the local tablet coating thickness over time. The distribution of coating thicknesses in the DEM simulation closely matched the experimental data. The simulation results show the importance of the image analysis approach when processing the OCT data and the limitations of in-line measurements in a drum coater.

Furthermore, metrics for the quality of OCT measurements, such as the velocity distribution of tablets passing the OCT probe and the fraction of valid measurements, were studied for a range of OCT positions in the simulation. The results show a detailed study of the expected quality of in-line measurement at individual PAT tool placement positions. While the presented case is for an OCT probe in a drum tablet coater, this approach is applicable to various industrial processes and PAT tools.

4 PARTICLE-FLUID SYSTEMS: FLUIDIZATION AND MULTI-PHASE FLOW

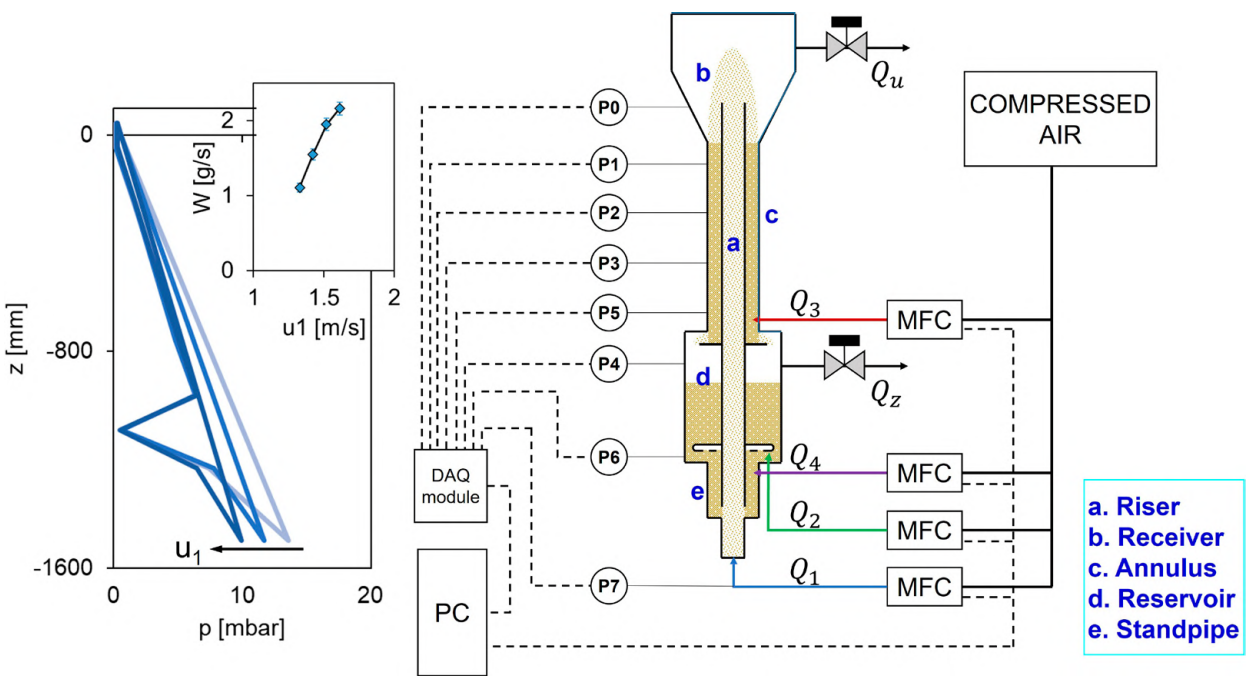
KEYNOTE LECTURES

Directly irradiated fluidized bed autothermal solar reactors

Padula, Stefano (1); Troiano, Maurizio (1); Tregambi, Claudio (2); Solimene, Roberto (3); Salatino, Piero (1)

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Keywords | Fluidized Beds, Circulating Fluidized Beds, Thermochemical Energy Storage, Concentrated Solar Thermal, Autothermal Reactor



Concentrated Solar Thermal (CST) energy coupled with Thermal Energy Storage (TES) systems is an appealing alternative to fossil sources for power production. In CST systems, sun-tracking mirrors concentrate solar beams onto a receiver, where it is absorbed by an energy carrier. The energy carrier is usually stored and used to drive a power cycle. Granular solids offer several advantages as energy carriers and open the path to Thermochemical Energy Storage (TCES), which consists in exploiting solar energy to sustain an endothermic chemical reaction. The energy is stored in the reaction products, allowing larger densities and longer times of storage.

A novel Directly Irradiated Fluidized Bed Autothermal Reactor (DIFBAR) is investigated (Tregambi et al. 2020). The key feature of the reactor is the autothermal operation: the sensible heat of the solid product is recovered to preheat the reactants. This is obtained by means of a solid-solid heat exchanger composed of two vertical coaxial tubes connected at the bottom of a conical receiver. The bed material is fed to the receiver through the inner tube (riser) by a fluidizing gas stream. There, particles are exposed to a high flux of solar radiation and undergo a chemical reaction. Then they fall into the outer tube (annulus) separating from the gas stream, descend through the annulus as a moving bed and transfer their sensible heat to the riser. A lab-scale prototype was designed and built up, in which the material is taken from and re-injected in a same reservoir, realizing a closed loop circulating system.

In this study the hydrodynamics of the DIFBAR is investigated with a Geldart B sand under various operating conditions. Solid circulation

rates are determined through an optical access and time sampling tests. Pressure profiles are acquired to control the sand level in the annulus and to deduce the effect of operative variables on solids circulation and gas by-passing. Gas tracing tests indicate the possibility of zeroing gas by-passing flowrates.

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Resolving Generation of Reduction Gas in the Raceway of a Blast Furnace Through a Coupled Euler-Lagrange Approach

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(1) Luxembourg

Keywords | xtended Discrete Element Method (XDEM), blast furnace, raceway, Euler-Lagrange coupling

Blast furnaces are among the largest and most complex reactors operated to produce pig iron. In order to reduce the iron ores, a reducing gas is generated by introducing a high-speed blast of hot air through the tuyeres at the lower section of the furnace [1]. The hot blast enters the packed bed of coke particles in the bottom part of the furnace with a velocity of app.

200 m/s and a temperature of app. 1500 K and forms the so-called raceway. Under these conditions, coke is oxidised to form carbon monoxide as the only stable product under the prevailing conditions. Experimental data is more than scarce due to the inaccessibility of the raceway and the hostile environment. Therefore, this contribution presents a multi-physics simulation environment that describes the multi-phase and multi-scale reacting three-dimensional and time-dependent flow of hot blast and coke particles with a high degree of resolution for the first time to the best of the author's knowledge. For this purpose, the Extended Discrete Element Method (XDEM) [2] is applied that is based on an Euler-Lagrange approach taking heat, mass and momentum transfer between the fluid and the particles into account. The results show the highly fluctuating formation of a raceway as a cavity inside the packed bed of coke particles including chemical reactions. It distinguished itself significantly from two-dimensional simulations in so far that still, a number of particles reside inside the cavity which is not observed in a two-dimensional approach [1].

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Keynote: Magnetic Resonance Imaging (MRI) of particulate systems

Müller, Christoph (1)
(1) ETH Zürich

Granular systems are ubiquitous in nature and industry. Yet, despite their frequent natural occurrence and industrial utilization, the physics controlling their behavior are still only poorly understood. One aspect that has complicated our endeavor to improve our understanding of such systems has been the difficulty in acquiring experimental measurements of typically visually opaque particulate systems. Besides X-ray tomography, magnetic resonance imaging (MRI) is a very powerful technique that has the potential to provide non-intrusive measurements in particulate systems, such as particle position, solid fraction and particle velocity (Müller et al., 2006). However, MRI of particulate systems is inherently challenging due to the unfavorable MRI characteristics of solids, making the acquisition of MRI measurements with high temporal resolution particularly challenging (Müller et al., 2010).

This presentation will describe first advantages and limitations of the different measurement techniques that have been used to probe the dynamics in particulate systems. This is followed by a description of the fundamentals of magnetic resonance imaging and recent advances in MRI technology to accelerate spatio-temporal resolution (Penn et al., 2017). This acceleration has been achieved by combining advances

in imaging hardware, particulate material engineering and MRI sequence protocols. The talk is concluded by providing examples of how the application of this new MRI technique has enabled to visualize highly-dynamic particulate systems such as fluidized beds (Penn et al., 2020).

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Müller et al., Physical Review E, 82, 050302, 2010 Penn et al., Science Advances, 3, e1701879, 2017 Penn et al., Physical Review Fluids, 5, 094303, 2020

Flow regime chart for pneumatic conveying and plugs analysis

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(1) Ben-Gurion University of the Negev

Keywords | Pneumatic conveying, Flow mode, Pressure drop, Plug flow, Dense phase flow

Dense-phase pneumatic conveying is preferred over dilute-phase flow in various industries because of its advantages. Therefore, numerous studies have been conducted on developing various models, thereby enabling the calculation of pressure drop. However, it is necessary for a designer to be able to predict the mode of conveying in advance. Therefore, in this study, we focus on developing a new flow regime chart. Based on the previous studies and new experiments presented in this study, all the required threshold velocities are defined by the Reynolds number as a simple power function of the Archimedes number. In addition, a careful analysis of hundreds of videos showing the flow of various materials in various operating conditions, revealed two new types of plugs. The comparison of the new flow regime chart with the previous experiments conducted for analyzing Plug-2 suggests that Plug-2 can be easily confused with Plug-2* (in which the layer between the consequent plugs is not stationary). To the best of our knowledge, the new flow regime chart is the first to include both particle properties and operating conditions. Comparing the results of this study with those of the previously published models, which characterized only material properties, revealed that the feeder plays a major role in controlling the final mode of flow. The flow regime chart combined with the analysis and models for pressure drop calculations for each plug type, enable to estimate the pressure drop in dense phase flow.

Fluidized Bed Reactor Application for Catalytic Pyrolysis of Waste Plastic

Singh, Raj (1); Badiola, Carlo (2); Wang, Song (3); Maller, Alexander (1); Marchant, Paul (1)
(1) Technip Energies, (2) Encina, (3) Encina

Keywords | Fluidized bed reactor, Multiphase flow and fluidization, Catalytic pyrolysis, Computational modeling, advanced chemical recycling

Fluidized Bed Reactor Application for Catalytic Pyrolysis of Waste Plastic
By
Carlo Badiola and Song Wang
Encina Development Group, LLC (Encina)
Raj Singh, Alexander Maller, and Paul Marchant Technip Energies (T.EN)
Abstract
for the paper to be presented at
World Congress on Particle Technology
WCPT9, Madrid, Spain, September 18 - 22, 2022

Abstract:

Fluidized bed reactors are widely used for processes that require intimate mixing of catalyst particles with reactant vapors Intimate mixing is key to achieving high mass and heat transfer rates. Encina has developed a novel fluidized bed catalytic pyrolysis reaction system to convert post-consumer scrap plastic to valuable products, such as light olefins and BTX aromatics, as a technology within advanced chemical recycling. The process is similar to conventional Fluidized Catalytic Cracking (FCC), where catalyst circulates between a Reactor and Regenerator via lift lines and standpipes. The heat of combustion released during regeneration supplies the energy for chemical conversion. This paper discusses the impact of the required fluidization regime on the overall process design and provides key highlights of the technology that differentiates it from other waste plastic recycling processes.

Also discussed is the commercialization approach taken by Encina to bring the technology to market. The approach taken is to parallelize

cold flow hydrodynamic testing, hot flow demonstration testing , and design development and scale up of the fluidized bed systems by Technip Energies. Understanding the physical properties and flow characteristics of particles along with the reaction kinetics is key when designing such units. The fluidized bed scale-up and the use of computational modeling tools to study the reactor bed hydrodynamics and optimize the design to enable fast prototyping will be discussed as well.

Keywords: Fluidized bed reactor, Multiphase flow and fluidization, Catalytic pyrolysis, Computational modeling, advanced chemical recycling,

ORAL COMMUNICATIONS

Mini-Torbed Technology for Carbon Capture Adsorbent Screening

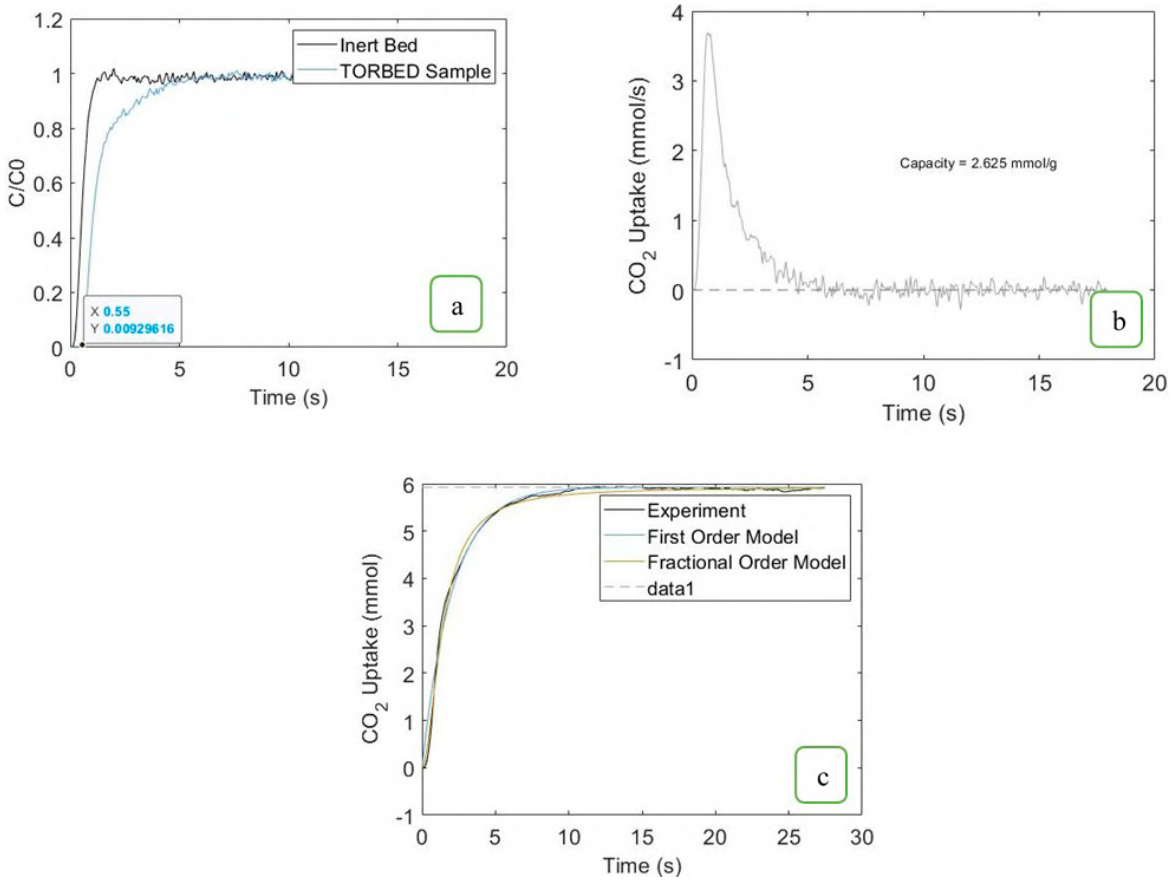
Jamei, Mirrouzbeh (1); Zikovich, Vladimir (1); Mcdonough, Jonathan (1); Reay, David (2)
(1) Newcastle University, (2) Newcastle University

Keywords | Mini-Torbed, Swirling motion, Maximum temperature, Bed loading, Carbon capture

The importance of carbon capture is made clear in the global target to rapidly transition to Net Zero emissions by 2050. Besides using renewable energy sources, carbon capture is an essential short-term strategy. Solid adsorption is a promising alternative to more established carbon capture methods, but its success depends on both the development of highly specific materials and improved gas-solid contact efficiency. Whilst fluidized beds achieve good heat/mass transfer and good mixing, they struggle with fines and 'oversized' particles that tend to form undesirable regimes.

Swirling fluidization provides an alternative approach for further intensifying gas-solid contacting that also overcomes the maldistribution issues. Swirling beds use inclined blades to create additional swirling motion of the particle bed that increases radial/tangential mixing whilst minimising axial mixing. This enables much higher gas velocities to be used without losing material from the bed, thus enabling the processing of a wider range of particle sizes whilst maintaining good mixing.

In the present paper, we use the mini-TORBED (a commercialised swirling bed) to capture CO2 from an artificial flue gas stream comprised of nitrogen and CO2 using a commercial adsorbent (mixture of polyethyleneimine, branched ethylenediamine and silica gel). Here we performed CO2 breakthrough experiments (Figure 1a) at different gas flow rates, CO2 volume fractions, adsorbent loadings, and gas temperatures. From these experiments, we observed a high sorbent capacity of 2.66±0.16 mmol/g (Figure 1b), which was slightly beyond the expectations of the sorbent manufacturer and higher than related materials in the literature. By monitoring the maximum bed temperature during each experiment, we observed no spikes corresponding to the heat of adsorption, implying that enhanced heat transfer potentially enables high sorbent capacities. We also found that either the pseudo-1st order or fractional order models are sufficient for describing the kinetics (Figure 1c). Our mini-TORBED platform enables the fluidization of Geldart C/D group powders as readily as Geldart A/B, meaning we can rapidly screen a wide range of potential adsorbents.



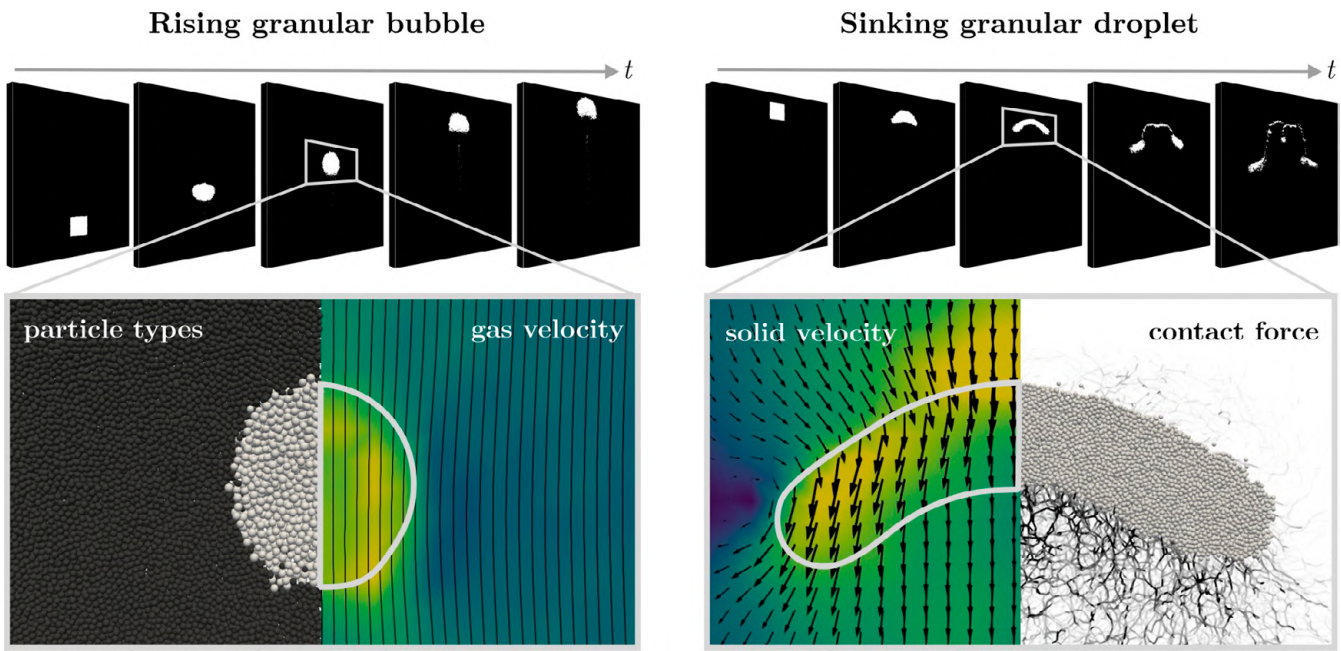
Rising granular bubbles and splitting granular droplets: Revealing the physics behind fluid-like phenomena in binary granular materials

Metzger, Jens P. (1); McLaren, Christopher P. (1); Pinzello, Sebastian (1); Conzelmann, Nicholas A. (1); Boyce, Christopher M. (2); Müller, Christoph R. (1)
(1) ETH Zurich, (2) Columbia University New York

Keywords | Vibro-gas fluidization, CFD-DEM, Granular fluids, Granular droplet, Granular bubble

Binary granular materials exhibit fluid-like characteristics when subjected to a combined vibration and gas fluidization. A cluster of particles immersed in a bulk of smaller and denser particles forms a coherently rising structure resembling a gas bubble rising in a liquid (McLaren et al., 2019). Conversely, if the surrounding bulk particles have a smaller density and larger size, the cluster sinks and performs consecutive splits reminiscent of a liquid drop falling in a miscible fluid. Due to the absence of surface tension between dry grains and the ability of granular matter to solidify readily, the physics governing the formation of granular bubbles and droplets in binary granular materials must be distinctly different to classic fluid dynamic principles.

This research elucidates the physics controlling the occurrence of such fluid-like phenomena in granular media. To this end, experiments in pseudo-2D vibro-gas-fluidized beds are supplemented with numerical simulations, which combine computational fluid dynamics and the discrete element method (CFD-DEM) to provide insight into the prevailing gas flow patterns and the emerging inter-particle force network (Metzger et al., 2022). Based on these results, an analytical model is derived to compile a dimensionless regime map predicting the emergence of bubble and droplet structures depending on size and density differences between the granular materials involved.



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Unstable sphere sinking in a fluidized bed at higher air velocity; 2) numerical study

Tsuji, Takuya (1); Oshitani, Jun (2); Harada, Shusaku (3); Washino, Kimiaki (1); Tanaka, Toshitsugu (1); Kajiwara, Hirokazu (4); Matsuoka, Kei (5)
(1) Osaka University, (2) Okayama University of Science, (3) Hokkaido University, (4) Ebara Environmental Plant Co., LTD., (5) Ebara Corporation

Keywords | Fluidized bed, DEM-CFD, Sinking-Floating

The floating and sinking of a large solid object in gas-fluidized beds are important in the applications such as combustion, gasification, and incineration. By injecting a gas flow from the bottom, a pressure gradient is formed in the bed. After the gas velocity (u0) reaches the minimum fluidization velocity (umf), the averaged pressure gradient does not change because the fluid force working on particles is balanced with the gravity force in the averaged sense. The floating and sinking of large objects in the bed have been explained by the balance between the averaged buoyant force due to the gas pressure gradient and the gravity force; namely, an object floats when the averaged buoyant force overcomes the gravity and vice versa. We experimentally found that large sphere floating was enhanced when the gas velocity is considerably higher than the minimum fluidization velocity (u0/umf > 4). In the present study, we performed corresponding discrete particle numerical simulations (Fig. 1) to understand the mechanism of the floating enhancement at the higher gas velocities. The numerical results revealed the occasional formation of denser regions accompanying a larger gas pressure gradient than the average. If a sphere locates in the dense region by chance (Fig. 2), a large buoyant force works, and the sphere floats intensively at the moment. These results show that the phenomenon is not explainable by the averaged picture and the importance of a local and instantaneous flow field.

Investigation of the wet contact behavior of particles in CFD- DEM simulations of a rotor granulator

Grohn, Philipp (1); Oesau, Tobias (2); Heinrich, Stefan (2); Antonyuk, Sergiy (1)
(1) Institute of Particle Process Engineering, Technische Universität Kaiserslautern, (2) Institute of Solids Process Engineering and Particle Technology, Hamburg University of Technology

Keywords | CFD-DEM simulation; capillary force; viscous force; coating model; wet particle; velocity-dependent maximum liquid bridge length; fluidized bed rotor granulator

In the pharmaceutical, chemical and food industries, coating of solid particles in fluidized beds with various substances is an important production step. In order to optimize these processes, it is essential to know and describe the particle kinematics and dynamics. An essential tool to obtain these data are numerical simulations of the multiphase flow with the computational fluid dynamics (CFD) coupled with the discrete element method (DEM). With CFD-DEM, the flow field of the gas in the process apparatus can be determined by the Eulerian-Lagrangian approach, which treats the fluid phase as a continuum. In DEM, the interactions are calculated for each single particle based on models describing physical properties of particles and their mechanical behavior stressing (Grohn et al., 2020).

In this work, we first experimentally investigated the dynamic wet particle contact. The influence of impact velocity and liquid bridge volume on the length of the liquid bridge were determined. A model for a velocity-dependent maximum liquid bridge length was developed and used in an implemented liquid bridge contact model in DEM. The additional dissipative forces were modeled according to the liquid bridge model described in Grohn et al. (2020). Then, the process was calculated with CFD-DEM simulations in order to obtain important information about the fluidization behavior of the particles and the influence of the liquid on the contact behavior of the particles. Furthermore, the simulation results were validated with experimental data obtained by magnetic-particle-tracking measurements (Oesau et al. 2021) of the particle movement in a rotor granulator.

The obtained results were used to determine the boundary conditions under which a successful fluidization in the rotor granulator can be realized.

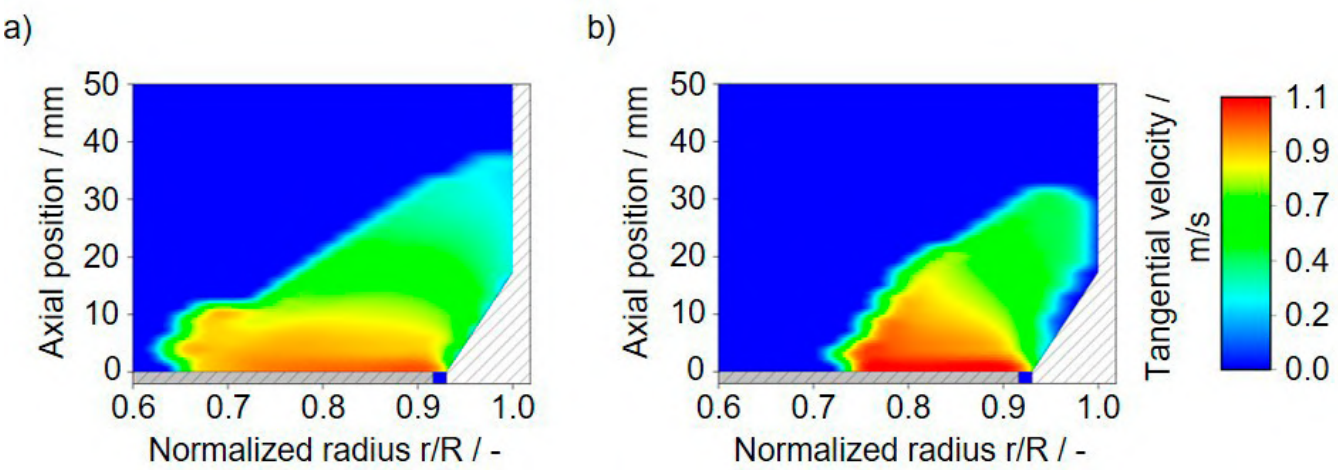


Figure: Simulated tangential particle velocity in the investigated rotor granulator for a) dry case and b) wet case with 5-vol% water.

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Oesau, T.; Grohn, P.; Pietsch-Braune S.; Antonyuk, S.; Heinrich, S., 2021. Novel approach for measurement of restitution coefficient by magnetic particle tracking, Advanced Powder Technology 113, 261.

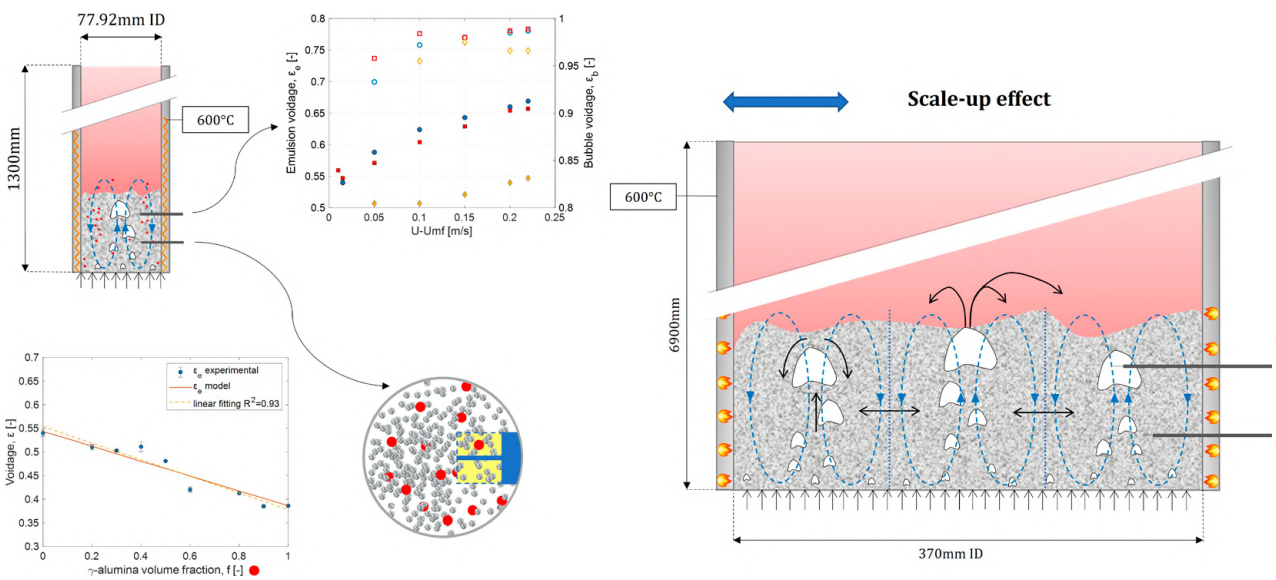
Gas phase distribution and solids mixing in high temperature dense fluidized beds

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Keywords | Gas-solid fluidization, High temperature, Bed porosity distribution, Binary mixture, Capacitance probe

Gas-solid fluidization represents an optimal solution for the exploitation of a wide spectrum of solid fuels by thermochemical processes. Successful employment of such energy resources strongly depends on the level of mixing/segregation of solid fuel and bed material in the dense reactor zone (Salatino and Solimene, 2017). Despite plenty of results in literature on the mentioned issue, very few works report data from pilot-to-large scale units at typical industrial conditions. The major challenge of such studies is to find an experimental technique suited to high temperatures and 3D-units. Profitable results from fluid-dynamic down-scaling do not account for the role of interparticle forces when high temperature conditions are adopted. The objective of the present work is to further investigate solid mixing and segregation phenomenology, and its impact on heat exchange in dense fluidized beds at industrially-relevant operating conditions. Custom capacitance probes for high temperature environments are purposely developed. First, single materials hydrodynamic characterization is carried out at 600°C in a medium-size bubbling laboratory scale column. Experimental results show the dependence of Geldart B fluidized beds voidage, measured by capacitance probes, on radial and axial bed coordinate and superficial gas velocity. Thus, simple two-phase theory cannot be adopted for accurate capacitance signal processing during mixing experiments. A comparison with tests at ambient conditions confirms a temperature dependent gas-solid distribution. High temperature calibration experiments with binary mixtures of known composition demonstrate that, for the employed solids, voidage in the emulsion phase presents a linear dependency on solid composition at incipient fluidization. The model is extended to dense phase evaluation at higher gas velocities in stimulus-response tracing experiments. Tracer feeding and mixing phenomena in the reactor are modeled in detail to assess solids dispersion. The medium-size laboratory scale unit allows for easier experiment handling and tracing technique validation. Then, results are compared with experiments in a pilot-scale unit and first outcomes about the dimensional scale effect on mixing of a tracer batch in the fluidizing medium are discussed.

Salatino, P., Solimene, R., 2017. Mixing and segregation in fluidized bed thermochemical conversion of biomass, Powder Technology, 316: 29–40.



Assisted fluidization of cohesive powders: insights from an X- ray tomography study

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Keywords | cohesive, assisted fluidization, gas channel, X-ray, agglomerate

Fine powders (from nanometers to a few microns), possessing a large specific surface area, excel as functional materials for chemical processes, but their complete fluidization is hardly attained due to the cohesive nature. Attraction forces, such as capillary force and van der Waals force, promote agglomeration. Subjected to a steady gas flow, agglomerates remain stagnant, and grow channels allowing for bypassing fluidizing gas. Such poor interphase contact greatly depresses particle mixing and both heat and mass transfer (van Ommen et al., 2012). Many attempts were conducted in the last decades to improve fluidization of cohesive powders via introducing assistance of various forces and energy sources, such as mechanical vibration, gas pulsation, stirring, micro-jets and so forth (Francia et al., 2021). Despite the enhanced fluidization witnessed, the underlying physics and hydrodynamic events caused are yet to be fully understood, which complicates

its scale-up and implementation in larger scale units.

In this work, we investigated experimentally assisted fluidization of inert micron-sized powders using mechanical vibration, gas pulsation and stirring. X-ray computational tomography, as well as pressure fluctuation measurement, were used to characterize the temporal evolution of bed hydrodynamics under different assistances, and detect events such as particle segregation, partial bubbling and channels rearrangement (see Figure 1). We also compare the resulting bed behaviour to unassisted units, quantify the degree of enhancement induced at different assistance intensities, and discuss the mechanism leading to improved performance. These results help to optimize existing units of solid processing and develop an efficient process for catalyst production.

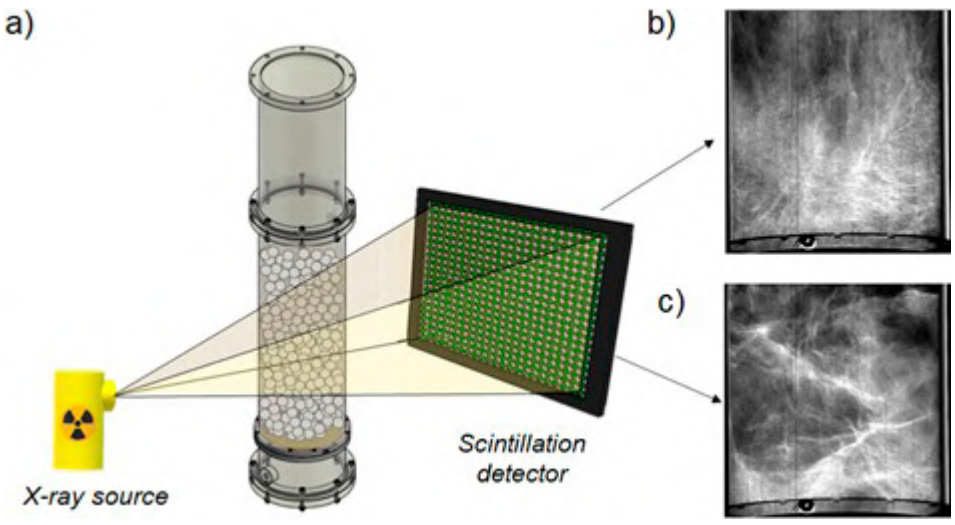


Figure 1 – (a) Schematic of X-ray tomography measurement setup, and examples of time-averaged profiles of gas channels created in the beds (b) under mechanical vibration and (c) without assistance. The superficial gas velocity is 0.3cm/s, powder size is 7.5μm and density is 1.9g/cm3.

Francia, V., Wu, K. and Coppens, M.-O. (2021) 'Dynamically structured fluidization: Oscillating the gas flow and other opportunities to intensify gas-solid fluidized bed operation', Chem. Eng. Process., 159, pp. 108143.

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Boundary of Geldart Groups A and C Based on the Magnitude of Interparticle Forces

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Keywords | Geldart classification, Fluidization, Interparticle forces

Geldart (1973) classification has long been adopted to predict fluidization characteristics of powders. It is based on the particle diameter and the difference between solid and gas densities. This classification does not explicitly consider the effect of interparticle forces (IPFs). Nevertheless, fluidization behaviors of Geldart groups A and C are significantly impacted

by appreciable relative magnitudes of IPFs acting on these particles to the weight of a particle ($/dpi100/smallW_p$) (Molerus, 1982, Shabanian and Chaouki 2017). In addition, Geldart behavior of a powder can shift upon a discernible change in the level of IPFs, for instance at elevated operating conditions (Lettieri et al. 2001) or in the presence of a small amount of liquid (McLaughlin and Rhodes 2001). A criterion based on the magnitude of IPFs is proposed in this work to formulate the boundary of Geldart groups A and C and predict the transition of the fluidization behavior from one to another.

Generalized forms of the dynamic Hausner ratio (DHR), i.e., the ratio of the minimum fluidization voidage to the loose bulk voidage ($/dpi100/small/varepsilon_{mf}//varepsilon_0$), and $/dpi100/smallU_{mf}$ (minimum fluidization velocity)

deviation equations (Soleimani et al. 2020) were used for the quantification of IPFs. It was postulated that channeling, as a distinct Geldart group C characteristic, occurs when the gas velocity at minimum fluidization conditions, i.e., $/dpi100/smallU_{mf}//varepsilon_{mf}$ / $/dpi100/smallU_{mf}$, exceeds the particle terminal velocity. Coupling the generalized DHR and deviation equations with this hypothesis led to two equivalent and novel equations for the boundary of Geldart groups A and C behaviors. These equations correlate $/dpi100/small/varepsilon_{mf}$ to $/dpi100/smallIPFs/W_p$ and $/dpi100/smalld_{agg,mf}/d_p$ (the ratio of agglomerate size at minimum fluidization conditions to the particle size). They suggest that IPFs corresponding to the boundary of Geldart groups A and C behaviors depend on $/dpi100/small/varepsilon_{mf}$ Experimental results showed that the transition from Geldart group A to C typically happens when $/dpi100/small/varepsilon_{mf}$ is about 0.5-0.7. This corresponds to $/dpi100/smallIPFs/W_p$ in the range of 7-19 and $/dpi100/smalld_{agg,mf}/d_p$ within 2.5 -4.5. Promising results were obtained for discriminating Geldart groups A and C behaviors of powders under various conditions in this work and with available data in the literature.

Bed densities in stationary and flowing gas-solids fluidized beds

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(1) Particulate Solid Research, Inc

Keywords | fluidized beds, strippers, bed density, FCC

Gas-solids fluidized beds have wide commercial applications in the physical and chemical processing industry, petroleum refining, coal combustion and renewable energy operations. Fluidized bed strippers, which are essentially flowing fluidized beds where an up-flowing gas removes (strips out) product entrained in a down-flowing stream of the emulsion phase (denser solid phase), have significantly higher flowrates of solids exiting the bed compared to the product removal rates in conventional fluidized beds, often referred to as stationary fluidized beds. Bed density is an important parameter in fluidized bed design and operation. Bed density enables estimations of bed heights or solids inventories in fluidized bed units, calculation of standpipe aeration rates, and determination of available pressure driving heads in various components of circulating fluidized bed systems. In fluidized bed strippers, bed density can indicate if there is flooding in the stripper. As is typical for two-phase countercurrent flow unit operations, flooding can be an issue with strippers. King (1989) correlation has traditionally been used for calculating the overall bed voidage in fluid catalytic cracking (FCC) catalyst fluidized beds. The correlation was developed from apparent bulk density of commercial equilibrium FCC catalysts and bed density measurements in hot commercial FCC regenerators and fluidized bed strippers. The expression is claimed to be applicable to fluidized bed superficial gas velocities of near zero up to at least 5 m/s. This paper compares King (1989) correlation predictions with bed density measurements conducted in cold flow stationary and flowing fluidized beds of FCC particles in our study. The fluidized bed units ranged in diameters from 0.76 to 1.52 m and the fluidized bed strippers were 0.6 and 0.9 m in diameter.

Heat transfer augmentation in horizontal dilute-phase pneumatic conveying

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Keywords | Pneumatic conveying, heat transfer, particle-gas flow

Pneumatic conveying is among the most common ways of transporting particulate materials and powders a relatively short distance within industrial plants. The convey of particles occurs between units in which mechanical, thermal, and chemical processes take place. The efficiency of the units can be increased by conducting a part of the process during the pneumatic conveying. For example, we can heat a pipe section to heat the particles. In this study, we focus on the pipe wall-suspension and on the interphase gas-particles heat transfer mechanisms, in horizontal dilute-phase pneumatic conveying.

The research is conducted experimentally using a unique comprehensive system that allows to establish, sustain, monitor, and measure all stages and features of the complex processes. This experimental system is divided into two main parts: (1) a flow system which allows to convey various particle types and determine different flow regimes, and (2) a heat transfer test section which allows to supply uniform heat flux and can be mounted at different locations along the pipeline. The experimental test rig is capable of measuring the parameters required for heat transfer analysis, such as the particle and gas average velocities and flow rates, the inlet and outlet gas and particles temperatures of the heated test section, and the temperature profile along the heated pipe wall.

Many experiments are conducted with various particle types and sizes, heat rates, and solid loading ratios. The data achieved is analyzed in a non-dimensional manner to correlate the heat transfer coefficient in terms of the wall-suspension and gas-particle Nusselt numbers. This analysis refers to the fully developed region (hydrodynamically and thermally), which will serve as a reference case for further experimental research of the particle acceleration zones.

Stratification in a mildly fluidized bed due to thermal inhomogeneities

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(1) Purdue University

Keywords | Fluidization, Experimental, DEM, thermal gradients

Inhomogeneous temperature profiles can occur in certain fluidized bed systems, such as heat exchanges where a steady supply of hot particles enters the bed while cold particles are removed from the bed. Under relatively weak fluidization conditions, the inhomogeneities in the temperature can lead to inhomogeneities in the fluidization behavior. We investigate such behavior using the discrete element method and experiments performed in a laboratory-scale fluidized bed. A fluidized bed, with a diameter of approximately 3.125", is built, and electric heaters are wrapped around the vessel and are used to heat the contents of the bed to 1200°C. The bed consists of silica sand particles with a Sauter mean diameter of approximately 625 microns. The numerical method used is Discrete Element Modeling (DEM), and the bed of particles is initialized with a non-uniform temperature profile to establish a thermal gradient. To verify the DEM simulations, defluidization curves are directly compared between the experimental and numerical data. The minimum fluidization velocities and curves matched well between the two methods. For low relative velocities (U/Umf), the particles do not mix and the temperature remains stratified. This stratification is observed both in the experimental setup and in the DEM simulations. The numerical method is used to create a regime map that establishes the relationship between the relative velocity and the bed temperature profile. For relative velocities between 1 and 1.5, the bed is fluidized with an inhomogeneous bed temperature. For relative velocities higher than 1.5, the particles mix, and the bed temperature becomes uniform. The regime map is created by the DEM method and verified by the experimental data.

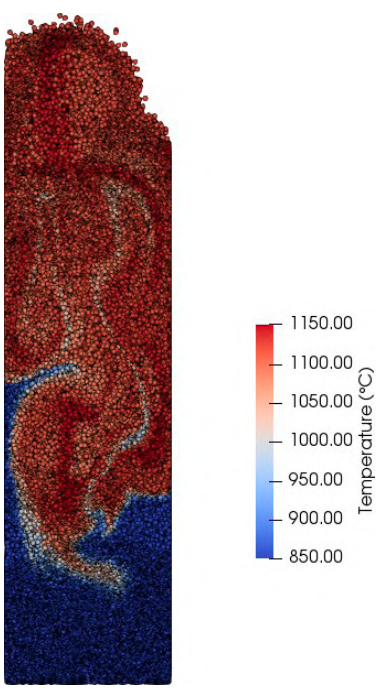


Figure 1. DEM particle temperatures in a mildly fluidized bed.

Packed-bed based thermochemical energy storage system

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(1) University of Birmingham

Keywords | thermochemical energy storage; system performance

This work is concerned with a packed bed based thermochemical energy storage (TCES) system. It is focused on the use of a water-sorbent pair. Despite significant efforts in the past, the sorption/desorption kinetics is less well understood, particularly at the device and system levels, leading to a relevant low system efficiency. Here we report our recent experimental work aimed at understanding the system performance through the examination of the effects of various operating parameters. An open thermochemical reacting system is used with silica gel as the sorbent. The effects of working fluid (air or air-water vapor) flow rates and temperature are measured as a function of time in both charging (desorption / energy storage) and discharging (adsorption / energy release) processes. These data are used to calculate the efficiency, temperature lifts, as well as charging / discharging kinetics. A lower flowrate is observed to give a longer discharging time and a higher temperature lift. The system efficiency, defined as the charging process efficiency discharging process efficiency) is found to increase with increasing charging air flow rate. The overall system efficiency is also called COP (Coefficient of Performance) in some literature. We find that the efficiency of the charging process can be as high as 90+%, whereas the discharging efficiency is around 50-60%, leading to the overall system efficiency or COP of ~50%. This is high compared with the literature data for thermochemical energy storage systems.

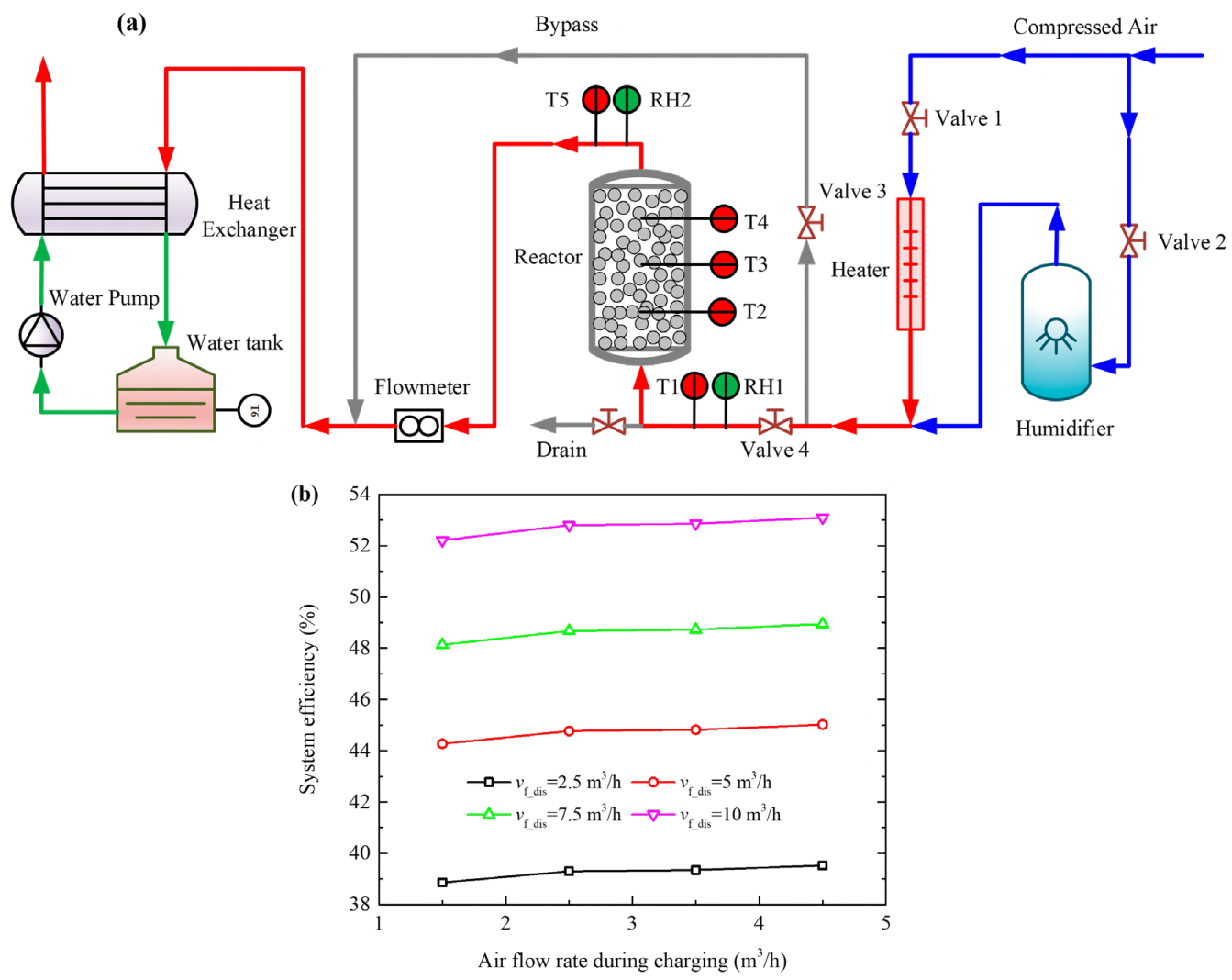


Fig. 1. (a) The schematic diagram of the TCES system and (b) system efficiency for various air flow rates. (*v*_dis represents discharging air flow rate)

Drag Force Acting on Irregularly Shaped Particles

Maramizonouz, Sadaf (1); Nadimi, Sadegh (1)
(1) Newcastle University

Keywords | Drag Force, Drag Coefficient, Drag Modelling, Irregular Particle Shapes, Fluid-Particle Interactions, Computational Fluid Dynamics, Discrete Element Method.

To provide sufficient traction for train acceleration and breaking conditions, sand particles are deposited on the rail in a process called rail-sanding. The velocity of these irregularly shaped particles significantly affects the rail-sanding efficiency. The widely-adopted rail-sanding practice has only ~20% efficiency in transferring and deposition of particles. The low efficiency of particle deposition poses safety issues for the train and is unsustainable due to limited sand resources. To reduce sand wastage and to enhance the process efficiency, the aerodynamic properties of the particles need to be better studied. The drag force and how to predict it to account for the irregular shape of the particles are crucial in estimating their behaviour.

Dioguardi et.al. [1] presented a one-equation model valid over a wide range of Reynolds numbers. In this method, the particle shape is considered using the ratio between particle's true sphericity and its circularity, limited to 2D particle shape classification. Through experiment and numerical modelling, Tagliavini et.al. [2] demonstrated that the harmonic mean of the drag coefficients calculated using the projected areas of minimum and maximum principal moments of inertia can estimate the drag coefficient of irregularly shaped particles with reasonable accuracy.

In this research, the drag model presented in [1] is compared with the one presented in [2] to define a suitable drag model for irregularly shaped particles used in rail-sanding. Using computational simulation, the drag coefficients are calculated from each approach, ther are then plotted in a unified framework using Zingg plot and are analysed in parallel with the data presented by Bagheri and Bonadonna [3]. The results stemmed from this investigation can help in tuning both CFD and DEM by discovering and utilising the most accurate drag model for rail-sanding applications. This will be used in the optimisation of the particle transfer and deposition process and finding the best combination of particle shape, size, and velocity to maximise the rail-sanding efficiency.

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Dioguardi, F., et.al. (2018). Journal of Geophysical Research: Solid Earth 123(1): 144-156. Tagliavini, G., et.al. (2021). International Journal of Multiphase Flow 140: 103652.

Chemical Looping Oxidative Dehydrogenation for Intensified Light Olefin Production

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Keywords | chemical looping, olefin, oxidative dehydrogenation, ethane, ethylene

Light olefins such as ethylene are predominantly produced via high-temperature steam cracking. The highly endothermic cracking process requires high operating temperatures and a large steam generation load, making it highly energy intensive (16-23 GJ/tonne ethylene). The process also emits significant amount of NOx and CO2 and requires periodic shutdowns for coke burnout. Moreover, single-pass olefin yield is often limited by thermodynamic equilibrium.

We proposed a chemical-looping ODH (CL-ODH) scheme that addresses these challenges. In this scheme, a metal oxide based redox catalyst donates its lattice oxygen for the ODH reaction. The oxygen depleted redox catalyst is subsequently oxidized in air, regenerating the catalyst, and releasing the heat needed for the process. The redox catalyst particles, which act as carriers of both oxygen and heat, facilitate reactive air separation with minimal energy consumptions. It also avoids direct mixing of oxygen and hydrocarbons, rendering a safer and more efficient process.

This presentation will cover key aspects related to the CL-ODH process development for ethane conversion. These include redox catalyst design, mechanism studies, reactor design, operational results, and process simulations. The redox catalyst we developed showed excellent stability for over 1,000 redox cycles in a lab-scale fluidized bed. ASPEN Plus® simulations based on the experimental results indicate that over 80% reduction in overall energy demand and CO2 emission can be realized. As such, the CL-ODH process exhibits an excellent potential to produce ethylene in a more sustainable manner. Although the present talk focuses on CL-ODH based ethylene production, redox catalyst design strategies for generating other light olefins such as propylene and butadiene are also discussed.

Numerical simulation of evaporation behavior of slurry droplets in spray drying process

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(1) Osaka Metropolitan University

Keywords | Spray dry, Euler-Lagrange approach, slurry droplet, Estimation of powder diameter

Spray drying is a fundamental process in several industries. As many factors affect the particulate product property in complex manners, optimal operating conditions have been determined through trial and error. In a spray dryer, numerous sprayed droplets form a tear-shaped droplet aggregate (droplet group), in which each droplet evaporates at a different rate. Owing to the complicated evaporation behavior of a droplet group, a detailed mechanism of dried particle generation has yet to be sufficiently elucidated. The objective of this study is to investigate the effects of the operating conditions of spray drying processes on the evaporation rate of a droplet group. The spray drying process of skim milk slurry droplets was numerically analyzed using the Euler-Lagrange approach with a reaction engineering model. The drying behavior of the slurry droplets was evaluated using a time-lag of first order function. The evaluation by time-lag of first order function was not suitable for the drying process without temperature distribution. On the other hand, the time-lag of first order function can be used to successfully evaluate the drying process in which temperature distribution exists and a large number of droplets are dried, such as in a spray dryer. The time constant, τ, was assumed to be the boundary between the constant rate drying period and the decreasing rate drying period. The average drying time (τ) were determined by simulations under various conditions. It was found that the τ could be estimated using the thermal efficiency and Nusselt number based on the operating conditions. Furthermore, skim milk powder was obtained by spray drying experiments under different conditions. The medium powder diameter strongly depended on the calculated drying time. In summary, the present numerical analysis revealed that the diameter of the resultant skim powder can be predicted from the operating conditions.

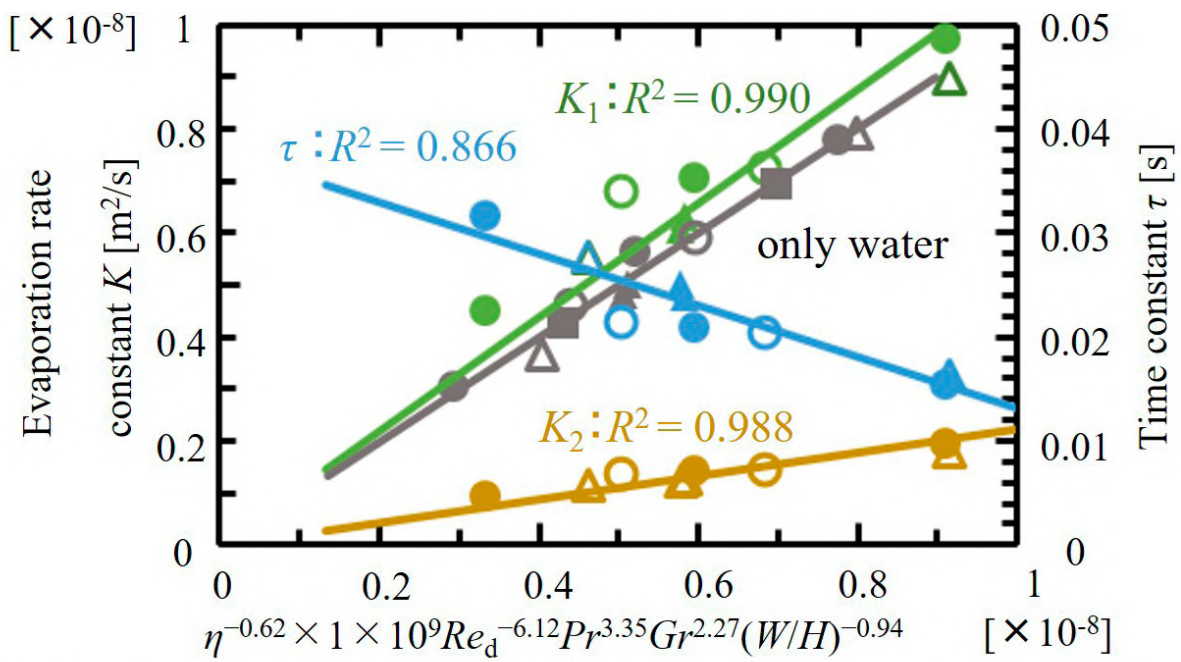


Fig. 1 Dimensionless correlation to estimate evaporation rate constant K and time constant τ

Pressurized Gas-Solid Feeder for Biomass Injection into Gas- Solid Fixed and Fluidized Beds

Tebianian, Sina (1); Massaro Sousa, Lucas (2); Amblard, Benjamin (1)

(1) IFP Energies nouvelles, (2) IFP Energies nouvelles, Wolfson Centre for Bulk Solids Handling Technology

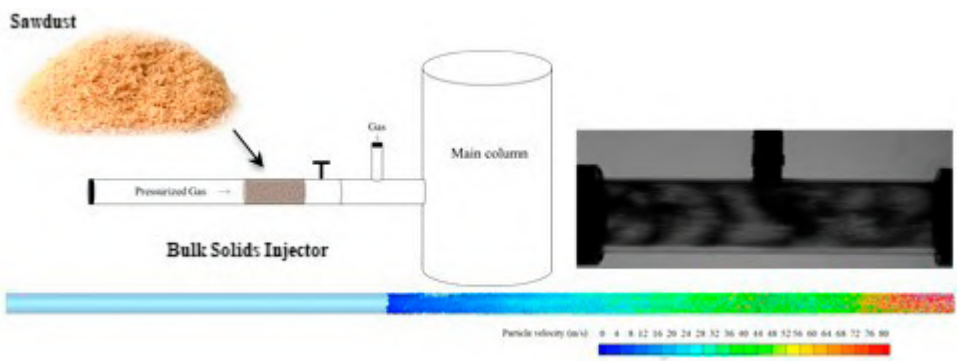
Keywords | pneumatic transport, Solid injection, fluidized beds, CFD, bulk solids handling

Efficient feed injection into reactors for thermochemical conversion of waste biomass is crucial for assuring the process performance. Pneumatic injection of a given amount of solid into a multiphase system is of interest for lab-scale batch unit reactors for kinetic studies and cold-flow models for characterization of mixing hydrodynamics between the injected solid tracer and the reactor medium.

Common physical properties of biomass powders, such as low particle density, irregular shape, wide particle size distribution, and moisture content make the feeding of these powders into reactors a recurring challenge (Dai et al. 2012).

In this work, a pressurized gas-solid feeder is utilized to inject a batch of sawdust powders into a fixed and fluidized bed cold- flow unit. The hydrodynamic features associated with biomass transportation by pressurized gas are characterized at different operating conditions through experimentation with a high-speed camera and CFD modelling. The operation map for this pulsed feeder was built by combining physical parameters that result in a certain transport flow regime in the injection line.

The penetration and dispersion of biomass powders injected in the unit are characterized by different measurement techniques such as fluorescent particle tracking, pitot tubes and Electrical Capacitance Tomography. The mentioned techniques provided complementary information on solid penetration into gas-solid fixed and fluidized beds. In general, a fair agreement was obtained among the jet penetration length measured with different techniques. A quantitative comparison of the results highlighted the advantages and disadvantages associated with each technique.



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Experimental and Numerical Study of Frictional Flows in a Vertical Millifluidic Tube

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(1) School of mines of Saint-Etienne, (2) École des mines de Saint-Étienne 158, cours Fauriel, (3) INSA Lyon, (4) Ecole des Mines SPIN Center

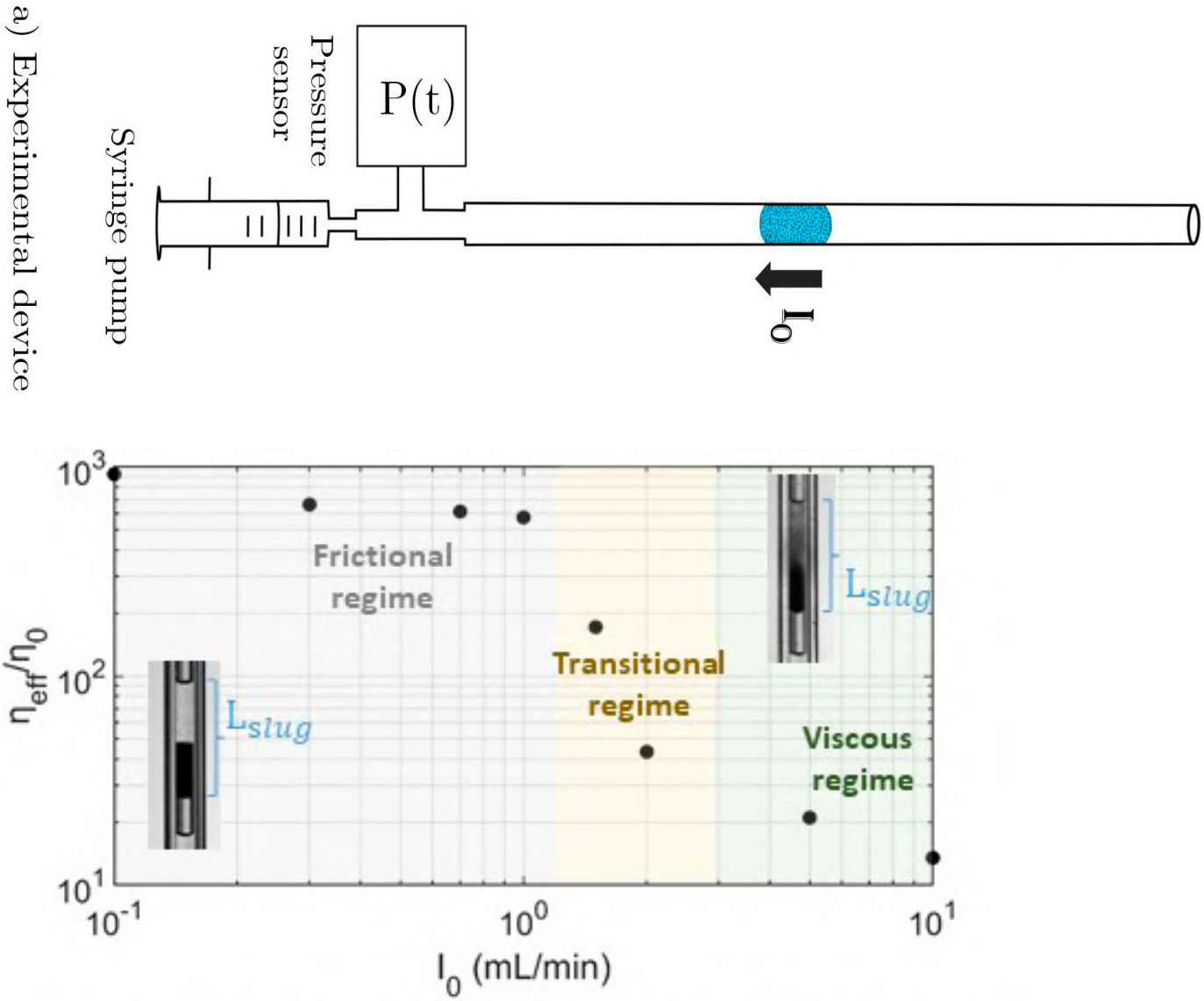
Keywords | Frictional Fluids Three-phase flow Frictional regime Viscous regime Discrete Element Method

We studied experimentally and numerically the displacement regimes in a confined geometry of a saturated granular phase (fig. a).

In our work we explored in detail in a vertical millifluidic tube a three-phase flow consisting of two immiscible fluids (water and air) and a granular phase (glass beads). The physical ingredients of such a system are the viscosity of the fluids, the solid friction between the grains, or between the grains and the confining walls, and the capillarity at the fluid-fluid interfaces.

In the case of supersaturated grains in downward motion two displacement regimes were found, a frictional regime for small flow rates ($I0 \leq 2$) and a viscous displacement regime for higher flow rates (fig. b). In upward movement the supersaturated grains have a stick-slip regime.

A numerical study is complementing these experimental observations with the aim of modeling by a discrete element method (DEM) the displacement of grains weakly saturated with water (Hygroscopic regime; Pendular regime), and modeling the effect of a capillary interface by a piston.



b) Effective viscosity η_{eff} normalized by the water viscosity η_0 as a function of the flow rates imposed by the syringe pump for a fixed grain fraction

Fluidization of a Geldart C Powder in a Micro-Fluidized Bed

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(1) Newcastle University

Keywords | Micro Fluidised Bed Reactor (MFBR), Fluidization, Geldart C, Pressure overshoot.

Recently, Micro Fluidised Bed Reactor (MFBR) technology has been proposed as a suitable platform for the evaluation of gas/solid reaction kinetics, owing to its low-cost, high-throughput testing capacity, low energy consumption, good heat/mass transfer properties, and improved safety. An emerging application area for the MFBR is the rapid low-cost screening of novel CO2 adsorbents, where the role of the MFBR is to ensure the collection of data under 'real-World' conditions for the purposes of decision making.

Hydrotalcites are regarded as an appropriate candidate for CO2 capture from effluent gas streams, but the novel high- capacity variants are often only synthesised in small quantities, which provides limited opportunity for testing. Accordingly, the small-scale MFBR are a suitable platform for evaluating these materials due to its characteristics. However, the success of MFBR technology ultimately depends on finding optimal conditions for the sorbent screening experiments. An additional challenge for hydrotalcites is their Geldart C classification. Their stronger inter-particle forces relative to other Geldart-type particles leads to significant particle agglomeration and very poor fluidisation quality.

Therefore, in this study detailed hydrodynamic experiments using the pressure drop characterisation approach have been performed in order to identify suitable operating conditions and to understand the role of wall effects. Here, 3D printing was used to manufacture the MFBRs; bed diameters of D = 10–15mm and static bed heights of H/D = 1–3 were considered. To control and fluidise the hydrotalcite, the powder was first sieved to a size of 53 µm (density of 2 g/cm3) to remove the more cohesive fines, then blended with inert silica powder demonstrating Geldart A characteristics (size and density of 93 ± 10 µm and 2650 g/cm3 respectively). Further, we also tested the use of 'pre-fluidisation' to improve the fluidisation quality by minimising overshoot behaviour.

Our results show that a simple combination of pre-sieving the particles to remove the fines and pre-fluidization drastically improved the fluidization quality. Moreover adding a secondary Geldart A particle to the hydrotalcite powder to create a mixture somewhat reduced the microfluidization characteristics whilst promoting better fluidization behaviour in general.

Optimize Gas Solid Distribution using Computational Modeling

Singh, Raj (1); Marchant, Paul (1); Shimoda, Steve (1)
(1) Technip Energies

Keywords | Fluid Catalytic Cracking (FCC), FCC riser, Riser termination device, Gas Solid distributor

Optimize Gas Solid Distribution using Computational Modeling
By
Raj Singh, Paul Marchant and Steve Shimoda Technip Energies (T.EN)
Abstract
for the paper to be presented at
World Congress on Particle Technology
WCPT9, Madrid, Spain, September 18 - 22, 2022

Abstract

In fluidized bed reactors, uniform distribution of gas and solids is critical to achieve the desired hydrodynamic and kinetic performance. For FCC units, Technip Energies uses a proprietary distributor design to terminate both reactor risers and spent catalyst lift lines to distribute the exiting gas-solid stream into a fluidized bed. This paper discusses Technip's gas- solid distribution technology and its recent developments for achieving improved performance.

In the Technip Resid FCC unit, air and spent catalyst is distributed into the 2nd stage regenerator catalyst bed. Proper distribution is key to achieve a uniform coke burn and an even temperature profile throughout the bed, resulting in high catalyst activity retention. In PropyleneMaxTM Catalytic Cracking (PMccTM) the riser reactor also terminates in a fluidized bed, requiring proper distribution of the gas/solid mixture. Proper distribution of the hydrocarbon vapor into the reactor bed is key to providing adequate contact for further cracking to valuable products. This paper discusses recent work by Technip Energies to develop an improved distributor for use as a riser termination device in both the PMcc reactor and the RFCC regenerator. Use of CFD modeling to screen and evaluate the concepts and optimize the final selected concept will be presented. Finally, the performance matrix used to quantify the benefits of the optimized design with respect to the existing will be highlighted.

Keywords: Fluid Catalytic Cracking (FCC), FCC riser, Riser termination device, Gas Solid distributor

Interphase transfer terms in the Reynolds Averaged Two Fluid Model for the simulation of high volume fraction suspensions of particles in turbulent liquids

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Keywords | interphase transfer, mixing, solid-liquid, Two-fluid Model, dense suspension

Modelling of turbulent solid-liquid mixing is still a challenging task due to the uncertainties in the closure relationship for fluid-particle and particle-particle interactions, which experience significant variations depending on the local solid volume fraction. Indeed, the boundary value of the particle volume fraction between dilute and dense suspensions is equal to 10-3 and typical industrial suspension conditions in stirred tanks are far from homogenous solid distribution. Thus, in some locations the particle distribution may depend on the turbulent liquid flow unaffected by the particles (i.e. one-way coupling), in others the particles may modify the liquid flow field (i.e. two-way coupling) and in other locations the particle interactions may prevail (i.e. four-way coupling).

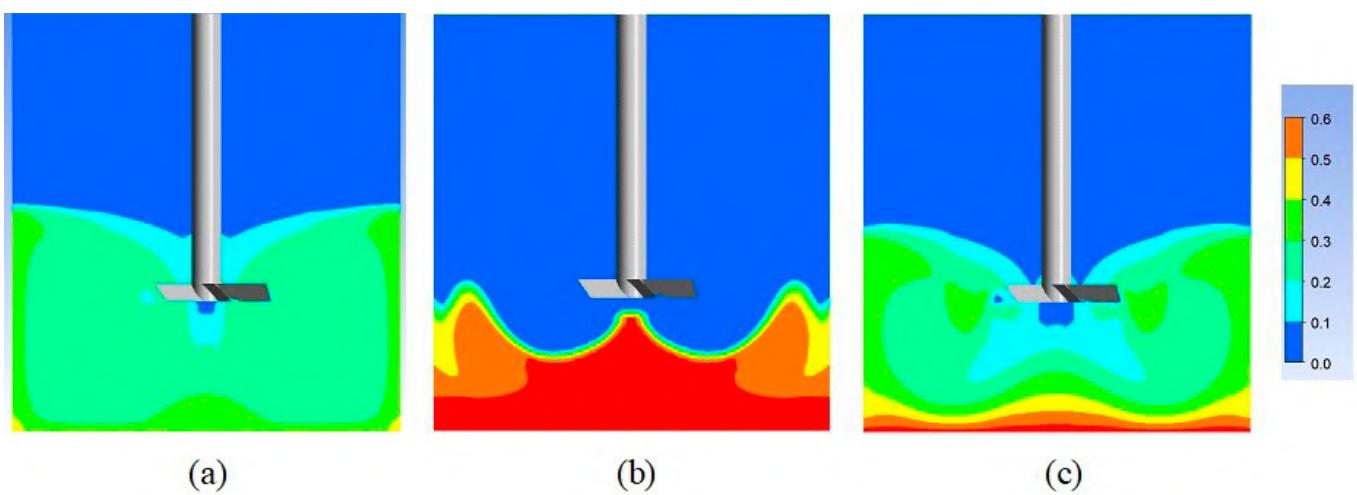
The aim of this contribution is to identify the effect of the interphase momentum and turbulent transfer terms formulation for obtaining fully predictive and reliable estimation of the solid concentration distribution and of the liquid mixing time in the realm of the Two-fluid Model, that is still the most suitable approach to simulate industrial equipment of complex geometry and large scale.

It is observed that the models, whose formulation depends on the equations averaging procedure, affect the simulated solid distribution mostly in incomplete solid suspension conditions. As shown in Figure 1(a), with one of the most widespread drag and turbulent dispersion force formulation (Burns et al., 2004), the contribution of the momentum transfer is included not only in the turbulent regions of the stirred tank, but it is artificially introduced also in the almost motionless region. It is apparent that a formulation for preventing the unphysical suspension of the solid phase in the regions of settled solids or almost stagnant liquid is required (Figure 1c), while neglecting the turbulent dispersion force leads to over predict the settled solid (Figure 1b).

The main modelling uncertainties and strength are discussed by comparing the computed results with the corresponding variables measured by Electrical Resistance Tomography. The results of this work can contribute to a better design and scale-up of many industrial operations involving particles suspensions in turbulent liquids.

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Multiscale modelling of moderately dense gas-solid flows using a hybrid discrete-continuum method

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Keywords | Multiscale modelling, CFD-DEM, TFM, Gas-solid flows

Gas-solid flows have been included in a variety of processes, including gasification, chemical looping combustion, and catalytic oxidation.

Fluidized, spouted as well as spout fluidized beds and blast furnaces are examples of the gas-solid contactors. Modelling of these flows is demanding since these involve a wide range of length scales. In the past decades, two main modelling approaches have been used to analyse such gas-solid flows. On the one hand, the Eulerian-Eulerian approach (e.g. two-fluid model, TFM), where both phases are treated as interpenetrating continua and the particle collisions are considered by the kinetic theory of granular flows (KTGF), is preferably used for larger scales. On the other hand, the Eulerian-Lagrangian approach (e.g., CFD-DEM), where the gas phase is considered as continuous phase and the particles are tracked individually, is commonly used for more detailed descriptions. Although tracking the individual particles leads to accurate results, it needs more computational effort than the Eulerian-Eulerian approach.

This research aims to apply a CFD-DEM magnification lens in moderately dense gas-solid regions exhibiting high gradients such as spouts in fluidized beds or obstacles as well as bends in reactors. The lens magnification adopts a small CFD-DEM area inside the TFM simulation in the regions mentioned earlier, and both methods communicate through a corona layer. Here, it is crucial to resolve interparticle forces, which are not sufficiently resolved in TFM. Thereby, we employ OpenFOAM in the case of TFM, where new boundary conditions (Schneiderbauer et al., 2012) and frictional models (Chialvo et al., 2012) have been implemented. In the case of CFD-DEM, the open source framework CFDEMcoupling is used.

Design Assessment of Pelton Turbine Injector for Hydro- abrasive Erosion Performance

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Keywords | Pelton turbine; Injector; Sediment Erosion; Turbine steel; CFD-DEM

The problem of erosion due to sediment-laden water in hydropower stations is quite severe, especially in those located in the Himalayan region. In fact, it is one of the major causes of reduction in the operational economy for hydro-power generation. In Pelton turbines, the injector assembly is subjected to severe erosion, yielding it inoperable in a relatively short period of time when compared to other parts of the hydropower plant. The surface erosion of the injector assembly due to solid particles has a detrimental effect on the water jet characteristics and hence, the turbine efficiency. Thus, installing an injector assembly with better erosive performance helps to improve the operational economy and reduces downtime. The present study aims to numerically investigate the effect of the design parameters of the Pelton turbine injector on its erosion. Three different injector designs, conventional design (nozzle and spear angle of 90o and 50o, respectively) and two other designs highly recommended for better hydraulic performance (nozzle and spear angles of 110o and 70o, and 150o and 90o, respectively) have been considered. The simulations have been conducted using the CFD coupled discrete element method (DEM). Multi-size sand particles in the range of 75 μm to 300 μm have been selected to conduct the study. A semi- empirical erosion model calibrated for turbine steel (CA6NM) has been adopted to estimate the erosion rate. The particle collision characteristics and erosion distribution on the nozzle and spear for different injector designs at full and partial nozzle opening conditions have been studied. Further, the zone of maximum erosion has been identified for different spear positions and injector designs. An understanding is developed on the relative erosion of the new injector designs to the conventional design. The current study contributes to the development of important engineering insights for the selection of injector designs for operation in sediment-laden water, as well as the control of the problem of jet breakage due to injector assembly erosion.

Investigation of fluid-particle flow in gas-solid cyclone separators using DEM-CFD simulation

Di Renzo, Alberto (1); Napolitano, Erasmo Salvatore (1); Alfano, Francesca Orsola (1); Di Maio, Francesco Paolo (1)
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Keywords | Separation; Cyclone; Simulation; DEM; Coarse graining

Gas-solid cyclones are utilized in numerous purification processes involving dedusting or demisting and in continuous separation applications such as in circulating fluidized beds. The complex fluid flow developing in tangential inlet cyclones has been the subject of several research

works. Simulations have revealed the key features of the gas flow as well as the solids separation efficiency. The great majority of simulation approaches considered one-way coupled strategy, with some exceptions considering two-way [1]. Recently, some authors considered four-way coupled simulations using Discrete Element Method (DEM-CFD) [2]. Such method provides a coarser description of the gas flow but incorporates a detailed modelling of the interparticle and particle-wall collisions, including angular motion, rolling friction and, if relevant, cohesion. Such features are highly desirable for example in studying high-loading cyclones. However, the number of particles often overcomes the feasible limits for DEM-CFD and coarse graining (CG) methods have been proposed to speed up the computations. CG methods produce interesting results in terms of the representation of the macroscopic quantities, but their ability to capture the microscopic features is still not fully demonstrated [3]. In this contribution, DEM-CFD strategies to perform simulations of the flow field in tangential (Stairmand's high efficiency design) and axial flow cyclones (Uniflow design) are illustrated and discussed. Results on the effect of the coarse graining level is illustrated with applications, along with the influence of several parameters related to the elastic and dissipative properties of the collisions and to the fluid- particle coupling. It is shown that while pressure drop and collection efficiencies are captured with satisfactory agreement, some features of the particles flow like strand formation at the walls can be lost when real particles are represented by computational grains (or parcels). Attempts to compensate for these effects are illustrated and discussed.

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A Combined PEPT/X-ray Study of the Influence of Liquid Loading on Fluidised Beds Dynamics

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Keywords | Fluidised Bed, Waste Plastic Recycling, Pyrolysis, Liquid Bridges

Fluidised bed plastic pyrolysis presents an opportunity to fight the global plastic waste crisis, by enabling a full circular economy of carbon in plastic. Despite decades of intense research into fluidised beds, there remain problems with these types of reactors. Defluidisation, dead zones, and hot spots are problems that effect even single phase systems. Melting plastic in the reactor creates a significant amount of liquid in the system and, despite the common understanding that this liquid evaporates very quickly, contemporary research shows significant time elapsing between melting and evaporating. Due to this, it is necessary to understand the effect of liquids on the hydrodynamics of the system. Using Positron Emission Particle Tracking (a high-resolution a Lagrangian particle tracking technique), fast X-Ray Radiography (capable of acquiring high-resolution Eulerian data), and conventional pressure drop analysis, we analyse the influence of liquid loading and viscosity on both micro- and macro-mixing and various other key properties pertaining to the dynamics of the fluidised bed. While confirming the phase states found in earlier studies we find new, stronger effects of the liquid loading on the minimum fluidisation velocity and demonstrate how dispersion and mixing of solids on a particle based level change. Furthermore, we show how bubble frequency, size, and channel sizes influence solid circulation and mixing, therefore contributing to mixing characteristics, providing new fundamental knowledge of these widely-used industrial systems.

A study on deep-sea nodule acquisition under different flow field conditions based CFD-DEM coupling method

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Keywords | Discrete Element Method; Nodules; Silts; CFD-DEM coupling method; Nodule Extractor

The development of deep-sea nodule resources is a huge and extremely complex project, which is restricted by the influence of the marine environment and strict marine environment protection, and the impact of the mining process of nodule extractor technologies on the

marine environment is also concerned. According to the geological conditions of potential exploring ocean areas, the silts and nodules can be simulated by adopting different sizes and shapes of particles in discrete element method (DEM). The flow state of water in various conditions can be well calculated based on the computational fluid dynamics (CFD) method, and the nodule extractor is also constructed in the numerical model. The numerical simulation on the interaction between nodules, silts, water and extractor are carried in this study based on CFD-DEM coupling method. The results show that the process of nodules collecting is affected certainly by the abundance and burial depth of nodules.

Similarly, the structure of the extractor (the size, angle and spacing of the helical gear), the rotational speed and advanced speed are also important, which will greatly influence the silt behaviour and nodule recovery rate. The research content of this paper can provide an advanced and effective analysis method for the future research on geotechnical and environmental problems, involved in the development of marine resources and new nodules extractor.

The drag conundrum: Modeling fluid-particle momentum exchange in fluidized beds

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(1) Particulate Solid Research Inc

Keywords | eulerian-eulerian modeling, fluidization, drag

Predicting accurate fluidized bed behavior with numerical fluid mechanics software provides promising advancements in various industrial operations via reduced operational costs, more streamlined design processes, mitigating costly shutdowns, enhanced troubleshooting, etc. The drag force governs the fluid-to-particle momentum transfer, and thus properly modeling drag is critical for predicting the overall fluidized bed behavior. Numerous drag models currently exist, and the resulting drag prediction can vary up to an order of magnitude between drag models. Understanding the influence of drag model formulation on fluidized bed behavior is an essential step towards enhancing the accuracy of model predictions. In this work, experimental measurements from bubbling and turbulent fluidized beds are compared to model predictions incorporating a wide range of drag model formulations. The simulation results demonstrate that the variability of the simulation predictions resulting from changes in the drag model formulation can have staggering effects on the simulation outcome. Furthermore, conclusions regarding the accuracy of model formulation change with particle properties and superficial gas velocity.

coatSim: A Simulation Based Digital Solution for Optimization of Production Processes for Particle-Laden Flows

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(1) ZHAW (Zurich University of Applied Sciences)

Keywords | simulation, particle laden flows, powder coating, digital twin, process optimization

In industrial applications involving particle-laden flows such as powder coating, often ad-hoc heuristic methods are employed to design or adjust processes in light of varying process-parameter settings. This labor-intensive approach often leads to inefficiencies and sub-optimal outcomes, as the entirety of the design space is not taken into consideration. The alternative, much more efficient approach entails knowledge-based process adjustments. However, the latter is often beyond the capabilities of industrial appliers, since it requires an extensive expertise and highly skilled labor force. The numerical Multiphysics-simulation software coatSim has been designed in order to bridge the gap between the required expertise and the need for a predictive tool for industrial knowledge-based process design. coatSim is a user friendly, cloud-based simulation software that can be customized for any industrial application involving flows laden with solid or liquid particles or indeed ionic transport mechanisms. Its core technology relies on an OpenFOAM based, Eulerian-Lagrangian solver, where particle flow-paths, particle-particle and particle-substrate interaction effects can be modeled in light of acting fluid dynamic-

, and body forces as well as particle dynamic effects. In the case of powder coating applications, the body forces are the electrostatic forces generated by the coating pistol and gravity. The solver has been validated on the basis of an experimental campaign, where substrate plates were coated in 72 configurations. Thereby measured relative coated volume could be predicted up to a variation of 5%. The underlying solver can handle domains comprised of several independent mobile regions, on each of which a periodic motion in terms of any arbitrary Fourier

series can be imposed. This enables realistic duplication of industrial applications, as motion is an intrinsic part of most of them. The core solver technology has been bundled with an intuitive user interface containing application specific databases, geometry manipulation tools and automatic mesh generation capabilities. Hence the user can generate a simulation case with minimal effort and as a next step expand it into a parameter study by prescribing the values of relevant parameters to vary. The software is directly coupled to cloud computing facilities to harness massive simultaneous cloud computing functionality.

Influence of pulsatile flow on solid-liquid mixtures in milli flow reactors

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(1) KU Leuven

Keywords | Solid-liquid mixtures; pulsatile flow; flow regimes; velocity profiles

The increasing industrial interest of performing heterogeneous solid-liquid reactions in flow, requires insight and control over the sedimentation, fouling and clogging behavior of solids. Therefore, determination of the behavior of solids in pulsatile flow reactors can be advantageous in prevention of sedimentation and for performing heterogeneous reactions in flow. This research determines the flow regimes and velocity profiles of solids in pulsatile flow with a maximum amplitude of 0.33 m and maximum frequency of 2.8 Hz via recorded images with a high-speed camera. With the knowledge of the velocity profiles and flow regimes, the influence of the pulsator settings can be researched and the correct pulsator settings can be chosen to prevent sedimentation and to execute heterogeneous reactions in milli flow reactors. The influence of the liquid density, solid density and solid size on the flow regimes and sedimentation of solids are researched. In our experiments, the used solids are glass beads (2500 kg/m^3) with a varying particle size of 40 – 120 µm and polymethylmethacrylate (1200 kg/m^3) with a particle size of 60 µm. The studied liquids are water (999 kg/m^3), ethanol (860 kg/m^3) and aqueous potassium bromide-solution (1260 kg/m^3). Next to the solid-liquid properties, the pulsatile flow is investigated by three different settings: amplitude and frequency of the pulse and feed flow rate of the mixture. Results show the importance of the settings of the pulsator on the behavior of solids. With glass-beads-water mixtures, it is visible that upon increasing the amplitude, the frequency can be decreased to prevent sedimentation of particles. Also, different solid-liquid properties have an influence on the flow regimes and sedimentation of particles. In case of a low density difference between the solid and liquid, the frequency can be decreased with respect to mixtures with high density differences between the solid and liquid. It can be concluded that a high-speed camera is easily implementable to research the flow regimes and to determine the pulsator settings to prevent sedimentation of particles.

FLASH COMMUNICATIONS

Unstable sphere sinking in a fluidized bed at higher air velocity; 1) experimental study

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Keywords | Fluidized bed; Dry separation; Minimum fluidization velocity

A gas-solid fluidized bed is formed when air is continuously driven into the bottom of a particle bed. It is well known that the fluidized bed has liquid like properties such as density and viscosity. Float-sink of spheres in the fluidized bed is one of the characteristic phenomena. Dry separation based on the float-sink can be used as a substitution for conventional wet separations. When the dry separation is applied to waste treatment, for instance, to separate aluminum and copper in waste nonferrous metals, a gentle fluidization at lower air velocity is utilized. The dry separation is also applied to waste combustion; waste is burned in the fluidized bed and non-combustible materials sink in it. For the waste combustion, the particle bed is generally fluidized at higher air velocity, in which the non-combustible materials “sometimes” don’t sink but float or drift. The origin and the mechanism have not been studied in details so far. Here we experimentally investigated the float-sink of spheres in the fluidized bed at wide ranged various air velocities using density adjusted spheres and glass beads as the particle bed. We found that basically the spheres float or sink based on the density difference. However, the spheres having the density close to the

fluidized bed density, which are supposed to sink, may float, drift or sink at higher air velocities. Intense fluidization would avoid the stable sinking of the spheres at the higher air velocities.

Numerical simulation of binary mixture of particles in a fluidized riser

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Keywords | riser, cluster, binary particle, CFD-DEM, fluidization

Gas-particle flow in the risers is characterized as a “core–annulus” pattern, where the more dilute particles flow upward in the core of the reactor, and the denser particles flow downward close to the wall. The clusters near the wall of the riser were found self-organized by several particles in stable configurations, falling faster than isolated particles. The clusters appeared as regions with a higher concentration of particles and exhibited uniform velocity spatially and temporally. The existence of the cluster alters the hydrodynamics and affects the performance of the reactor. Therefore, the cluster characteristic is in high concerned in the previous research.

In this study, the fluid dynamics of the binary particles in the riser is investigated via a numerical method, CFD-DEM, to predict the characteristics of clusters formed by the binary types of particles. The result illustrates the interaction of these different types of particles in the cluster, the velocity variation of particles affected by the others due to this interaction between the binary particles and the effect of the stability of clusters with binary types of particles.

Modelling entrained-flow slagging gasification of solid fuels with particle deposition and near-wall segregation

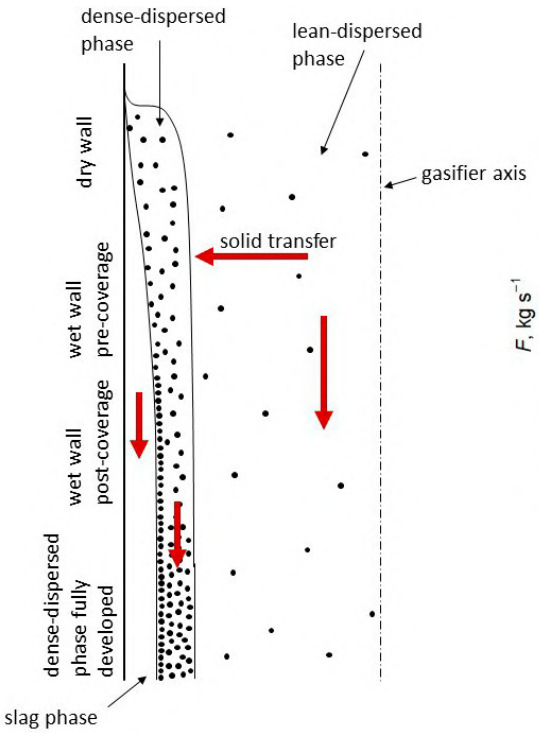
Troiano, Maurizio (1); Montagnaro, Fabio (2); Solimene, Roberto (3); Salatino, Piero (1)
(1) Department of Chemical, Materials and Industrial Production Engineering, University of Naples Federico II, (2) Department of Chemical Sciences, University of Naples Federico II, (3) Institute of Sciences and Technologies for Sustainable Energy and Mobility, National Research Council

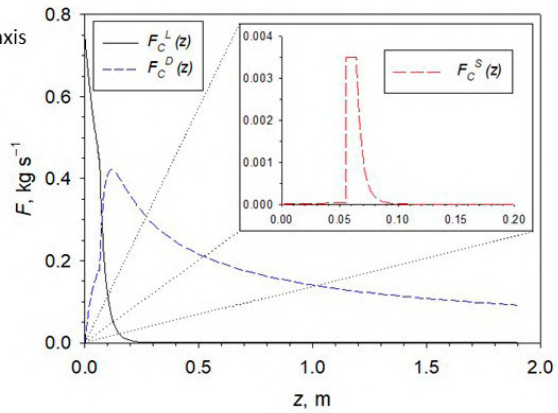
Keywords | Entrained flow; gasification; ash; deposition; segregation; slag

Gasification keeps a pivotal role in the exploitation of solid fuels for energy conversion and production of chemicals. Entrained-flow gasifiers (EFG) show very high carbon conversion, good adaptability to the fuel quality (from bituminous to low-grade coals, waste and biomass), production of high-purity syngas with very small amount of tars. Most industrial EFGs operate in slagging mode: char/ash particles are heated above the ash melting point, hence they deposit on the wall, forming a slag layer which acts as a protective coating. However, slag build-up needs to be carefully controlled to avoid plugging and clogging as well as refractory bricks corrosion.

The performance of slagging EFG is critically affected by the behaviour of char/ash particles as they interact with the wall. Different micromechanical patterns can establish, depending on parameters such as particle and wall temperatures, stickiness of the particles and of the wall layer, char conversion, particle kinetic energy, surface tension of the slag layer. Despite several numerical studies on the behaviour of particles and slag, the fate of char/ash particles in EFGs still lacks predictive tools based on mathematical and physical modelling of particle–wall interaction. In the present work a compartmental model of an entrained-flow slagging coal gasifier is reported, which embodies a sub-model for the description of particle-wall interaction and formation of segregated phases. For this task, impact experiments were performed to develop closure equations describing deposition and inelastic rebound of char/ash particles after collision with the wall.

Results of model computations highlight that the dense-dispersed phase develops very soon after particle feeding, triggered by the presence of the slag layer which induces particle adhesion and inelastic rebound. The establishment of this dense- dispersed phase represents a very stable condition. It is beneficial to the performance of the gasifier, as it increases the particle residence times and promotes near-complete carbon conversion. Furthermore, the extent of particles undergoing inelastic rebound and the particle velocity of the dense-dispersed phase dictate the early vs late formation of the dense- dispersed phase and the coverage of the slag layer.


dense-dispersed phase
lean-dispersed phase
dry wall
gasifier axis
solid transfer
wet wall pre-coverage
wet wall post-coverage
dense-dispersed phase fully developed
slag phase


F_C^L (z)
F_C^D (z)
F_C^S (z)
F, kg s^{-1}
z, m

5 PARTICLE FORMATION AND DESIGN

KEYNOTE LECTURES

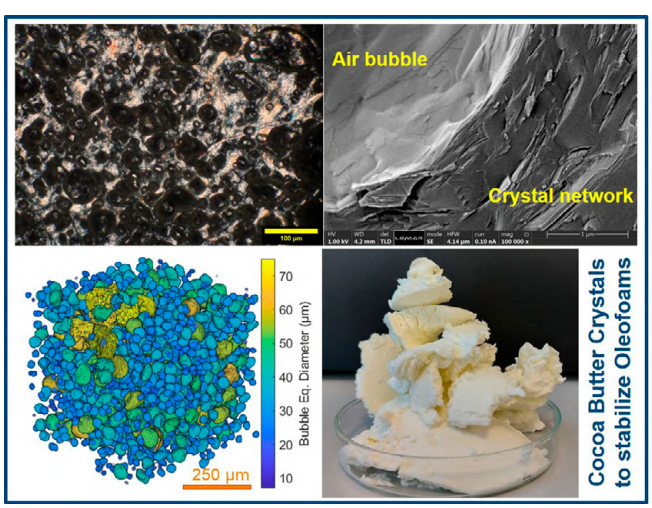
A Crystal Engineering approach for rational design of novel sustainable food, agrochemical and pharmaceutical formulations

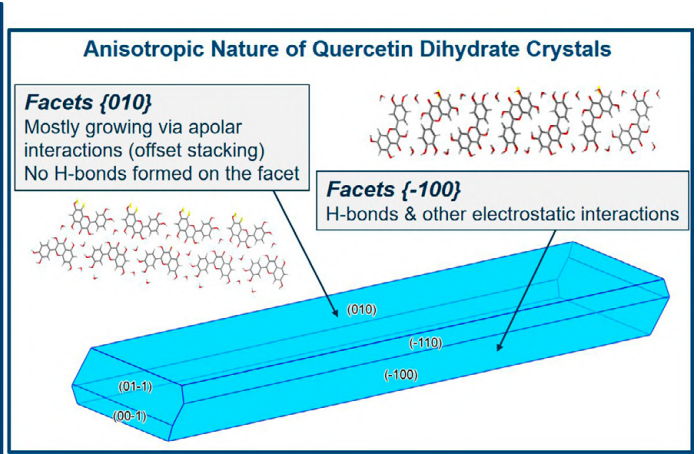
Simone, Elena (1); Klitou, Panayiotis (2); Metilli, Lorenzo (3)
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Recent progress in pharmacology, plant biology and biotechnology has led to a dramatic increase in potency and specificity of new generation drugs, active agrochemical ingredients and food nutraceuticals. Unfortunately, this has been accompanied by poor bioavailability and water solubility: it is estimated that around 40% of the active pharmaceutical ingredients currently on the market and 60% of the ones still in development are poorly soluble due to their high molecular weight and structural complexity. These issues have pushed scientific research towards the design of complex formulations, with enhanced dissolution rate and bioavailability, which allow more efficient and targeted delivery of active ingredients (AIs). Multiphase systems (e.g., emulsions, foams) are a convenient and effective encapsulation and delivery strategy, particularly for oral and topical formulations. Currently, synthetic excipients, surfactants and specialty polymers are used to create formulations with enhanced properties. However, these compounds are derived from non-renewable resources through some of the most greenhouse gas-intensive manufacturing processes. For this reason it is now necessary to replace the common synthetic stabilizers used for these products with natural, biocompatible and biodegradable materials. These include natural micro and nano-particles (Pickering stabilizers) such as proteins, polysaccharides and various crystalline materials including cellulose, fats and polyphenols crystals.

Pickering systems are particularly promising since particles adsorb at interfaces more strongly than surfactants, providing significantly more stable formulations. The stability of Pickering systems is strongly affected by particle size and shape, but surface wettability is the most important property of Pickering particles. For faceted, anisotropic crystals surface wettability is not easy to determine. In fact, crystals present multiple crystallographic facets, whose surface properties depend on the type and directionality of the intermolecular interactions that characterize each facet.

The aim of this work is to understand how crystal properties affect the surface chemistry of Pickering particles and their adsorption at interfaces. Two case studies are presented: (1) a molecular modelling analysis where the crystallographic structure of different solid forms of quercetin was related to their facet-specific surface chemistry; (2) an experimental study on the ability of cocoa butter crystals to stabilize oil based foams for confectionary or cosmetic applications.





Digital Strategies to tailor Crystallization and Precipitation Processes in Industrial Applications – The Role of Rational Process Design

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(1) BASF SE

Keywords | Crystallization Precipitation Digitalization Digital Twin

Solid products gained from industrial crystallization and precipitation processes commonly have to meet a variety of requirements for processing or product design. For instance, crystallization processes, often dominated by their intrinsic thermodynamics, need to deliver defined grain sizes, purity levels, yields and targeted enantioselectivity or polymorph. In addition, product properties in downstream processing such as filterability, washing and drying behavior are of major importance. For precipitation processes, different parameters such as temperature, pH, reactant concentrations and additives are in a complex interrelation to each other, which collectively control phase composition, morphology, yield and the behavior in downstream processing as well.

Moreover, in fast developing markets, the task to find process optima with reasonable research efforts, the need to develop “smart” strategies for up- and down-scaling of complex processes with high probability of success and the competition- driven requirements for improved products are challenging.

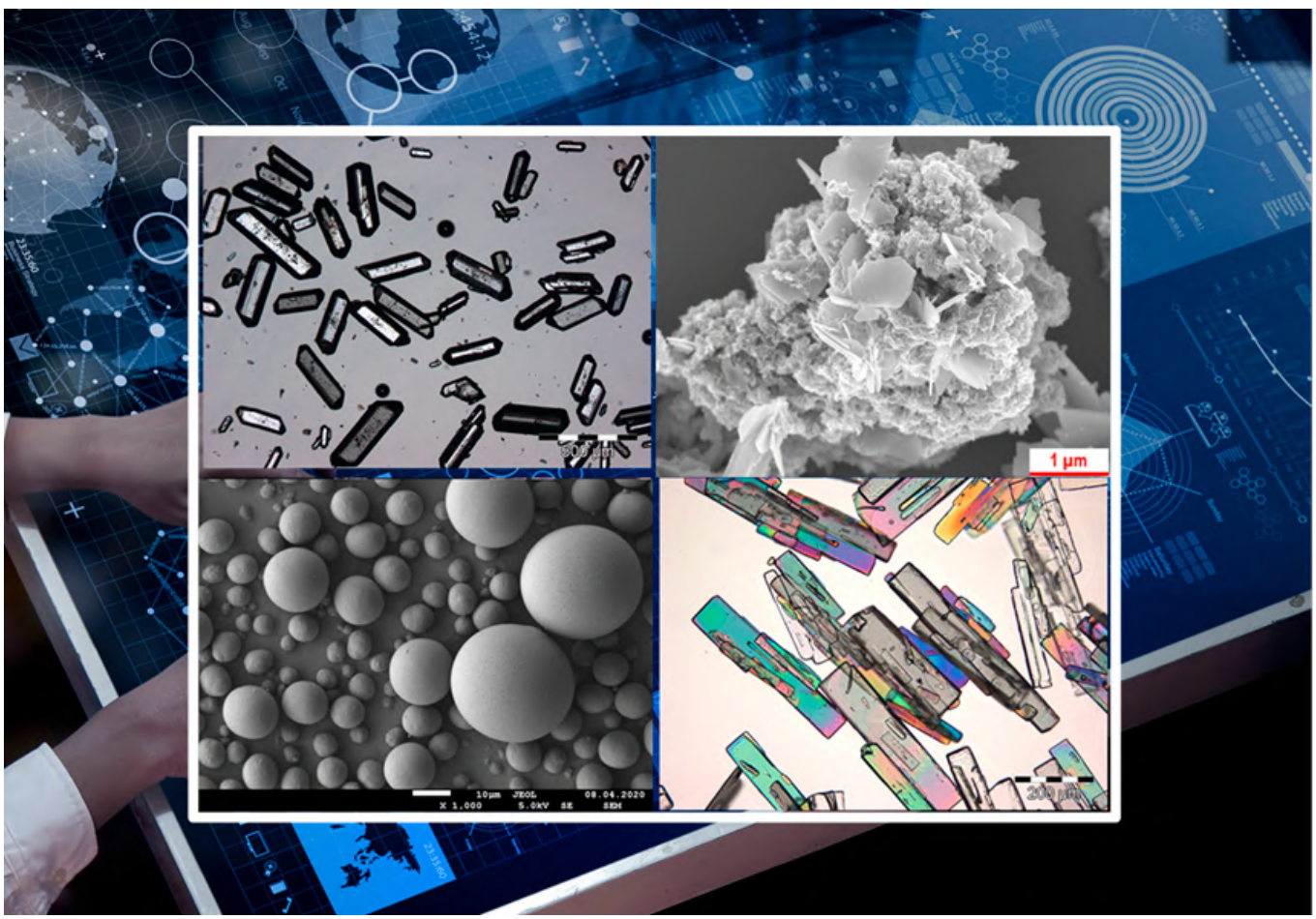


Figure 1: Towards computer-aided methods for solid formation products.

To meet these challenges, the contribution emphasizes the importance of rational process design and the growing opportunities by using digital methods for industrial solid-process engineering.

Based on industrial examples from crystallization of organic molecules such as agrochemicals and monomers to the precipitation of inorganic, functional solids such as battery materials and catalysts, it will be demonstrated how general, fast- moving buzzwords such as “digital twin”, “scale-comprehensive simulation” and “KI-supported processes design” can be filled with life. The contribution targets to present ideas on

how to improve existing industrial operating procedures using computer-aided methods.

Hydrophobic nanocellulose-based aerogels for selective oil removal: effects on aerogel characteristics and absorption performance

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Keywords | aerogels; modification; nanocellulose; porosity;

Cellulose nanofibers (CNF) have become one of the main topics of research for cellulose and polymer scientists and technicians. Aerogels made of CNF are a product of interest due to their lightweight, great specific surface and, thus, their huge porosity. Even they have been considered in the colloquial jargon as “solid smoke”. Moreover, aerogels made of CNF present great mechanical properties, fact that confers them a good dimensional stability. CNF, in its native form, have a huge hydrophilic character (even greater than the abovementioned sawdust), what would make also difficult their use for oil removal. In this sense, chemical modification of CNF is also in a growing stage, mainly focused on the hydrophobization of their surface. In this sense, aerogels made of cellulose nanofibers from bleached kraft hardwood pulp will be developed by three well differentiated methodologies: TEMPO-mediated oxidation, enzymatic hydrolysis and mechanical. In addition, before preparing the aerogels, the obtained CNF will be properly modified by the addition of AKD, ranging the dosage between 0 to 10% and they will be submitted to some static and dynamic tests in order to determine the feasibility of using them as selective oil removers, as well as their capability to be used more than once (recycling tests).

The visual aspect of the obtained aerogels was similar regardless the type of CNF used, as well as the added amount of AKD. Those aerogels modified with AKD presented, at first sight, a high water contact angle, which was quantified during aerogels characterization for each type of CNF and each modification degree. The use of the obtained aerogels showed a strong and environmentally friendly alternative for oil removal in the high seas.

Remote control release of antibiotics from structured microparticles

Lizoňová, Denisa (1); Navrátil, Ondřej (1); Slonková, Karolína (1); Mašková, Lucie (1); Zadražil, Aleš (1); Štěpánek, František (1)
(1) University of Chemistry and Technology Prague

Keywords | antibiotic depot system; remotely controlled release; core-shell microparticles; magnetic nanoparticles; wet- milling; phase change material

Drug depot systems are usually based on the spontaneous dissolution and diffusion of drugs or prodrugs from a reservoir. While this might be useful in cases that require constant plasma concentration of the drug over time, there are also situations where this may not be desired and rather multiple bursts of the drug at well-defined time intervals are preferred. This work presents an antibiotic depot system that enables the repeated release of norfloxacin in precise doses, controlled by an external radiofrequency magnetic field. This system consists of composite microcapsules with a core-shell structure. The core contains micronized drug particles embedded in a wax-based hydrophobic matrix. The shell is formed by an alginate hydrogel with immobilized magnetic nanoparticles, which allow for heating when exposed to a radiofrequency magnetic field. When the melting point of the core is locally exceeded, the antibiotic particles are mobilized, and some of them get in contact with the external aqueous phase and dissolve. Such drug release can be controlled in an on/off manner by a chosen sequence and duration of radiofrequency pulses. The capacity of the presented depot system is shown to be significantly higher than that of only diffusion-controlled systems containing a pre-dissolved drug. The functionality of the depot system is demonstrated in vitro, where 3 radiofrequency cycles were shown to effectively kill all bacteria in the samples.

ORAL COMMUNICATIONS

Modelling and Identification of Mg(OH)2 precipitation kinetics from highly concentrated Mg2+ solutions

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Keywords | Crystallization, Precipitation, Modelling, Precipitation kinetics

In recent years attention has been placed on the recovery of minerals from unconventional resources. In this context, Cipollina et al. (2015) demonstrated the feasibility of producing Mg(OH)2 from saltworks bitterns. The Mg(OH)2 precipitation is characterized by an extremely fast chemical reaction and it is ruled by a mix of complex phenomena. In this work, the Mg(OH)2 precipitation is described and modelled developing a 1-D precipitation model of a 2 mm diameter circular cross- shaped T-mixer. For the first time, Mg(OH)2 precipitation kinetics are reported for highly concentrated Mg2+ solutions, which can be used for the design of innovative crystallizers unit.

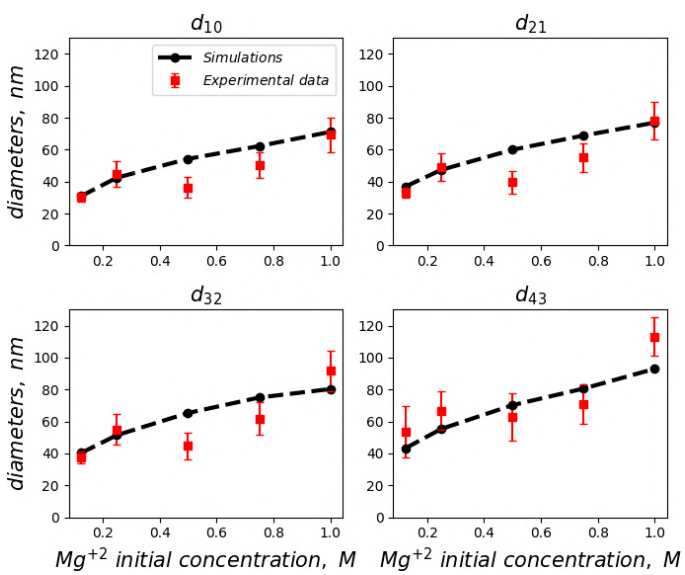
Micro-mixing significantly affects precipitation processes, (Baldyga et al., 1995), therefore a deep knowledge of the turbulence in a reactor is required. In this regard, micro-mixing was studied by computational fluid dynamics simulations studying the neutralization reaction between HCl and NaOH solutions mimicking the Mg2+ and OH- ions one. The approach (Marchisio et al., 2016) was implemented for the description of the extremely fast chemical reaction. The developed CFD model was validated through comparison with experimental results (Romano et al., 2021). mean turbulence profiles were then extracted and employed in the 1-D precipitation model.

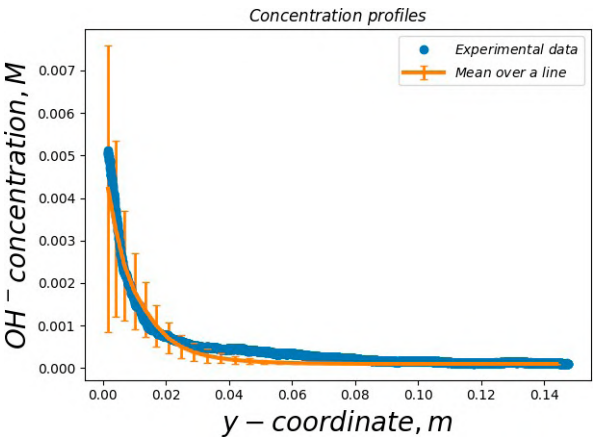
The 1-D precipitation model, implemented in MatLab® environment, solves the Population Balance Equation (PBE) for Mg(OH)2 particles taking into account: the reaction between Mg2+ and OH- ions, with Bromley activity coefficients correction (Bromley, 1973), primary nucleation, molecular growth, aggregation kernels (Bałdyga et al., 2001). The PBE is solved using the Quadrature Method of Moments approach (Marchisio et al., 2003 a; b).

Figures show model results against experimental data. A very good agreement can be observed both for neutralization and precipitation processes.

Acknowledgment

This project has received funding from the European Union's Horizon 2020 research and innovation programme under Grant Agreement No. 869467 (SEArcularMINE). This output reflects only the author's view. The European Health and Digital Executive Agency (HaDEA) and the European Commission cannot be held responsible for any use that may be made of the information contained therein.





Development of ann calibration model for estimating xylometazoline hydrochloride concentration in crystallization process

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Keywords | crystallization, process analytical technology, ATR-FTIR spectroscopy, calibration model, xylometazoline hydrochloride, artificial neural network

Active pharmaceutical ingredient (API) crystallization is among the least understood manufacturing processes (Yu et al. 2004), causing many batch failures and leading to significant financial losses. Therefore, modern pharmaceutical production tends to apply process analytical technology (PAT) that allows in-depth process understanding, real-time monitoring of product quality, and a Quality by Control (QbC) approach for the purpose of right-first-time production. In order to monitor and control the crystallization process in real-time, the development of the calibration model for the continuous estimation of solution concentration is necessary. During the last decade, artificial neural networks (ANNs) has proven to be a powerful tool for modeling in chemometrics, enabling analytes concentration prediction and compound classification (Boger 2003)

In this work, the development of the ANN-based calibration models using in-situ ATR-FTIR spectroscopy for estimating the xylometazoline hydrochloride concentration in the cooling crystallization process is presented. Spectra and temperature have been collected for 10 different solution concentrations during the cooling process. The input data matrix consisting of the temperature and spectra for the corresponding concentration was further preprocessed by scaling, normalization, standardization, and smoothing techniques. The quality of developed models was evaluated by statistical validation criteria on an independent dataset. The results show significant applicability of particular models for continuous assessment of the xylometazoline hydrochloride concentration.

Acknowledgment:

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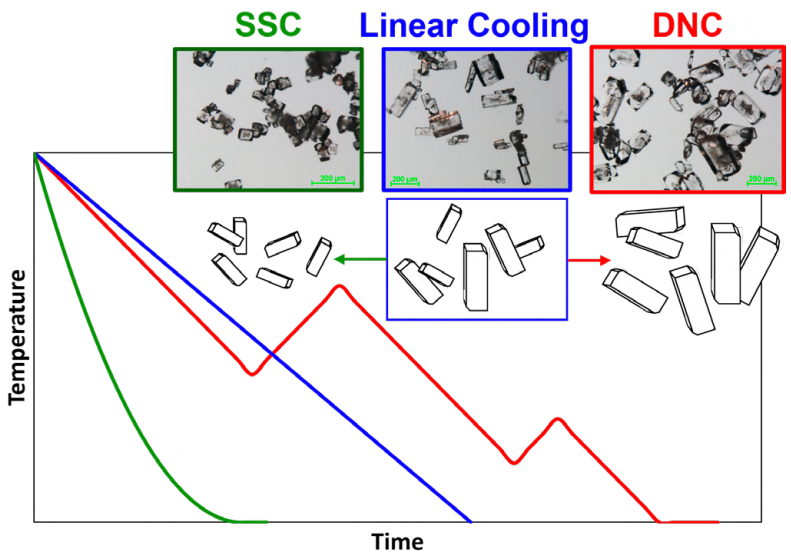
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Process Intensification of Energetics Crystallization via model- free Quality-by-Control Direct Design and model-based Digital Design Approaches

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Keywords | Crystallization, Energetic Materials, Process Control, Process Analytical Technology Tools, Process Intensification



Crystallization is an essential process of solids manufacturing and is left unoptimized in several fields, including energetics manufacturing. Unoptimized crystallization protocols can lead to particles with undesired physical and chemical characteristics, such as particle morphology, sensitivity, detonation potential, manufacturability, and overall crystal quality. First manufactured for use in World War II, nearly a century ago, Research Department/Royal Demolition Explosive (RDX) and High Melting Explosive (HMX), have been applied to military munitions, propellants, and general explosives and continue to be two of the largest manufactured energetic materials. Although researchers have extensively studied the solubility of the common energetic materials, RDX and HMX, little work has been completed on the process intensification of RDX and HMX crystallization.

In this work, we demonstrate the combination of model-free Quality-by-Control (mf-QbC) and model-based digital design to develop a robust process intensification framework for the crystallization of energetic materials. mf-QbC allows for the minimization of experiments and exposure by utilizing feedback control strategies for desired critical quality attributes (CQAs) (Simone *et al.*, 2015). Small-scale experiments completed with Crystalline showed that RDX and HMX have high solvent power (solubility) in γ-Butyrolactone and good temperature sensitivity which is desirable for crystallization control. The two applied direct design approaches are direct nucleation control (DNC) and supersaturation control (SSC). DNC uses particle counts measurements collected with an in-situ focused beam reflectance measurement (FBRM) probe in a model- free closed feedback control approach, which introduces temperature cycling, stimulating controlled nucleation and crystal growth (Bakar *et al.*, 2009). SSC uses solution concentration measurements collected with an in-situ infrared (IR) probe in a model-free closed feedback control approach that drives the temperature profile to maintain constant supersaturation (Saleemi *et al.*, 2012). In application with in-situ process analytical technology tools, DNC and SSC allowed for the selection of crystallization design parameters that control the desired CQAs of RDX and HMX and the further optimization of the selected design parameters via model-based digital design. Intensifying the industrial crystallization of common energetic materials, such as RDX and HMX, will improve the quality of manufactured energetic materials, and future process development for the manufacturing of energetic materials.

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Preparation of rare-earths oxalates from double sulfate salts obtained by selective precipitation of Ni-MH battery leachates

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Keywords | precipitation process/rare earths/characterization/batteries recycling

Spent nickel-metal hydride (Ni-MH) batteries can serve as an attractive secondary source of rare earths elements (REEs). The valuable powder, called black mass (BM), extracted after crushing these batteries contain up to 15 wt % REEs. Recycling by hydrometallurgical processes at an industrial scale is a key issue. It involves two main steps: dissolution of the BM in an acid solution and recovery of these metals by selective precipitations [Zielinski et al., 2021]. The quality of the recovered REEs, depends on the control of the operating conditions of the precipitation stage and the washing of the filtered precipitates. Due to an initial material of complex composition and structure, the number of constituents likely to precipitate from the battery leachates is significant.

This work implements the precipitation of REEs, at a pilot scale, from an industrial leachate containing 15 g/L of REEs. The product of this first step is a solid solution of sodium-potassium lanthanide double sulfate, identified as (Na0.9K0.1)(La0.65Ce0.24Nd0.07Pr0.04)(SO4)2H2O by complementary characterization techniques (X-Ray Diffraction, elemental analyses and thermo-gravimetric analyses). In order to remove Na, K and S elements from this intermediate compound, the powder was then dissolved and re-precipitated as a hydrated rare earth oxalate REEs2(C2O4)3.nH2O using oxalic acid. The resulting oxalates were then characterized and valorized in the oxide form through calcination at 800°C. The precipitation experiments were conducted in a semi-continuous jacketed stirred reactor (0.6 L), temperature-regulated and equipped with a pH probe. During a standard experiment, the reagent was introduced by simple jet, at a constant flow rate. The filtration of the precipitates was carried out on a Buchner filter (pore diameters 16-40 µm). The influence of several operating parameters on the efficiency and kinetics of precipitation was studied: temperature, pH, reagent concentration and addition rate.

The results show that, depending on the operating conditions, the process yield can be higher than 95% and that it can be easily implemented at industrial scale.

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Development of continuous crystallization processes

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(1) KU Leuven, (2) KU Leuven

Keywords | Continuous cyrstallization, Solid handling, Fouling, Ultrasound assisted nucleation, Process design, Inline microscopy

Continuous tubular crystallizers have the potential to improve product quality and reduce costs for the pharmaceutical industry. However, solid handling, limited residence times and slow nucleation kinetics are still major issues for these crystallizers. In the last decade, dozens of tubular crystallizer designs have been proposed and evaluated and while a lot of progress has been made, these issues remain a challenge.

To overcome these challenges a new tubular crystallizer design was proposed and built as a proof of concept. This tubular process included:

- ultrasound to induce and control nucleation, even for kinetically slow compounds;
- a double jacketed heat exchanger to obtain a cubic cooling profile, which allows the reactor volume to be use as efficiently as possible;
- oscillatory flow to decouple mixing from the flow rate and to prevent fouling, clogging and sedimentation; microscopy coupled to open source image analysis to analyze the process.

The process was able to operate continuously for 4 hours while obtaining small particles around 75 µm with a narrow span of 1 and high yield between 95% and 100%. This makes this design one of the best performing tubular crystallizers of the moment and shows that tubular crystallizers are a realistic option for the pharmaceutical industry.

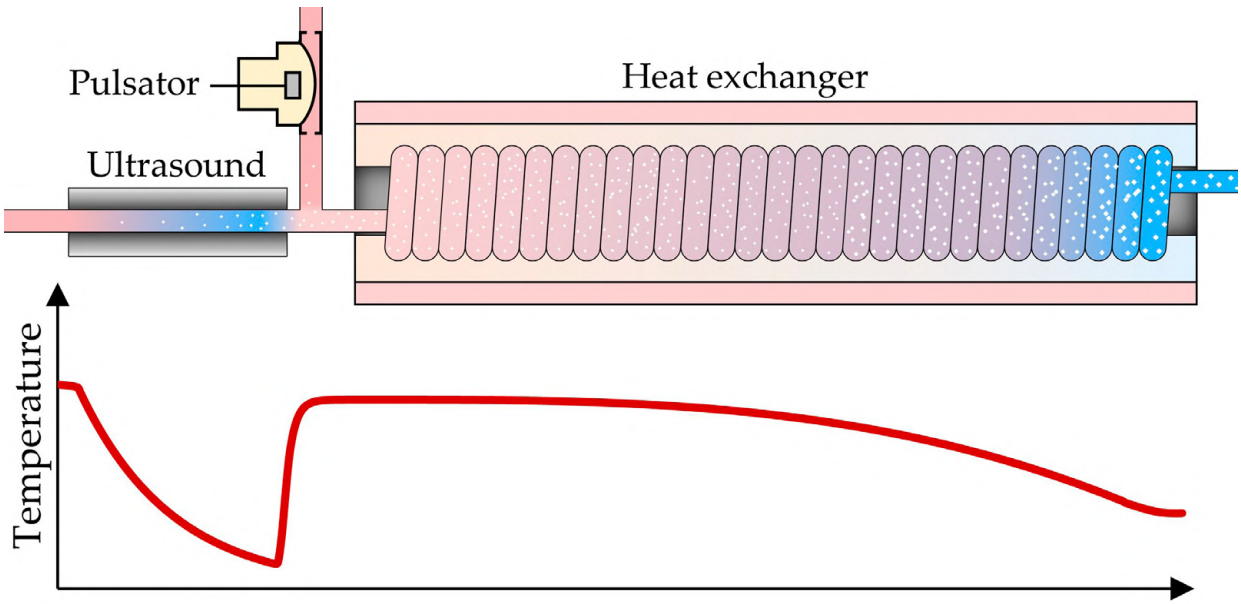


Figure 1: Graphical abstract.

Influence of particle structure on lipid oxidation stability of plant-based milk powders

Kurtz, Teresa (1); Haas, Klara (2); Schafer, Olivier (2); Meunier, Vincent (2); Heinrich, Stefan (3)
(1) Hamburg University of Technology - Institute of solids process engineering and particle technology, (2) Nestlé Research, (3) Hamburg University of Technology

Keywords | Encapsulation Freeze-drying Spray-drying

Dried emulsions such as milk powders have been widely used for decades across multiple categories and applications, from infant formula to milk chocolate. In recent years, environmental, health and animal welfare concerns have strongly increased the demand for plant-based milk alternatives, and with it the interest in plant-based milk powders (Vanga & Raghavan, 2018). In order to achieve the long shelf-life required for milk powder substitutes, present lipids and proteins need to be protected in the powder matrix against oxidation by environmental oxygen.

Particle structure is considered a crucial impact factor for shelf stability of dried emulsions (Fig. 1). Changes in surface to volume ratio, size and porosity of dried emulsion particles influence the access of oxygen towards encapsulated oil droplets as well as the amount of non-encapsulated, unprotected fat (free fat). Furthermore, vacuole size inside spray dried particles determines the amount of entrapped air available for lipid oxidation (Haas et al., 2019). Drying methods such as spray- or freeze drying and structuration techniques like spray granulation are established methods to alter particle structure.

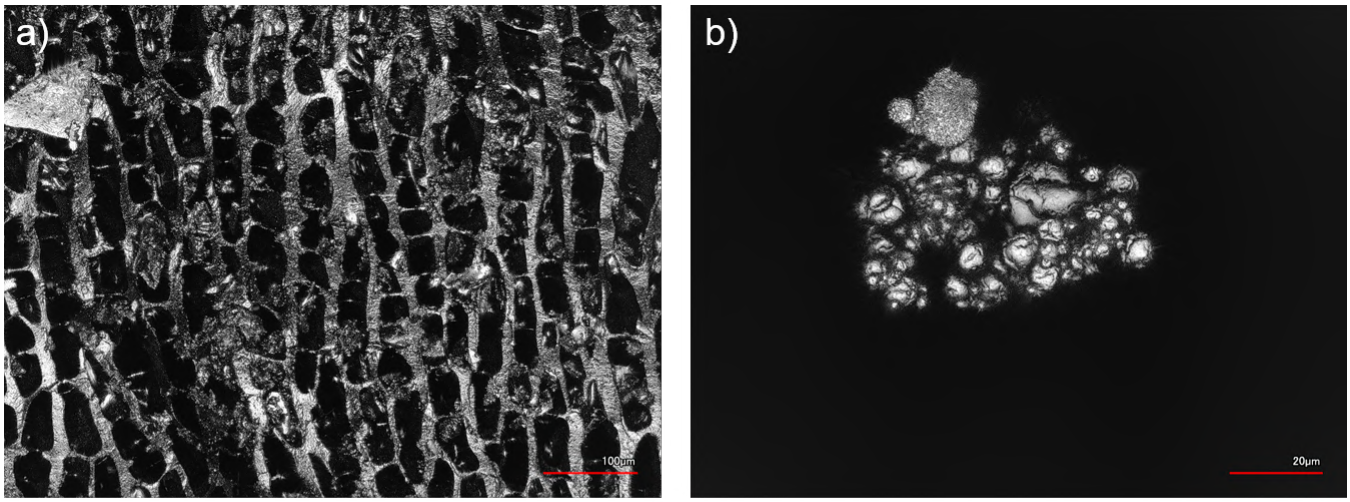


Figure 1 – Changing particle structures induced by different drying technologies. Confocal microscopic images of a) freeze-dried particle b) spray dried particles.

The present work investigates and compares the influence of certain structural parameters like drying kinetics, total solids and spray pressure on lipid oxidation of dried emulsions stabilized by pea and soy protein. Results show differences for particle morphology, encapsulation efficiency (ratio of encapsulated to non-encapsulated fat) and oxidation stability for powders produced with various drying and structuration techniques. The obtained data reinforces the significance of particle structure for the production of shelf stable powders.

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Fabrication of organic-inorganic hybrid microcapsules and their applications as drug carriers

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(1) Soka University, (2) Tokyo Institute of Technology

Keywords | Organic-inorganic hybrid microcapsule, Natural polymer, Aminosilane, Drug carrier.

Layer-by-layer (LbL) technique is one of the promising approaches to the design and development of microcapsules. Although numerous polyelectrolytes have been used to fabricate diverse multilayer shells with tunable compositions and structures, the encapsulation of drugs within polyelectrolyte capsules still presents a significant challenge because of the shell's high permeability. To overcome this, the functionalization of polyelectrolyte microcapsules through sol-gel processes would be very attractive because of its unique approach to fabricating hybrid microcapsules with novel physicochemical features, including a low permeability and intracellular degradability. However, only a few examples were reported for fabricating organic-inorganic hybrid microcapsules without the use of organic solvents and catalysts. Herein, we propose a simple method for the preparation of organic-inorganic hybrid microcapsules using natural polymers and aminosilanes by using layer-by-layer (LbL) method.

At First, CMC/AAPTS capsule was fabricated in two steps using sodium carboxymethyl cellulose (CMCNa) and aminoethylaminopropyltrimethoxysilane (AAPTS). Based on SEM and digital microscope observation, FT-IR analysis, and amino group contents measurement, successful preparation of CMC/AAPTS capsule with dense membrane was confirmed. Then, ibuprofen (IBU) was

loaded into CMC/AAPTS capsule as a model drug, and its release behavior was evaluated. The release rate of IBU in CMC/AAPTS capsule was dramatically slower than that of IBU powder without encapsulation. Therefore, encapsulating IBU in CMC/AAPTS capsules and delivering them into the human body may prevent the rapid release of IBU in the body and prolong the effectiveness of the drugs.

Next, the nucleic acid-encapsulated Alg/AAPTS capsule was fabricated in three steps by using sodium alginate (AlgNa) and (AAPTS). In this study, DNA primers were used as model nucleic acids. Under optimized conditions, the maximum DNA encapsulation efficiency of approximately 55% was obtained. Finally, we compared our results with those of conventional organic-inorganic hybrid microcapsules and found that although the encapsulation efficiency of nucleic acids was slightly lower, our proposed method is very simple requiring much fewer steps without using organic solvent and catalyst. These results indicate the effectiveness of our proposed method and the possibility of their application to various drug delivery systems.

Animal-Free and Environmentally Friendly Perfume Microcapsules

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(1) University of Birmingham, B15 2TT, (2) Birmingham

Keywords | Microplastics-free, Perfume Microcapsules, Complex Coacervation, Micromanipulation, Modelling

INTRODUCTION:In recent years, fast-moving consumer goods (FMCGs) have been enriched with fragrance-laden microcapsules towards maximising end-use costumers' fulfilment (Baiocco *et al.*, 2021). However, outstanding concerns around the use of animal-derived raw materials and microplastics have arisen worldwide. Increased self-awareness and enforced regulations against animal derivatives and pollutants have urged research to shift towards natural and plant-based ingredients. The present study aims to develop a novel microencapsulation approach using gum Arabic (GA) and fungally fermented chitosan (fCh) complexes as the shell precursors to preserve value-added oils against degradation, with a potential application in fabric care.

METHODS:GA-fCh based microcapsules with a core of floral-scented hexylsalicylate (HS) were fabricated via complex coacervation (CC) based on electrokinetic-turbidimetric studies. Highly reticulated microcapsule shells were assayed for their morphologic and structural properties *via* scanning (SEM) and transmission electron microscopy (TEM). Their mechanical properties were in-depth characterised using a micromanipulation technique. Moreover, both loading efficiency and leakage of microcapsules were quantified by UV-Vis spectrometry, and the corresponding data led to estimate the effective permeability of microcapsules shell by mathematical modelling.

RESULTS & DISCUSSIONS:HS-laden microcapsules with a relatively smooth surface were obtained (Figure1A). Their mean diameter, rupture force and Young's modulus in comparison with those of pure coacervate beads (CBs) which looked porous internally (Figure1C) are displayed in Table1. After 1 month, the amount of HS released from the microcapsules in 36% (w/w) aqueous propanol as an accelerated test was ~55% resulting in an effective permeability of $(2.6/pm0.2)/cdot10^{-14}m^2/cdots^{-1}$, which is similar to that of commercially available melamine-formaldehyde microcapsules (Long *et al.*, 2010). The shell thickness was determined to be 0.78±0.06 µm (Figure1B) which is in very good agreement with its theoretical thickness estimate (0.825 µm) and that based on finite element analysis results (0.95±0.05 µm) (Mercadé-Prieto *et al.*, 2011).

CONCLUSIONS:The electrokinetic-turbidimetric analysis of the biopolymers led to the successful encapsulation of perfume oil resulting in robust microcapsules, with a potential for fabric care products. Overall, the results suggest that animal-, microplastics- and hazard-free GA-fCh microcapsules may pave the pathway towards more sustainable applications in consumer goods, which can be appealing to more and more communities globally.

Table 1. Mechanical properties and encapsulation efficiency of microcapsules (Mean ± Standard Error)

	Particle size (μm)	Nominal rupture tension (N/m)	Young's modulus (GPa)
HS	27.5±1.5	71.6±4.0	1.0±0.1
CBs	17.2±0.8	456.1±47.6	0.6±0.2

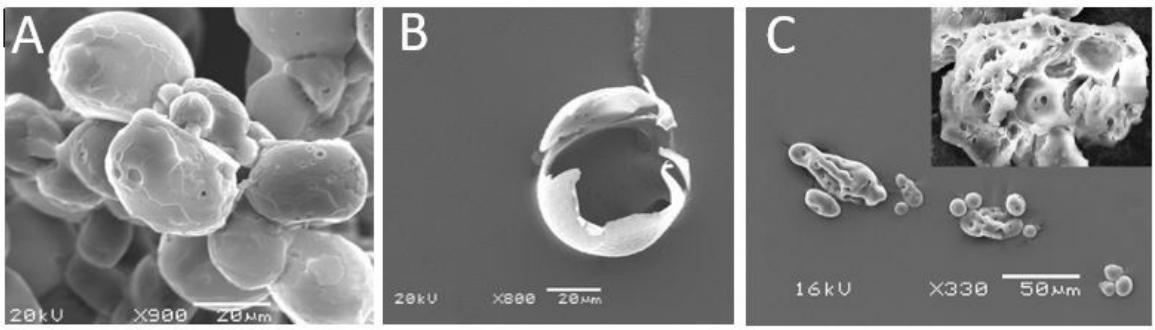


Figure 1: SEM images of intact (A) and broken microcapsules (B)

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Gel point simplifications as a tool to evaluate dispersion degree in nanocellulose suspensions and its application in papermaking

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(1) Universidad Complutense de Madrid

Keywords | gel point; aspect ratio; nanocellulose; papermaking; dispersion; pulp agitation

Cellulose nano/microfibers (CNFs) need to be well characterized to control its performance during industrial applications. Gel point (Øg) is used to estimate the aspect ratio (AR) of CNFs and is defined as the lowest concentration at which all flocs are interconnected forming a self-supporting network [1]. This methodology could be convenient to estimate the best CNF dispersion in water suspensions before a final application, such as in papermaking reinforcement. Nevertheless, Øg determination is tedious and time-consuming requiring at least 5 sedimentation experiments at different initial concentrations (Co) to obtain the curve Co vs. relative height (Hs/Ho) (Eq. 1). This procedure must be repeated for each dispersion degree, so a simplified method is first proposed to reduce experimentation and calculation time. In this research, the mathematical models commonly used to obtain Øg have been compared using 25 CNFs. A better correlation is observed using the MATLAB CSAPS function optimizing the "p" parameter. Once the best method to calculate Øg has been selected, a simplification of the experimental technique is presented based on one or two sedimentation experiments according to Eq.2 and using an optimal Co.

$$\emptyset_g = \lim_{Hs/Ho \to 0} \left(\frac{dC_o}{d\left(Hs/Ho\right)} \right) \qquad \text{(Eq. 1);} \qquad \emptyset_{g(estimated)} \approx \frac{C_o(optimal)}{\left(Hs/Ho\right)} \qquad \text{(Eq. 2)}$$

This methodology is successfully validated by using other CNFs, showing an error lower than 7% in Øg and 3% for AR. At least 60% of the experimental labour is reduced due to only one sedimentation is required in each dispersion degree or two if the Co selected is far to the

optimal. Results showed that as dispersion degree increases with the agitation, Øg reaches a minimum with a maximum in AR. Then, Øg increased again. Suspensions with lower Øg, in the intermediate region of agitation, present good dispersion behaviour with an open and spongy network structure without clusters. Higher stirring speeds shorten the nanofibrils and the networks collapse. Papermaking results showed that CNF dispersion at the minimum Øg before pulp addition, produces higher mechanical properties, even higher than when CNFs and pulp are agitated together. This knowledge would be crucial to understand why some industrial trials did not give satisfactory results.

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Engineered salbutamol sulfate balloon-like particles for career-free high dose delivery in DPI applications

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Keywords | Particle Engineering, Hollow, Ultrasound-assisted crystallization, Pulmonary therapeutics, Carrier-free formulation, High dose

The study presents balloon-like inhalable salbutamol sulfate micro-particles (SSμb) as a potential carrier-free drug delivery alternative to the drug-excipient formulation for dry powder inhalation (DPI). The SSμb particles are engineered through an ultrasound-assisted crystallization technique. The engineered particles exhibited a hollow spherical microstructure of inhalable size (4.26±1.91 μm) with rough surface of meso-macro porosity. Time-resolved static light scattering results using optical microscopy reveal that hollow structure is induced by mass diffusion from the interior area where crystallites are smaller and less compact to the exterior area at the two liquid interfaces. The P-XRD, DSC, FT-IR and LC-MS results of SSμb particles display 87.19% crystallinity with the same chemical identity as commercial salbutamol sulfate (SS, active pharmaceutical ingredient) with excellent flow properties. The in-vitro aerosolization shows the improvement in fine particle fraction (% FPF) and emitted dose (%) upto 37.4% and 95.1% respectively for SSμb compared to only 5.9% and 75.9% for SS as carrier-free formulation respectively. The increased %FPF (84% improvement) and emitted dose (20 % improvement) is mainly attributed to the spherical shape, low bulk density, high porosity, improved flowability and low surface charges of SSμb particles. This work provides a novel SSμb strategy for carrier-free DPI applications to achieve high drug dose requirements. The method has potential application for other water-soluble DPI drugs.

Mechanistic Modelling of Spherical Agglomeration Processes: Analysis of an Immersion-Driven Mechanism through Population Balance Modelling

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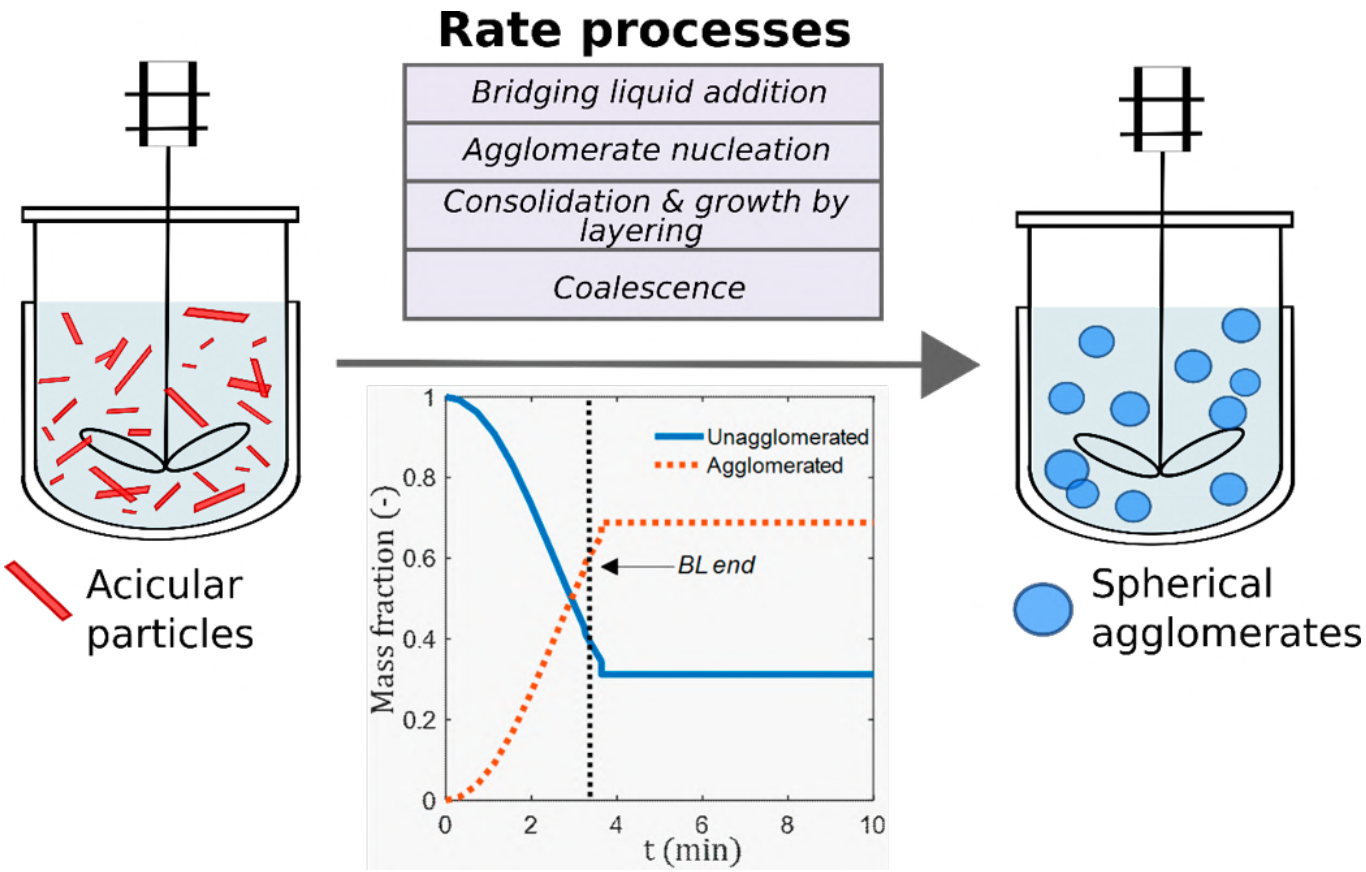
Keywords | Spherical agglomeration, mechanistic understanding, population balance modelling

A key challenge for the processing of pharmaceutical particles is the formulation of needle shaped crystals into the final drug product. Spherical agglomeration (post-crystallisation) is an emerging particle formation technique which has the capability to improve challenging particle shapes (needles, rods, flakes) and increase particle size. As a result, agglomerates display larger bulk densities, improved flow, easier filtration and downstream processing [1].

Whilst numerous studies have reported on experimental methods for the preparation of spherical agglomerates typically through a 'trial and error' basis, there remains a lack of mechanistic understanding and modelling concerning the fundamental rate processes and controlling parameters. Achieving mechanistic understanding of all the rate processes will provide a solid foundation to control the attributes of the produced agglomeration, such as size distribution and strength, as a function of process parameters and material properties. Interestingly, many of the mechanisms are understood to occur through stages similar to wet granulation: (i) wetting and nucleation of the particles by the bridging liquid; (ii) consolidation and growth of agglomerate nuclei and; (iii) breakage and attrition.

In this work, a new population balance model comprising key rate processes is developed and investigated. Here, initial wetting and nucleation, consolidation and growth of agglomerates by layering and coalescence mechanisms are described within a model framework. A parameter sensitivity analysis is undertaken on the effect of key process parameters on agglomerate attributes: average liquid volume fraction, size and size distribution and the time to completion kinetics.

Bridging liquid droplet size and bridging liquid to solids ratio are the most important parameters to control to achieve a specified product attribute range. The primary crystal size and process mixing intensity have more impact on the agglomeration formation and time to completion kinetics. This new proposed population balance model along with mechanistic rate kernels provides greater fundamental and process understanding for future design of spherical agglomeration processes.



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Multiple-unit dosage system: a flexible tool for combination therapy

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Keywords | MUDS, mini-tablets, polypharmacy, combined therapy, personalized medicine

Polypharmacy, defined as the regular use of at least five medications by a patient, is a growing concern for the geriatric population. It is associated with many negative consequences like complex medication regimens, increased total health care expenditure, and a higher risk for adverse drug events. A potential solution lies in the development of Multi-Unit Dosage Systems (MUDS) consisting of mini-tablets combined into a single hard gelatine capsule. As a result, instead of taking several medications per day, the patient will take only one MUDS with an equivalent substitute.

Another problem associated with the use of conventional tablets is their splitting. Although this is a very common practice in medicine

resulting in an optimization of drug dose and facilitating the administration of large tablets to patients, the physical inability to split tablets makes this process challenging and leads to patient non-adherence. Moreover, splitting not only reduces the amount of API delivered to the patient but is also inappropriate for formulations with a modified release. The MUDS, on the other hand, is designed, so one mini-tablet represents the minimum strength of one API, and by counting and filling the required number of subunits in a capsule, can be achieved the right dose for each patient.

In this study, we explored the formulation of individual mini-tablets for a selected set of drugs and their combination into MUPS. Three APIs used for combination therapy for the prevention of cardiovascular diseases were chosen as model drugs. Mini-tablets with a diameter of 1.5 and 2 mm were produced on a multi-tip press machine. Tableting excipients were carefully selected, and various process-related parameters were optimized in order to obtain the required dissolution behavior and convenient mechanical properties. Mini-tablets with predefined release profiles were filled into a capsule, the overall release profiles were determined by an appropriate dissolution method with HPLC analysis.

Membrane-assisted antisolvent crystallization: which factors control the crystal properties?

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Keywords | Membrane-assisted antisolvent crystallization; crystal size distribution; supersaturation; amino acids

Membrane-assisted antisolvent crystallization (MAAC) is a crystallization technique in which the transport of antisolvent is controlled through a membrane either in the liquid or vapour form. As the antisolvent reaches the crystallizing solution, it changes its composition, so solubility decreases and the solute crystallizes; thereby, MAAC can be used as an efficient technique to control crystallization kinetics (Curcio et al., 2020). This work investigates the potential of MAAC, over conventional antisolvent crystallization, using flat-sheet porous hydrophobic membranes — Polyvinylidene fluoride and polypropylene.

Amino acid crystallization in water, with the controlled addition of the antisolvent ethanol, was chosen as the model system for the study of MAAC. Amino acids are small molecules that form the elementary basis of peptides and proteins crucial for life; on the other hand, the amino acid-water-ethanol system allows us to evaluate the sole impact of the membrane process itself in controlling the increase of supersaturation.

The resulting Crystal Size Distribution (CSD), crystal morphology, and crystalline form were evaluated for various flow conditions, correlating with the transmembrane flux of the antisolvent and its mixing on both sides of the membrane, affecting supersaturation directly. It was observed that with the increase of the crystallizing solution flow rate, the transmembrane antisolvent flux decreased. This resulted in a higher concentration of the dissolved solute in the feed solution, hence higher supersaturation, eventually a narrower CSD. The resulting crystal properties differed according to the difference in antisolvent-solute-membrane interactions. The physicochemical characteristics of the membranes, such as porosity and surface energy, influence the kinetics of crystallization significantly (Chergaoui et al., 2022).

These results show the potential of using MAAC to intensify crystallization processes for various applications including polymorph selection, variation of crystal morphology, or narrower crystal size distribution. MAAC is particularly advantageous over conventional crystallizers in potentially purifying challenging reactions or developing thermally sensitive nanoparticles (metal-organic frameworks, catalysts, active pharmaceutical ingredients ...).

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Investigating the continuous flow seeded growth of metallodielectric patchy particles with in-line optical spectroscopy

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Keywords | Patchy Particles Seeded Growth Continuous Flow Colloidal Synthesis Electroless Plating In-line Characterization

Patchy particles are particles that feature clearly distinct regions with different chemical or functional properties on their surface. This arrangement makes patchy particles very interesting for applications in a wide range of fields. In the case of particles with noble metal patches having a localized surface plasmon resonances (LSPR) these include, pigments, sensing and theranostics.

We produce silver patches on colloidal silica nanoparticles using two decoupled T-mixer based continuous flow processes for seeding and growth. In the first process, the core particles are decorated with a very small number of ultrafine gold nanoparticles using added salt to induce coagulation between the same-charged species. This results in stable and long- term storable seed stocks. The seeded particles then enable the production of plasmonic silver patches with highly tunable optical properties via an electroless plating process. We found that the peak position of the main LSPR scales linearly with a dimensionless number that describes the ratio of concentrations of reaction components. As we will show, this ability to place the LSPR peak at arbitrary locations in the visible or near-Infrared spectrum results from tuning of the patch morphology, in particular the degree of coverage.

Another possibility the decoupled seeding offers is the optical analysis of the patch growth kinetics independent of nucleation effects. We designed a custom continuous flow reactor in which spectroscopic measurements can be performed at arbitrarily defined and corresponding residence times. The current design allows volume flows of several 100s ml/min and allows us, depending on the process conditions, to acquire data on the evolving product from only a few 100s of milliseconds after the T-mixer up to residence times where patch growth is complete (after several seconds). Using the measured spectra, aspects of the patch growth mechanism and kinetics can be inferred from changes in the height and position of LSPR peaks. Such in-line measurements for patchy particles produced under a systematic range of reaction conditions revealed several interesting and unexpected effects during their growth. Understanding these, with the help of advanced characterisation and electrodynamic simulations will contribute to the establishment of a process-structure model for this system.

Application of detailed modelling in the good, bad and ugly world of particles in pharmaceutical processing

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Keywords | Pharmaceutical engineering; Particle engineering; Modeling and simulation

Particles and their characteristics are among the most discussed topics in pharmaceutical processing during drug development and manufacturing. Many unit operations are dedicated to creating good particles, tuning the bad ones to behave well, and eliminating the ugly ones. Traditionally, this has been a playground of formulation scientists trying to find the best compromise among the available formulations, unit operations, and combinations using a trial-and-error approach. While regulatory expectations to transit from quality-by-testing to a quality-by-design approach are forcing the industry to look beyond the fence, the mathematical modelling of unit operations prominently applied in the chemical industry is finding its new niche in the pharmaceutical industry. Pharmaceutical engineers are exploring both existing and new mechanistic modelling frameworks to investigate and solve challenges associated with creating good, tuning bad and eliminating ugly particles in otherwise opaque systems requiring a lot of trial-and-error experiments. In this presentation, with case studies from some key pharmaceutical operations such as spray drying, fluidized-bed system, and freeze-drying, it will be shown that mechanistic modelling and simulation can be a valuable approach to look at the previously mentioned three dimensions of particle handling in pharmaceutical processing. The advantage and challenges of using detailed modeling in pharmaceutical R&D will also be discussed to explain why mathematical models are still not routinely used in handling pharmaceutical particles. Across these challenges, modelling and simulation hold tremendous opportunities for particle processes in the pharmaceutical industry.

Protection of ZnO nanoparticles against acid corrosion by TiO2 coatings

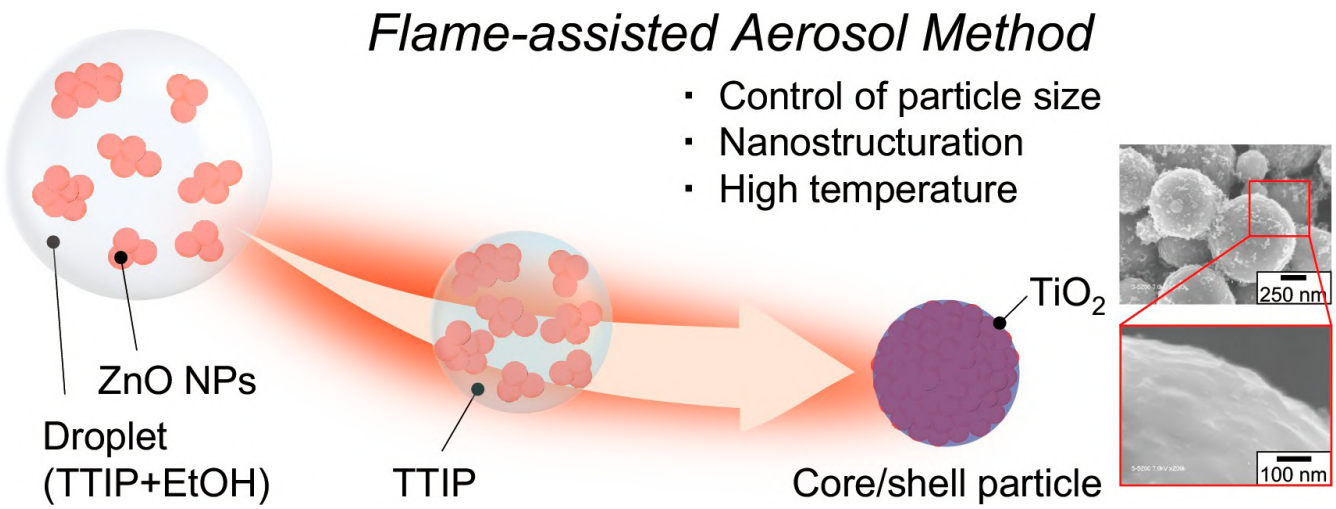
Hirano, Tomoyuki (1); Kaseda, Shogo (1); Le Anh Cao, Kiet (1); Ogi, Takashi (1)
(1) Hiroshima University

Keywords | acid resistance, zinc oxide, titanium oxide, UV protection, aerosol

The increase in ultraviolet (UV) radiation caused by the depletion of the ozone layer in recent years has had a negative impact on living organisms and various materials. Under these situations, the development of UV-protective materials is being actively pursued to protect

human skin from UV radiation. UV scatterers such as zinc oxide (ZnO) are used in cosmetics that are applied directly to human skin. ZnO has the excellent ability as a UV-protectant, but it has low durability against acids and degrades on the surface of human skin, which is weakly acidic.

In this study, ZnO/TiO2 core/shell particles were synthesized via flame aerosol process to enhance the acid resistance properties of ZnO nanoparticles. ZnO nanoparticles dispersed in titanium tetraisopropoxide (TTIP) ethanol solutions were used as starting materials. Precursor liquids were transformed into droplets using an ultrasonic nebulizer and then carried into a flame reactor by a carrier gas stream. Spherical ZnO/TiO2 composite particles with TiO2 weight percentages ranging from 0 to 30% were successfully prepared by a flame-assisted aerosol method. The synthesized ZnO/TiO2 had a multicore/shell structure with multiple ZnO cores inside the TiO2 shell. The acid resistance evaluation test by dropping sulfuric acid confirmed that the dissolution of Zn2+ was suppressed by core/shell structuration with TiO2, and that the acid resistance was improved. The multicore/shell structure is expected to have higher acid resistance performance because acid-resistant compounds exist in the gaps between the multicore particles, preventing acid from penetrating the inside of the particles.



FLASH COMMUNICATIONS

Production of high-temperature polymer microparticles via liquid-liquid phase separation and precipitation

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(1) Friedrich-Alexander-Universität Erlangen-Nürnberg, (2) EOS GmbH Electro Optical Systems

Keywords | polyetherimide (PEI), precipitation, solution-dissoltion, particle analysis

The development of new polymer materials is of great relevance in the context of powder-based additive manufacturing. Commercial available materials are mainly polyamides (PA12, PA11 and PA6), thus few other materials like polypropylene (PP) and polyether ether ketone (PEEK) are available. As the mechanical and thermal characteristics of the final product are strongly depending on the used feed material, the request for tough and durable materials is present. High-temperature thermoplastics, like PEEK, polyethersulfone (PES) and polyimide (PI), show the preferred tough material characteristics, however, suitable feed material is scarce.

This study focuses on the development of a novel powder material for the usage in powder-based additive manufacturing, whereby the feed material is polyetherimide (PEI). The production is performed using the liquid-liquid phase separation and precipitation approach, also known as LLPS. The latter has been proven as a feasible approach for the production of polymeric powder materials. The obtained particles show a proper characterization regarding their shape and morphology; hence, the product requirements for the processability in the aspired process are fulfilled.

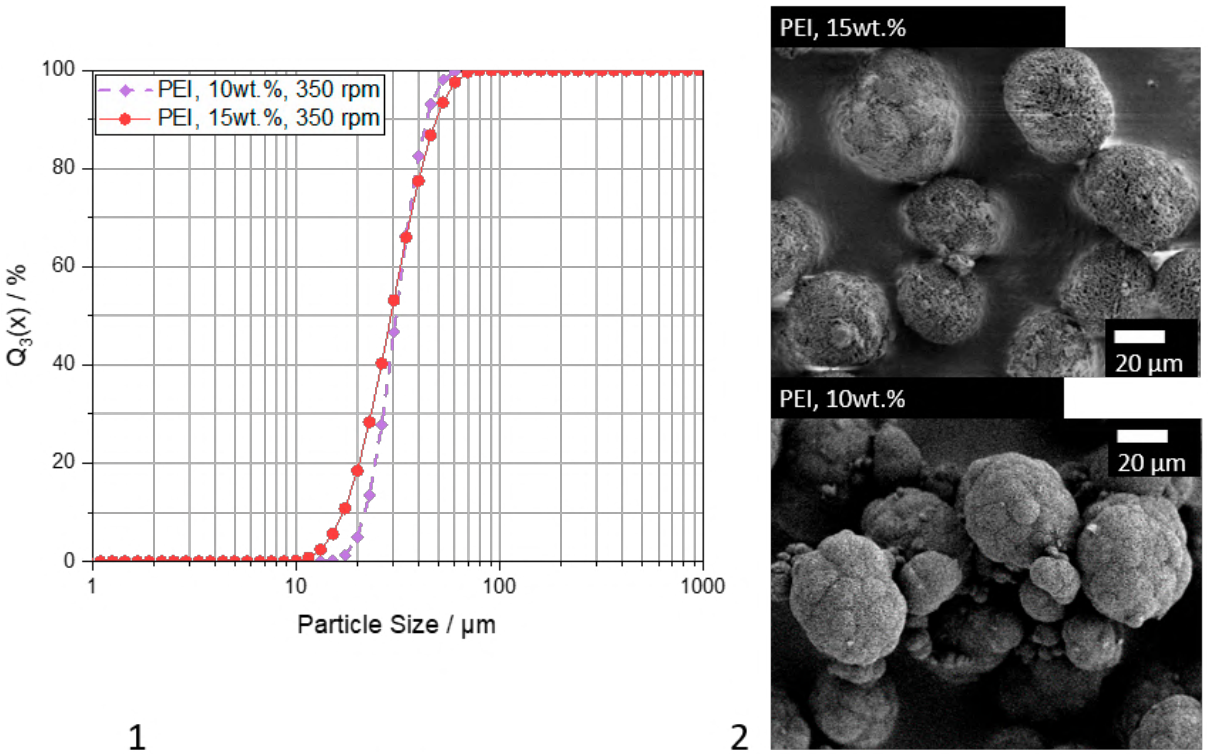


Figure 1: (1) Volumetric particle size distribution in dependence to the polymer concentration; (2) Scanning electron microscopy images of the particles

Seeded crystallization in a multiphase continuous microcrystallizer

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(1) KU LEUVEN

Keywords | Crystallization, microfluidic, continuous, seeding

Problem statement

Seeds are often added prior to a crystallization process, to improve control over nucleation and growth. Seeding in continuous plug flow crystallizers is distinctly different from seeding in batch crystallizers. The major challenge is the continuous delivery of a suspension with seeds. Solid delivery is particularly difficult in tubular microreactors, which have a tendency to clog. Previous research by Fatemi et. al has shown that the addition of microbubbles in a microcrystallizer can reduce clogging and improve crystallization [1].

Objectives

The objective was to design a setup which could deliver a seed suspension to a continuous multiphase microcrystallizer, to characterize the effect of microbubbles in a seeded microcrystallizer.

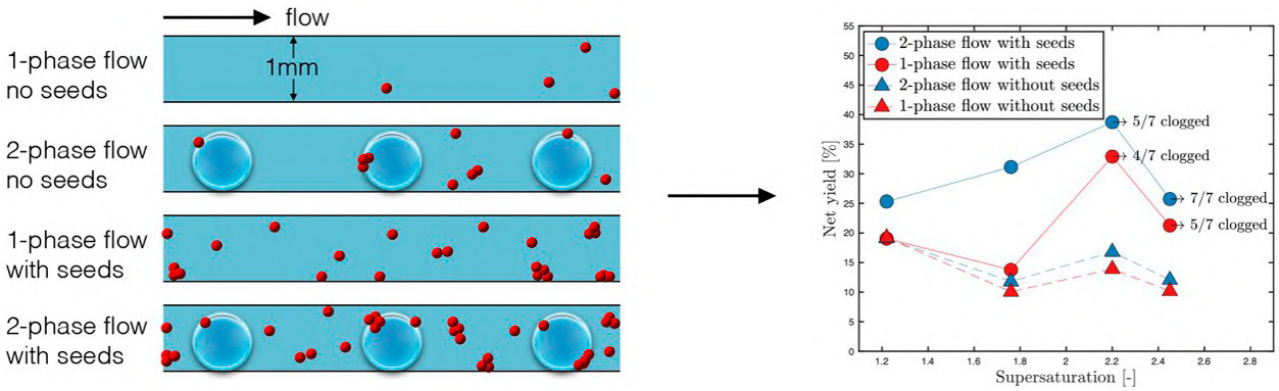


Figure 1 The left side gives a graphical overview. The right side shows the net yield for varying supersaturations.

Methods

A modular setup is built, in which a magnetically stirred and temperature-controlled syringe pump delivers the suspension. Ultrasound is used to keep seeds in suspension prior to entering the microcrystallizer and to control the temperature. The modular nature of the setup allows us to analyze both the size and the amount of crystal seeds entering and leaving the microcrystallizer. This is used to calculate the delivery efficiency and the mean net yield.

Most significant results

A delivery efficiency of approximately 50% was achieved, which can be improved by oscillating the flow. The addition of seeds causes a stastically significant increase in the net yield. The net crystal yield is larger for two-phase flow compared to single-phase flow. In addition, the seeds cause a significant decrease in the crystal size distribution. This decrease is due to the faster supersaturation consumption, as predicted by Eder et al. [2].

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Evaluation using machine learning in batch and continuous crystallizer processes for hydrocalumite from concentrated seawater

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Keywords | Concentrated seawater, Hydrocalumite, Machine learning, Crystallization, Deep neural network

A number of Ca resources are dissolved in a concentrated seawater discharged from utilization processes of seawater resources. In order to reduce environmental burden and utilize the Ca as a resource, a development of novel recovery methods for Ca is demanded. As a recovery method, a reactive crystallization of hydrocalumite (HC) was focused on. HC particles are utilized in various industrial fields because of the anion exchange ability. Clarification of the correlation between the experimental conditions and the powder properties of HC is important because controlling the powder properties of HC leads to manifestation of desired effects in each purpose. In this study, the optimization of the HC crystallization process using deep neural network (DNN) was attempted.

In the experiment, a raw material solution and NaOH solution were continuously supplied into a crystallizer, and HC was carried out at various experimental conditions. The raw material solution composed of CaCl2 and AlCl3 were prepared to be the Ca concentration of 0.10–1.0 mol/L. The Al/Ca ratio in the raw material solution was set at 0.50. The concentration of NaOH solution was 1.0 mol/L. The rotation speed of the crystallizer was varied from 500 to 4,000 min-1. The solution pH was maintained at 11.5. In order to optimize the crystallization

process of HC, the experimental data were utilized for the construction of DNN model predicting the powder properties of HC. As the powder properties predicted by DNN model, particle size (*D*) and Al ratio (*x*) in HC particle were selected. When DNN model was constructed, activation function, the number of hidden layers (*N*H.L.), neurons (*N*Neur.), and learning times (*N*L.T.) were varied. The accuracy and the performance of the constructed models were evaluated by determination coefficient (*R*2) and predictive coefficient (*Q*2).

As the result of the experiment, HC particles were obtained at all experimental condition in this study. Further, when DNN and LSM models were constructed, DNN model showed the higher *R*2 and *Q*2 values than that of LSM model in both *D* and *x*.

Potential and limitations of fluidized bed crystallization for continuous enantioseparation

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Keywords | separation of enantiomers, fluidized bed, continuous, integrated product classification

Enantiomers are optical isomers, which possess identical physical and chemical properties but behave differently in chiral environments, i.e. living organisms. Consequently, their separation is a challenging separation task for the life science industries. An efficient and cost-effective solution can be Preferential Crystallization. Applying this separation technique in fluidized beds leads to a robust process for continuous enantioseparation [Binev et al., 2016].

The enantioselective fluidized bed crystallization exploits conical-shaped tubular crystallizers, which are continuously fed with supersaturated mother liquor from the bottom. The mother liquor consists of both enantiomers at 50:50 (racemic) composition and the solvent. By continuously seeding with crystals of the desired enantiomer, only this is selectively crystallized and thus, separated from the unwanted counter-enantiomer. The conical shape of the tubular crystallizers causes a varying fluid velocity along their heights, which provides a size classifying effect and hence, an adjustable and narrow product crystal size distribution.

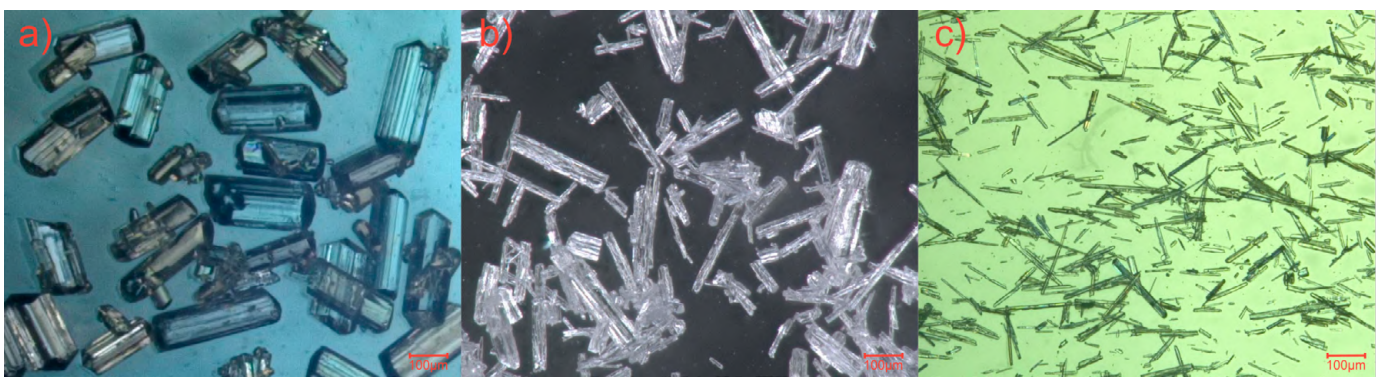


Fig.1: Microscopic images of the three compounds studied, DL-asparagine-monohydrate (a), DL-threonine (b) and (RS)- guaifenesin (c).

The continuous enantioseparation, with an outstanding productivity of up to 100 g/L/h, and the adjustable narrow product crystal size distribution were recently demonstrated for the amino acid DL-asparagine-monohydrate [Temmel et al., 2020, Gänsch et al., 2021]. Aims of our current work are to prove the process applicability for more challenging separation tasks and to explore its limitations. Therefore, we studied the process applicability for the amino acid DL-threonine and the API (RS)-guaifenesin. In contrast to the above-mentioned DL-asparagine-monohydrate, they possess particularly slow crystal growth rates and needle-like crystals (Fig.1) with significant lower solid densities, i.e. properties which deteriorate Preferential Crystallization, size classification and overall process performance. In the contribution, the process and its applicability will be discussed and evaluated considering product purity, productivity and the analyzed product crystal size distributions.

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Nanoencapsulation of 5-O-caffeoylquinic acid by Spray Drying

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(1) Laboratoire de Génie Chimique, Université de Toulouse, (2) Laboratoire de Génie Chimique, Université de Toulouse, CNRS

Keywords | Nanoencapsulation, Spray Drying, Chlorogenic acid, Nutraceutics, Pharmaceutics, Health

Nanoencapsulation of 5-O-caffeoylquinic acid by Spray Drying
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Nano spray-drying may be used for the encapsulation of diverse active ingredients (AI) and allows obtaining ultrafine powders with high recovery yield (Arpagaus et al., 2018). The wall materials in which the AI is confined protects it from the surrounding environment as well as enables to control its release (Fang et al., 2010).

In the present work, maltodextrin and arabic gum are being used to coat a phenolic compound from the chlorogenic acids (CGAs) group: the 5-O-caffeoylquinic acid (5-CQA). Several studies show that 5-CQA has potential benefits in treatments of oxidative and inflammatory stress, neurodegenerative and cardiovascular diseases, gastrointestinal dysfunctions and cancer, among others (Lu et al., 2020).

To study the nanoencapsulation process, a laboratory scale Nano spray-dryer is used. In this work, different operating parameters were modified to study their impact on the properties of the nanoparticles obtained. On one hand, there are parameters of the feed formulation like concentration, wall materials and active molecule ratio, surfactants type, etc. On the other hand, it is important to control the process parameters including spray mesh size, inlet temperature, gas flow rate, liquid feed rate, etc. Chemical and physical properties of raw and spray-dried nanoparticles were analyzed by using complementary techniques: size and morphology by SEM, composition and encapsulation efficiency by TGA and HPLC, antioxidant activity and storage stability among others.

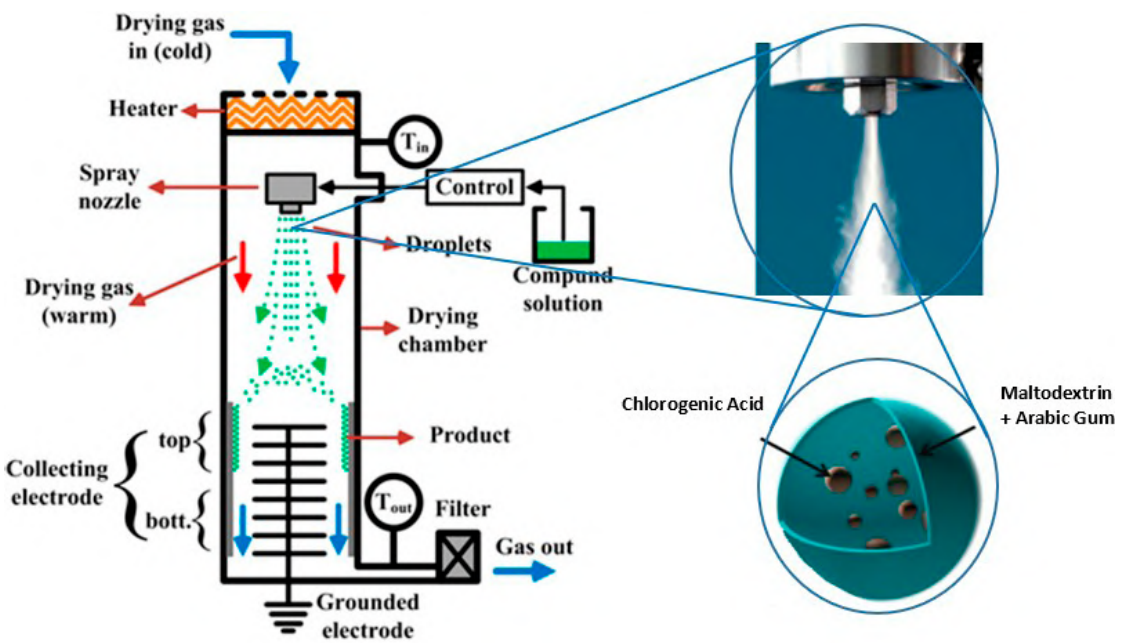


Figure 1: Schematic Nano Spray-Dryer set-up

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Detergents additives encapsulation using silica nanoparticles with a metal coating

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(1) Universidad de Granada, (2) Universidad de Granadfa

Keywords | Encapsulation Detergents Emulsion Biocide

An easy method for silica beads preparation by agglomeration of colloidal silica nanoparticles adding a high concentration salt to produce the precipitation after the emulsion is prepared. Due to versatility of this method, these beads could be used in many applications (catalysis, detergents, coatings, ...).

Emulsion is prepared employing sunflower oil as continuous phase where silica particles solution (Ludox – 30 %wt silica) are dispersed using a high-power stirrer. Then, 1 M CaCl2 is added to produce the silica beads. Finally, after centrifugation, the particles are redispersed in deionized water.

Characterization of particles is done using a Zetasizer and a Scanning Electron Microscopy.

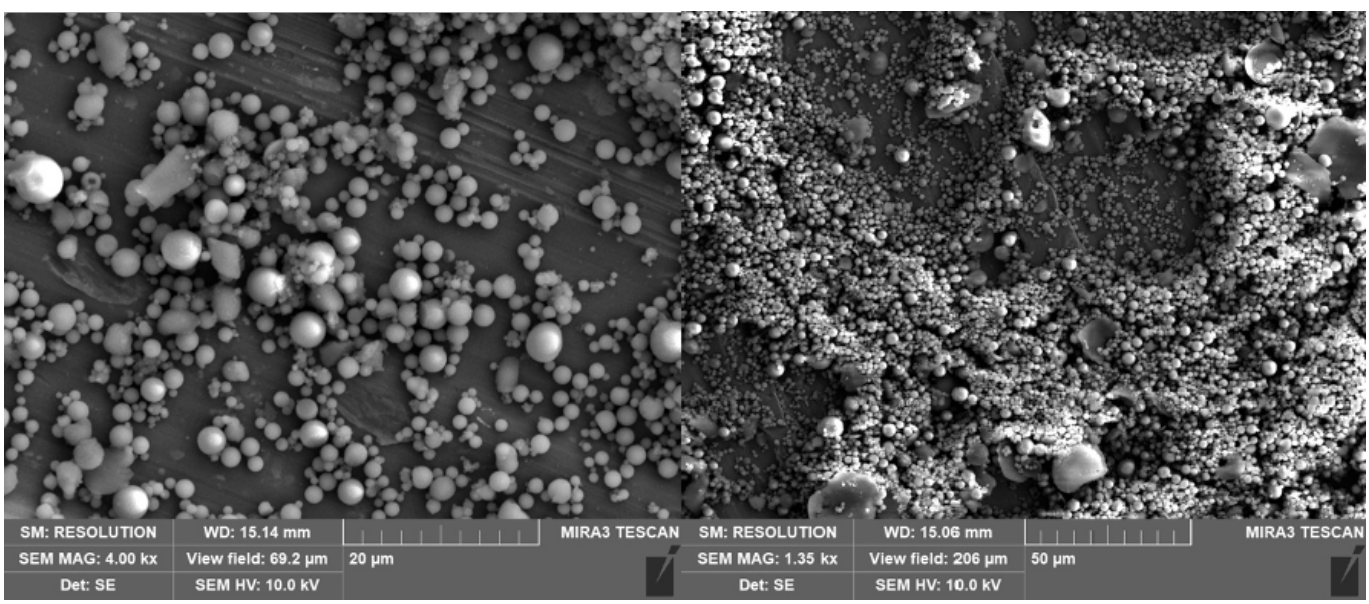


FIGURE 1 – Formed silica beads using exposed method

To test the viability of these beads as carriers, release tests are performed, using an ultrafiltration membrane, with dye and bronopol (biocide), in both, a very similar behavior is observed. From these, it can be concluded that the silica beads are capable of transporting the dye, but not of retaining it.

Therefore, an extra layer that provides impermeability to particles is necessary. For this purpose, the coating will be produced by in situ formation of the metal layer from the reduction of its salt. The metal selected for this is copper, due to its low cost (very important for detergents application), biocidal capacity and non-toxicity.

The coating formation reaction is carried out at a pH equal to 6, where it has been seen that the silica and copper particles have zeta potentials of different signs and would facilitate the formation of the new layer.

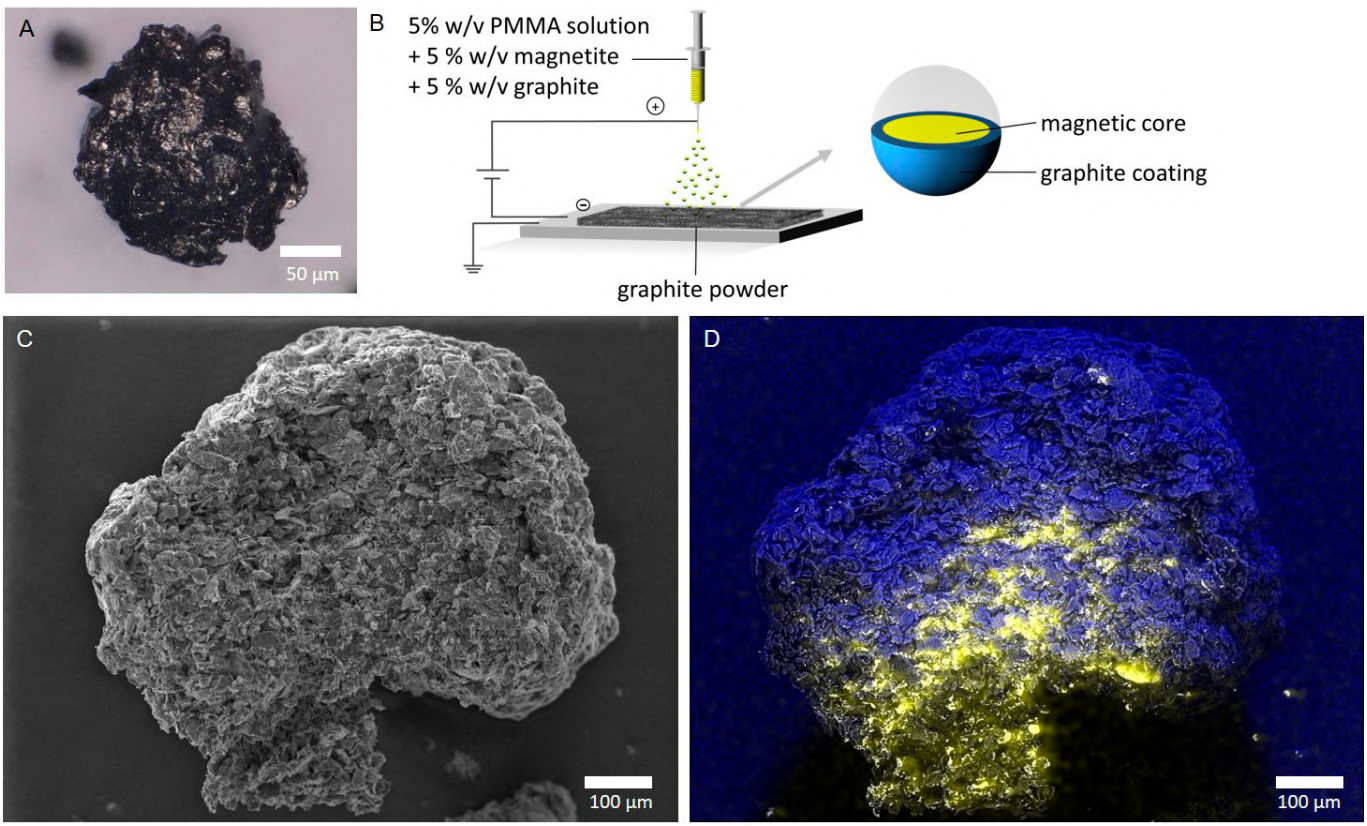
Thus, when the biocidal compound is encapsulated, the function of which would be increased due to the biocidal nature of the copper that forms the coating. In addition, the core biocide could be changed for another interesting additive in detergents (enzymes, perfumes, bleaches, ...), achieving capsules with a double effect.

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Multifunctional Core-Shell Particles Obtained by in Situ Coating of Electrosprayed Polymer Particles

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(1) Karlsruhe Institute of Technology, (2) University of Michigan

Keywords | Core-shell particle, particle electrode, electrospraying, coating, magnetic particle



Particle electrodes are well-suited for a wide range of electrochemical reactions due to their large specific surface area. In addition, the magentic stabilization of a particle electrode enhances the particle-particle contact and therefore improves the electrochemical conversion rates (Tschoepe et al., 2020). However, the lack of suitable electrode materials currently undermines the design of particle electrodes with high conversion rates.

In this work, we describe the design and preparation of novel multifunctional core-shell particles suitable as electrode materials. Core-shell particles are obtained by electrospraying of a magnetite / poly(methyl methacrylate) suspension into a powder bed. A range of different powder beds such as graphite, silver, titanium dioxide or active carbon has been evaluated. Energy-dispersive X-ray spectroscopy maps and Raman spectroscopy of particles confirm the core-shell structure. Core-shell particles were further tested as an electrode material in a magnetically stabilized fluidized bed reactor.

Using these multifunctional core-shell particles, we achieved conversion rates for the reduction of potassium ferricyanide that were three times higher than literature-known reference electrodes (Schneider et al., 2020). These results support the high potential of core-shell particles for a wide range of electrochemical fluidized bed reactors.

Schneider, M., Tschöpe, A., Hanselmann, D., Ballweg, T., Gellermann, C., Franzreb, M., and Mandel, K., 2020. Adsorber particles with magnetically-supported improved electrochemical conversion behavior for waste water treatment processes.

Particle & Particle Systems Characterization, 37(2), 1900487.

Tschoepe, A., Wyrwoll, M., Schneider, M.,Mandel, K., and Franzreb, M., 2020. A magnetically induced fluidized-bed reactor for intensification of electrochemical reactions, Chem. Eng. J., vol. 385, 123845.

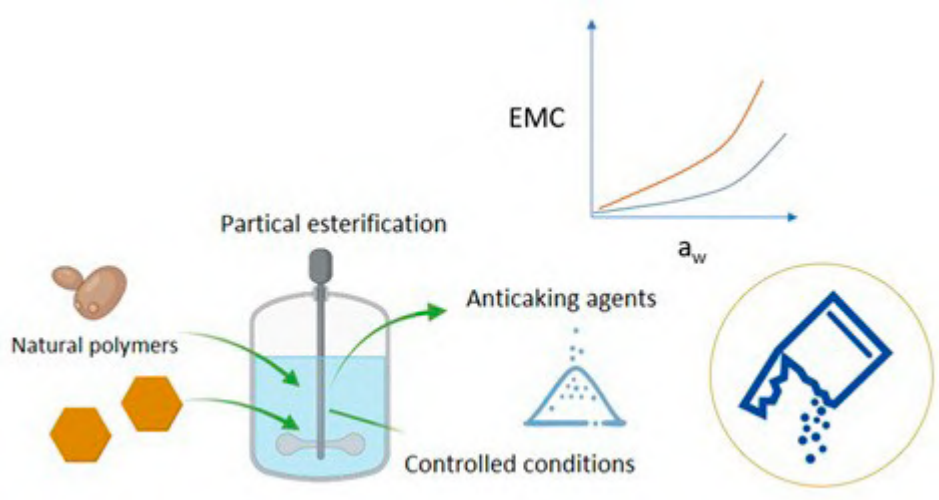
Anticaking agents with superabsorbent properties

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(1) University of Greenwich

Keywords | Anticaking agents; flowability

The widespread phenomenon of undesired powder agglomeration called caking in the chemical and process industries is usually arising from dissolution followed by recrystallization of the hygroscopic solid substance present. This powder transformation, either due to the aggregation of a free-flowing powder into a coherent mass that resists flow and deformation, or deliquescence of a highly hygroscopic material, is a common problem for many industries. There are several mechanisms, which will be reviewed in this presentation, that may lead to powder caking, but it is uncommon for studies to take a comprehensive holistic view. Powdered ingredients may be sensitive to environmental and operating conditions and their propensity to cake in the supply chain is currently unpredictable. A greater knowledge of powder hygroscopic and caking behaviour will lead to a predictive capability that will enable both optimally performing products and reduce costs.

This presentation will ultimately review the experimental methods for preparation of anticaking agents prepared from natural polymers which are then transformed into super absorbent polymers that do not contain silicon, are not in the nanometer size (but millimetre range) and are “safe”.



6 PARTICLE SEPARATION

KEYNOTE LECTURES

Fundamental analysis of particle-based pore systems in filtration using X-ray microscopy

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Keywords | cake filtration, capillary pressure, porosity, tortuosity, de-saturation

The structure of a porous network built from particles determines the process of cake filtration. It is significantly influenced by the particle properties leading to local and integral property variations. Current models and experimental concepts describing the relationship between particle properties and the porous structure of the filter cake are based only on single, integral parameters for both the particle and the pore system, and thus describe local variations and distributions inadequately. To gain insight in locally distributed structural parameters such as porosity (Löwer et al., 2020a), coordination number (Löwer et al., 2021a), three-phase contact angle (Löwer et al., 2021b), nature of pores and isolated liquid regions (Esser et al., 2021), liquid loading of individual particles, tortuosity, and capillary length (Löwer et al., 2020b) (cf. Figure 1) it becomes necessary to resolve these structures to the particle level. Here, X-ray microscopy (XRM) provides direct insights into the structure of the filter cake generates a comprehensive understanding of the micro-processes taking place during the formation and de-saturation of the pore system.

For the evaluation of the tomograms characterizing the pore space, we apply own models and coding in addition to routines established in commercial software. Geometric model developments offer the tortuosity distribution in flow direction as well as an associated capillary size distribution. The latter derives from different model approaches, i.e. inscribed sphere, pore neck or special geometrical models using the 3D-structure. The capillary size distribution itself can be used to determine the capillary pressure distribution, which is the starting point for process modelling, since it describes the equilibrium condition of liquid saturation in the filter cake as a function of the applied pressure difference. The variation of the particle size distribution, e.g. bimodal properties, shows its effect on further cake properties like neighborhood relations between the particles. De-saturated filter cakes, or cakes formed with a suspo-emulsion generate a three-phase problem. The tomographic analysis is able to quantify the size, shape and location of liquid and oil bridges in the pore space, which allows new concepts for mechanistic modelling.

Acoustic streaming and particle fractionation in acoustic fields

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Keywords | Aerosol classification, Finest particles, Acoustic streaming, Particle separation in gases

Acoustic streaming and particle fractionation in acoustic fields
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The growing demand for fine particle systems with narrow specifications challenge the operating mechanisms of established separation processes. In the particle size range between 0.5 and 10 µm conventional processes tend to have a lag in efficiency and selectivity.

In this contribution, a separation and fractionation process is introduced, which is capable of addressing and separating particles of 0.5 to 10 µm with high selectivity. The process is based on acoustic resonant waves which influence and separate the gas born particles according to their inertia and material properties as shown in figure 1.

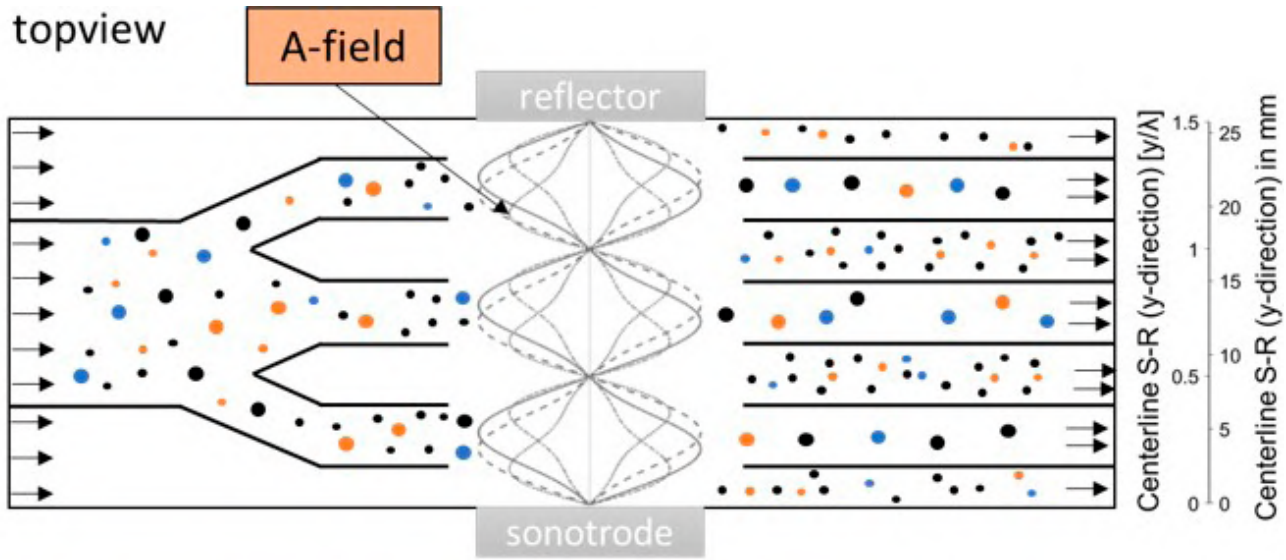


Figure 1: Process scheme of ultrasonic classification of fine particles

Mathematical modelling can illustrate, how the particle trajectory can be influenced in an resonant wave field in such a way that an inertia-dependent separation of the particles in the acoustic field takes place.

However, in experiments it has been found that the larger particles can be found at the expected locations while the small particles are influenced by side effects as acoustic streaming that inhibit proper separation.

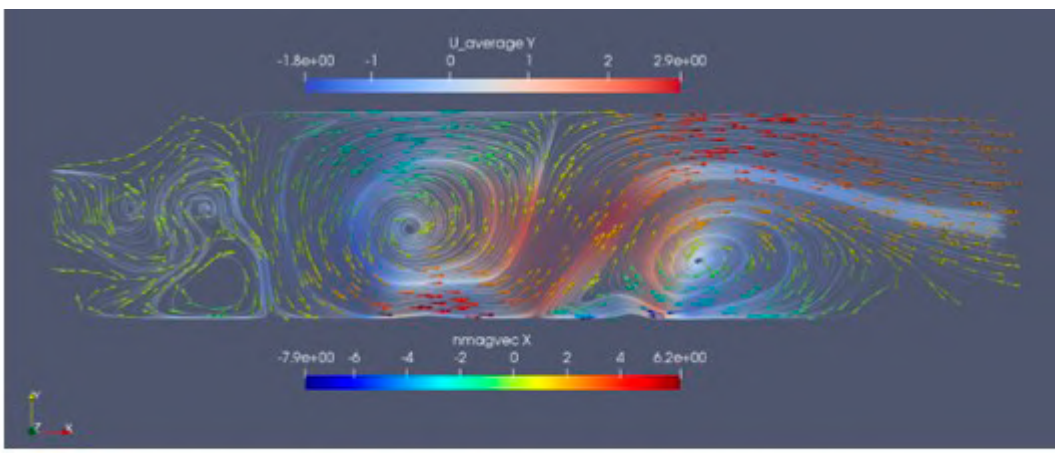


Figure 2: Time averaged flow field with intense acoustic streaming

Therefore the gas flow behaviour in the resonant acoustic field is investigated using CFD. In these studies pronounced acoustic streaming is found, an effect were non-linear wave propagation behaviour lead to a time independent flow from the radiating surface to the reflecting surface. This leads to large vortex formation and back mixing. To reduce this effect countermeasures are investigated and by this the acoustic streaming intensity can be reduced by up to 80 % compared to the untreated system.

(Sandmann et al., 2020) Sandmann, K. and Fritsching, U. (2020), Selective Particle Classification in Ultrasonically Excited Aerosols. Chemie Ingenieur Technik.

Applying circular economy towards turbidity reduction of sugarcane juice

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Keywords | Circular economy; natural polyelectrolytes; flocculation; sugar juice treatment; turbidity

Brazilian mills are able to produce crystal sugar, ethanol and energy in large scale from sugarcane plants. After extraction of juice which is rich in sugars like sucrose, glucose and fructose, the juice should be treated to remove most of the impurities extracted together with the sugars. These impurities reduce the quality of the final products or the efficiency of the processes. In large scale, mills employ high amounts of synthetic polymers as promoters of sedimentation, but some studies have shown that its use could be associated to human health problems by degradation into acrylamide. The aim of this study was to develop cellulose-based polyelectrolytes using as raw material cane bagasse which is a waste resulting from sugar production. The cane bagasse was treated and anionic cellulose based polyelectrolytes (ADAC) with different charges were produced, based on previous experience of modifying and charging cellulose chains. We simulated the industrial process of sugar treatment at laboratory-scale, replacing the synthetic polymer by cellulose-based polymers (Figure 1a). The ADAC obtained from cane bagasse was compared with the one obtained from wastes of acacia wood. Additionally, the treatment with the cellulose based polymers was compared with conventional treatment using commercial synthetic polymer. In general, the synthetic polymers reduce the turbidity of the sugarcane juice, one of most important parameter in sugarcane mill process control, around 98%, while the cellulose-based polymers present 99% of turbidity reduction, meaning that the impurities were removed from the cane juice in both cases. Figure 1b shows the reduction of turbidity using three different polymers of cellulose nature produced under different conditions. It is possible to observe the pronounced reduction of turbidity in all cases, including when the polymer obtained from cane bagasse is used. Turbidity removal was also followed using Laser Diffraction Spectroscopy (LDS).

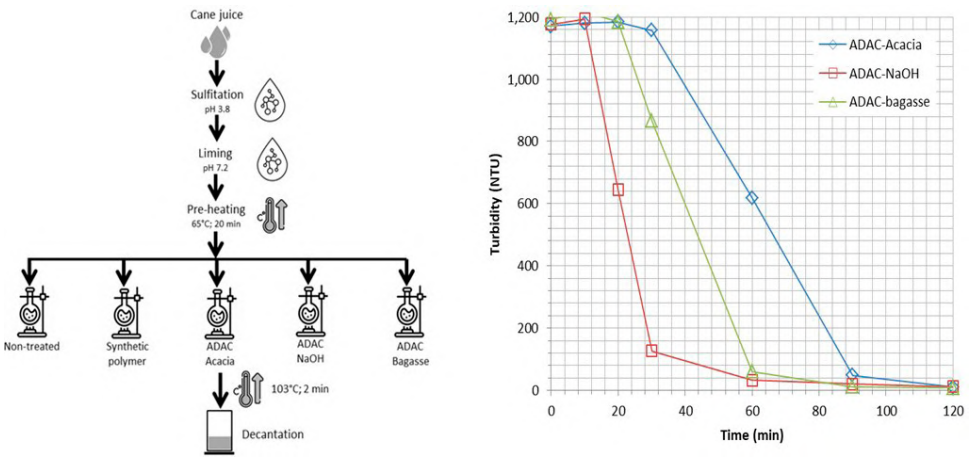


Figure 1. a: Protocol of sugarcane juice treatment. b: Reduction of turbidity in sugarcane juice treated with cellulose-based polyelectrolytes to promote sedimentation increase.

In conclusion, the results show that it is possible to replace the synthetic polymers in crystal sugar production by cellulose- based polyelectrolytes without loss of efficiency and reducing impact in human health.

ORAL COMMUNICATIONS

Aerosol particle penetration in a respirable cyclone sampler at different flow rates

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Keywords | Cyclone sampler, penetration, pressure drop, Large Eddy Simulation

Aerosol sampling is one of the largest and growing application areas of small-scale cyclone separators. Personal cyclone samplers have been widely used to measure the respirable mass of particles in occupational and ambient environments. In respirable dust sampling, it is essential that the cyclone cut-off characteristics be known and constant, and that each cyclone is operated at a flow rate that produces the desired cut-off. The objective of this study is to investigate the performance of a sampling cyclone at different flow rates and reveal the

dependence of main cyclone performance characteristics on the flow rate (Reynolds number). This was realised by the usage of Large Eddy Simulations (LES) that were validated based on the experimental data of the cyclone performance.

The particle-laden flow within the cyclone has been investigated with LES applying a dynamic Smagorinsky-Lilly subgrid- scale model. The air phase was treated using an Eulerian approach while the Lagrange method was applied to model the movement of particles, which were tracked through the cyclone.

The performance characteristics of a sampling cyclone, pressure drop and particle penetration, have been studied at a wide range of flow rate 0.22–7.54 LPM using the LES simulations that have been validated based on experimental measurements of the cyclone pressure drop and penetration data. The cyclone performance has been described by three dimensionless characteristics, the Euler number, the cut-size and the slope of the penetration curve. Three main flow regimes and four sub- regimes have been revealed. The effects of the flow rate (Reynolds number) on the dimensionless cyclone performance characteristics have been described and a one-term power series model has been proposed. Additionally, the effect of the aspiration efficiency on the cyclone cut-size has been determined.

Numerical modeling of a gas cyclone separator using a hybrid CFD-DEM-DDPM approach

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(1) Engineering Simulation and Scientific Software (ESSS)

Keywords | CFD, DEM, multi-GPU, hybrid CFD-DEM-DDPM

Gas cyclone separators are devices commonly found in industrial applications and home appliances. The dusty air that comes in undergoes a strong swirling motion that has the objective of separating particles from the main air stream. Cleaner air then flows through the overflow section while particles are collected at the underflow section.

Particles separate from the air stream due to their inertia. Those with high-enough inertia will move towards the cyclone walls and progressively find their way out due to gravity. Those not able to separate from the air stream end up carried upwards by the inner vortex. As those larger particles accumulate at the walls, they tend to increase their influence on the flow field. It is also in the wall adjacent regions that both particle-wall and particle-particle interactions take place. The cyclone core flow, on the other hand, is mostly dominated by smaller particles in a more dilute fashion, meaning that particle-particle interactions can be disregarded compared to the flow effect on particle motion. However, the lower end of the particle size distribution represents most of the particle count, increasing computational cost by a significant amount.

Since this kind of equipment usually operates with micron or sub-micron sized particles, even for low particle mass loadings, simulating a close-to-real scale problem would require handling several billion particles. When accounting for particle- particle and particle-boundary interactions, the DEM share can easily become the simulation bottleneck. In this study, we propose a hybrid CFD-DEM-DDPM approach. Particle-particle and particle-wall interactions are only taken into account for particles within the boundaries of a region-of-interest (ROI). For the remaining particles, only non contact forces such as gravity and fluid-interaction forces are considered. The simulations are carried out on a powerful commercial multi-GPU solver. The results are compared against reference literature data and the performance improvements are discussed.

Design of Novel small diameter gas cyclones for accurate particle classification

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(1) Imperial College London

Keywords | Gas Cyclone, Particle Classification, CFD, Experimental Design-CCRD, Response Surface Methodology(RSM)

Abstract:

Traditionally, gas cyclones are used as particle separators for the complete removal of dust from gas streams. These devices have very small cutsizes, generally less than 1 micron. In very few studies the desired operation mode of gas cyclones is particle classification rather than particle removal. These few large diameter cyclones studies have focused on design changes of the apex region[1-3].

Small-diameter gas cyclones can operate as particle classifiers, offering low flow rates and a broad range of cut sizes. In the current study, we have used Computational Fluid Dynamics (CFD) to evaluate the effect of the shape of the curved conical wall on particle classification in a 31mm diameter gas cyclone. The CFD model predictions of the standard small-scale gas cyclone were first validated with experimental data[4]. Subsequently, the curvature of the conical wall was varied from convex to concave. It was found that the sharpness and imperfection are improved for the novel designs proposed and that the cut size can be manipulated readily.

A robust experimental design – the Central Composite Rotatable Design (CCRD)[5] was used to develop an optimization model for cut size and sharpness, with both design and operational parameters as input variables.

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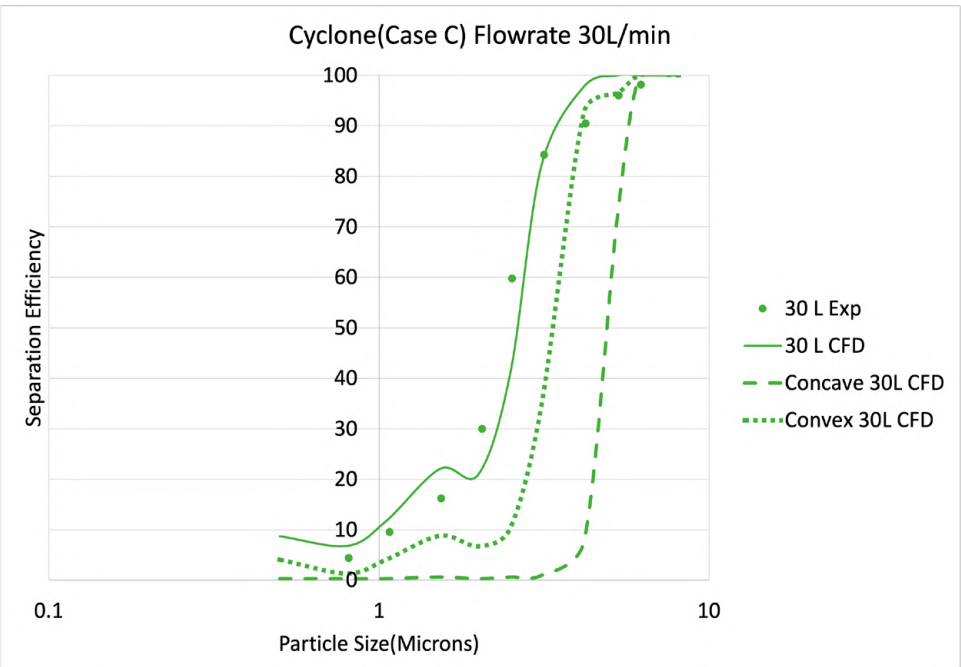
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Development and Numerical Simulation of an Efficient Cyclone Separator with a Recirculation System

Nedumaran, Praveen Kumar (1); Misiulia, Dzmitry (1); Antonyuk, Sergiy (1)

(1) Technical University of Kaiserslautern

Keywords | Gas Cyclone, Recirculation system, Numerical Simulation, PM2.5 exhaust gas flow, Eulerian-Lagrangian

DEVELOPMENT AND NUMERICAL SIMULATION OF AN EFFICIENT CYCLONE SEPARATOR WITH A RECIRCULATION SYSTEM
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ABSTRACT

Dedusting or the separation of a dispersed phase from a continuous phase is an important process in all the process engineering industries. Gas cyclones are the most preferred separators for exhaust dedusting in industrial plants. They are simple in design and have the ability to operate at high temperatures. These gas cyclones are characterized by their separation efficiency and pressure loss. For higher separation efficiencies, pressure losses should have to be compromised. Even with higher pressure loss, gas cyclones have relatively low separation efficiency for dedusting PM2.5 gases. Gas cyclones are commonly used as pre-separators with additional downstream exhaust cleaning systems, increasing the overall installation, operational and maintenance costs. Hence, the main objective of this project is to develop a gas cyclone that handles PM2.5 gas flow and separates the dust particles with a higher separation efficiency without the downstream cleaning systems.

An operational cyclone from an industrial partner has been taken for an initial study, and numerical simulations were conducted to identify the separation efficiency and the pressure. Simulation results have shown that particles with smaller aerodynamic diameters leave the inner vortex separation region and also escape with secondary flow through the lip of the vortex finder.

A recirculation system has been introduced to redirect the unseparated dust particles leaving the cyclone back into the inlet of the cyclone. This recirculation system creates a static pressure difference between the wall of the vortex finder and the inlet pipe, which helps the particles closest to the vortex wall re-enter the cyclone. A Numerical Investigation with PM2.5 gas flow has been conducted using the Reynolds Stress Model with the Eulerian-Lagrangian approach, which tracks the particles through the mainstream gas. The simulation reveals that the particles re-entering the inlet through the recirculation system agglomerate and get separated, increasing the overall separation efficiency of the cyclone. Additionally, a new cut-size and pressure drop have been identified.

Analytical and numerical model for the description of the separation and pressure drop behavior of small-scale unidirectional cyclone separators

Dwars, Gunnar (1); Mehring, Carsten (1)
(1) Institute Of Mechanical Process Engineering (University of Stuttgart)

Keywords | Cyclone, Separator, Small-Scale Separator, Discrete Particle Method, Random-Walk Modell

This paper presents an analytical model for the design of miniaturized unidirectional air cyclones serving as gas-liquid separators for the removal of oil aerosols from a gas stream. Conservation equations for mass, momentum and energy form the basis for the pressure loss prediction within the model; a modified residence-time model is employed for describing the separation behavior of oil droplets on the outer cyclone wall. Results from a CFD analysis of the investigated flow geometry are also presented, providing details about the flowfield, pressure loss across the cyclone, aerosol deposition and overall separation efficiency. The numerical model is based on the incompressible RANS equations using the k-ω-SST turbulence model and a Lagrangian description of the dispersed phase (Discrete Particle Method, DPM) with a random-walk model for turbulent dispersion. Numerical solutions have been obtained using the commercial FV (Finite-Volume) solver Ansys Fluent version 2022R1. Both (analytical and numerical) models are benchmarked against experimental data obtained for a large series of cyclone geometries, all having an outer wetted diameter of 20 mm. Separation efficiency measurements were carried out using a white light aerosol spectrometer by Palas GmbH. Good agreement between the experimental, numerical and analytical results is shown. Accordingly, the derived analytical model is suitable for rapid design of uniflow cyclones in the investigated configuration spectrum.

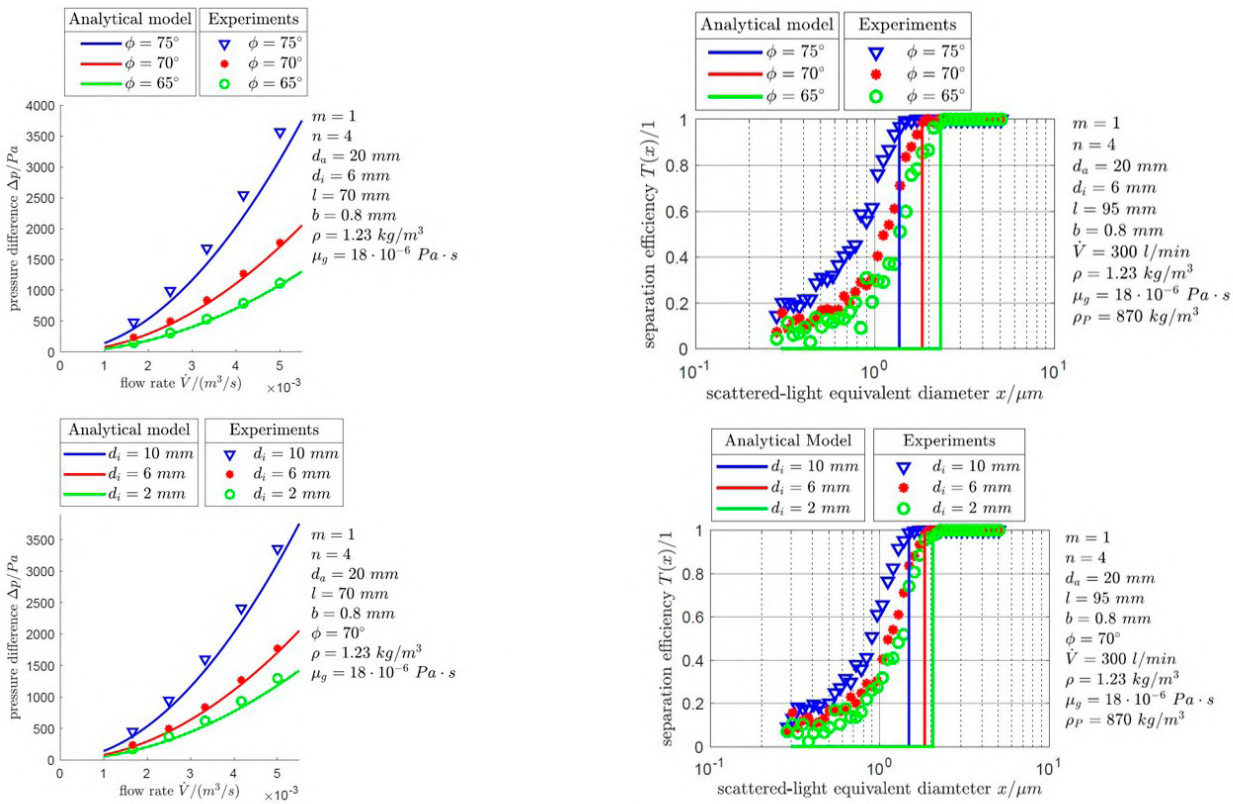


Fig. 1: Comparison between analytical and experimental pressure difference (left) and separation efficiency (right) of different cyclone geometries

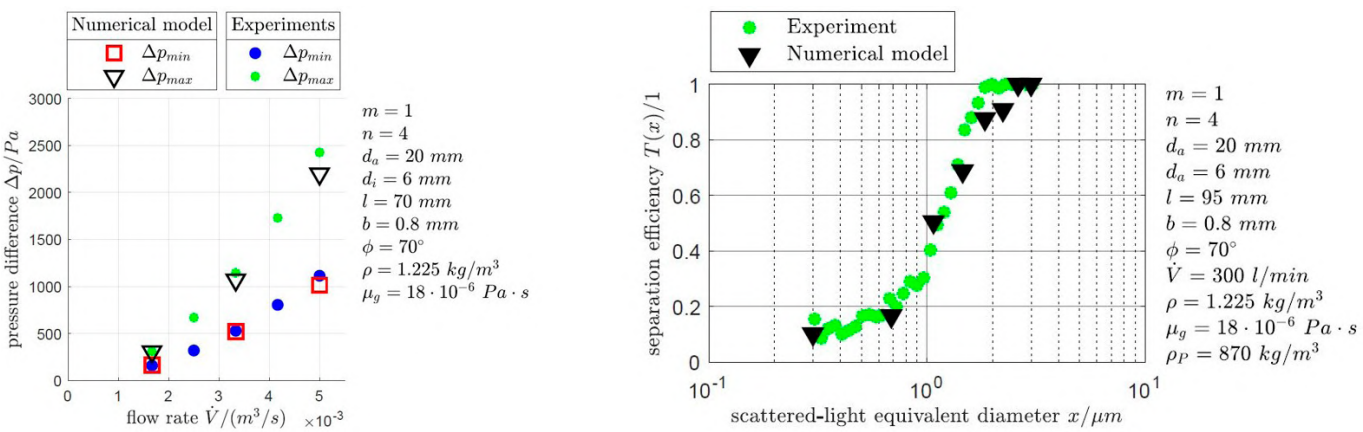


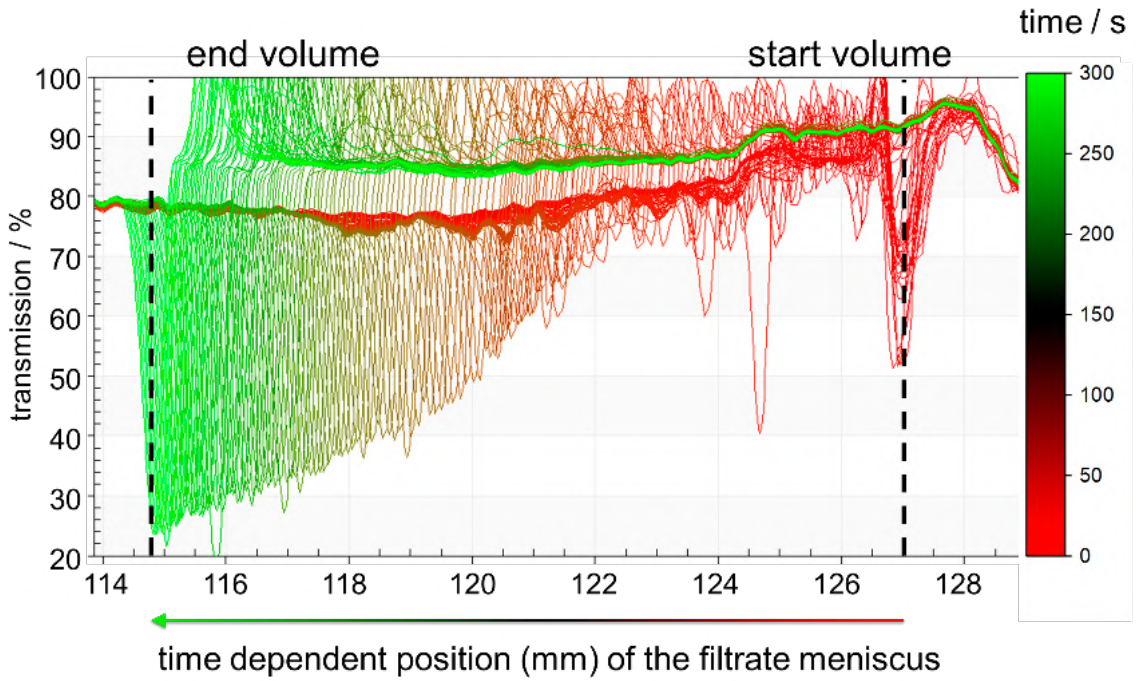
Fig. 2: Comparison between numerical and experimental pressure difference (left) and separation efficiency (right)

Small Scale Filtration Experiments by Analytical Multisample Photo-centrifugal Filtration (ACF)

Loesch, Philipp (1); Boldt, Sebastian (2); Krause, Daniel (2); Lerche, Dietmar (2); Antonyuk, Sergiy (1)
(1) Technische Universität Kaiserslautern, Institute of Particle Process Engineering, (2) LUM GmbH

Keywords | Cake Filtration, Membranes, characterization

The continuous development of new products in pharma and biotechnology leads to a need of required separation processes for further processing. Especially proteins or vaccines, which are obtained from cell suspensions. To achieve the best filterability for a special application, tailor-made membranes have to be developed. For the design of filtration processes, standardized laboratory tests are usually performed. To cover a wide range of processes, the standardized test requires a quite large amount of filter media and a large amount of the suspension to be filtered.



For cost reduction in both the use of valuable pharmaceutical products and newly developed membranes, it is advisable to reduce the required material input. A new possibility is to perform the filtration experiments by means of analytical photo- centrifugation filtration (ACF) which allows a simple and fast membrane characterization and filterability with high throughput [1, 2]. With ACF it is possible to analyze up to 12 different samples of about 1 mL in the centrifugal field at the same time. A special developed filtration cell (Fig. 1) holds the suspension that has to be filtered, the filter media and as well as the resulting filtrate. Furthermore, by adjusting the instrument parameter by applying different rotor speeds (influencing the applied pressure) at different time stages or temperature profiles during one experimental run makes the design very flexible. ACF by a multi-sample photo-centrifuge is based on continuous in-situ measurement of space resolved light transmission through a specially designed filtration module monitoring the volume increase of the filtrate at the bottom of the cell by STEP-Technology® [3]. In this contribution the clean membrane resistances and the filter cake resistance are determined. A comparison to classical measurements is shown. The challenge and opportunity of comparing a rotational filtration to a pressure driven filtration will be discussed.

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Frequency-modulated dielectrophoretic particle chromatography

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(1) University of Bremen

Keywords | dielectrophoresis (DEP); microparticles; polystyrene; chromatography; interdigitated electrodes; microfluidic; separation

Separating microparticles (< 10 µm) according to properties such as size, shape, or material is a field of recent research, since conventional separation techniques do not cover this size range. However, many separation tasks for biological applications or material recycling include particles below 10 µm. Therefore, novel separation techniques are required that are capable of addressing (multiple) properties of the particles. Dielectrophoresis is one candidate for these separations.

Dielectrophoresis is the movement of polarizable particles in inhomogeneous electric field. The dielectrophoretic force depends on several particle properties such as size, shape and material. Additionally, it is influenced by process parameters as the composition of the medium in which the targeted particles are suspended (water, air or oil) or the frequency and voltage of the applied electric field. Generally, particles will move towards field maxima (positive DEP) or away from them (negative DEP), depending on their relative polarizability.

In a recent publication we introduced frequency-modulated dielectrophoretic particle chromatography (DPC) (Giesler et al. 2020). Here, a particle suspension is injected into a carrier flow in a microfluidic device flowing over an electrode array, which is connected to a voltage source. The electrodes generate an inhomogeneous electric field. In the approach, we modulate the field frequency by a (triangular) function which leads to a periodic change of the frequency between two values. Since strength and direction (positive or negative DEP) of the dielectrophoretic motion are frequency dependent for a variety of microparticles, this leads to different particle trajectories inside the channel. Similar to the well-studied field-flow fractionation, this approach, in combination with the laminar carrier flow profile, leads to characteristic retention times for each particle type, thus allowing particle separation. In our lab we could achieve a separation of particles (size 2 to 6 µm) with respect to size and surface properties (Giesler et al. 2021).

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Dielectrophoretic filtration for selective separation of submicron particles at high throughput

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(1) University of Bremen

Keywords | Dielectrophoresis (DEP), electrokinetics, electrofiltration

Recovery of precious materials concentrated in fine dust is essential for the recycling industry e.g. electronic waste or batterie recycling. Despite the vast number of separation technologies, the handling of submicron particles remains a challenge.

Dielectrophoresis (DEP), the motion of polarizable matter in non-homogeneous electrical fields, can be utilized to effectively separate such micron and sub-micron particles by a variety of properties. DEP is highly selective regarding particle size, material, and morphology, without requiring particle labelling or charge. However, most conventional DEP applications are developed for biological or analytical separation tasks using microfluidic devices. In contrast, we develop high-throughput DEP-based separators for potential industrial applications. Here, we present dielectrophoretic filtration, an electrically switchable filtration process, which is based on the application of an electric field across an insulating filter (porous media). The filter provides numerous tortuous flow paths in which the applied electric field is scattered, causing many local field maxima. When pumping a suspension through the filter, target particles which are more polarizable than the surrounding fluid are pulled to the field maxima by DEP, where they are trapped, while non-target particles pass the filter.

We show how particle separation is influenced by key parameters such as applied voltage, throughput, and filter geometry and present design rules for DEP filtration (Lorenz 2020, Pesch 2018). We will also present recent results of selective separation of particles according to their electric conductivity. In this context, as model particles, we separated graphite dust from polystyrene (PS) beads and copper flakes from PS beads. Finally, we address the quantification of purity of DEP trapping of metal flakes mixed with PS particles of comparable size. These results demonstrate a notable step towards selective DEP filtration at high throughputs and further application of DEP for metal recovery.

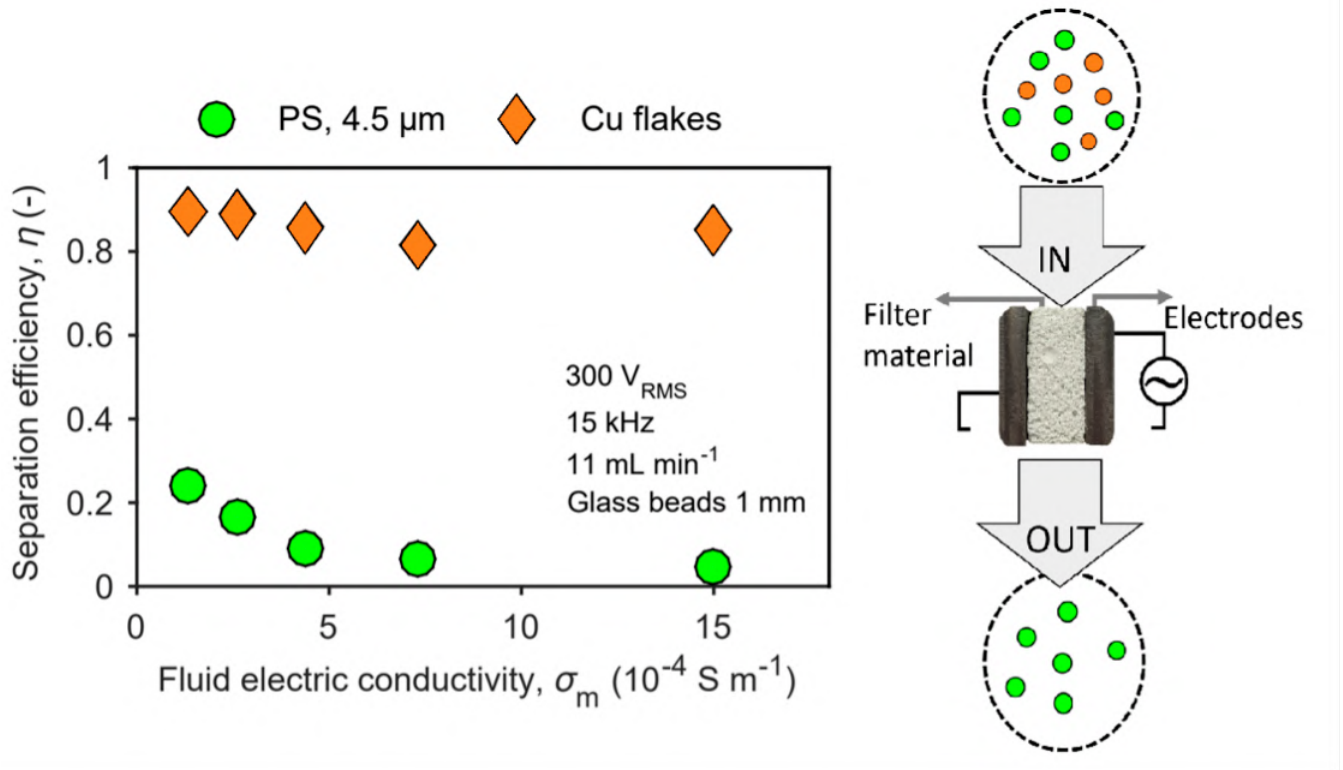


FIGURE 1 Separation efficiency of copper flakes mixed with PS beads at 11 mL/min throughput.

Lorenz, M. et al, Anal Bioanal Chem 412, 3903–3914 (2020). https://doi.org/10.1007/s00216-020-02557-0 Pesch, G.R. et al, Sci Rep 8, 10480 (2018). https://doi.org/10.1038/s41598-018-28735-w

Electret filter media: Experimental and numerical study of submicron aerosol deposition

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Keywords | Direct Numerical Simulation, Discharging methode, Electret, Optimization potential

Electret filter media made of submicro- and microfiber nonwovens are commonly used in aerosol filtration to remove particles from gases (face masks, indoor air filtration, etc.). In the initial phase, the particle filtration efficiency is high combined with a low pressure drop as the fibers of the electret filter media are electrostatically charged. In addition to the mechanical deposition mechanisms (diffusion, impaction and interception), charged particles are deposited by electrophoresis and any (charged or uncharged) particles by dielectrophoresis. However, the evaluation of the electrostatic charge is challenging and there is no established measurement method so far.

Furthermore, particle deposition is reduced with increasing exposure, since already deposited particles, especially in the submicron size range, can alter the fiber charge and thus weaken the above-mentioned electrostatic effects. This was indicated by rudimentary long-term deposition experiments measuring the filter efficiency over certain time intervals. A detailed examination of the interaction of particles and fiber charges has not been performed and thus a relevant understanding of loading-dependent particle deposition with influencing parameters is also still lacking.

Thus, the aim of this work is to develop and validate a simulation method for systematic investigation of the initial and loading-dependent deposition of submicron particles in electret filter media. In the latter case, the simulation method considers the interaction between deposited particles and the fiber charge, whereby the amount of the fiber charge is changed by the amount of the particle charge. In the case of submicrofiber, slip flow must also be taken into account and suitable boundary conditions must be implemented in the simulation

method for this purpose.

The performed experiments with test electret filter media using differently charged sodium chloride particles were applied to validate the simulation method. The comparison of the results from the simulation method and the experiments is also used to evaluation the fiber charge properties and to indicate whether neutralization or shielding is relevant for the reduction of the particle deposition with increasing exposure time.

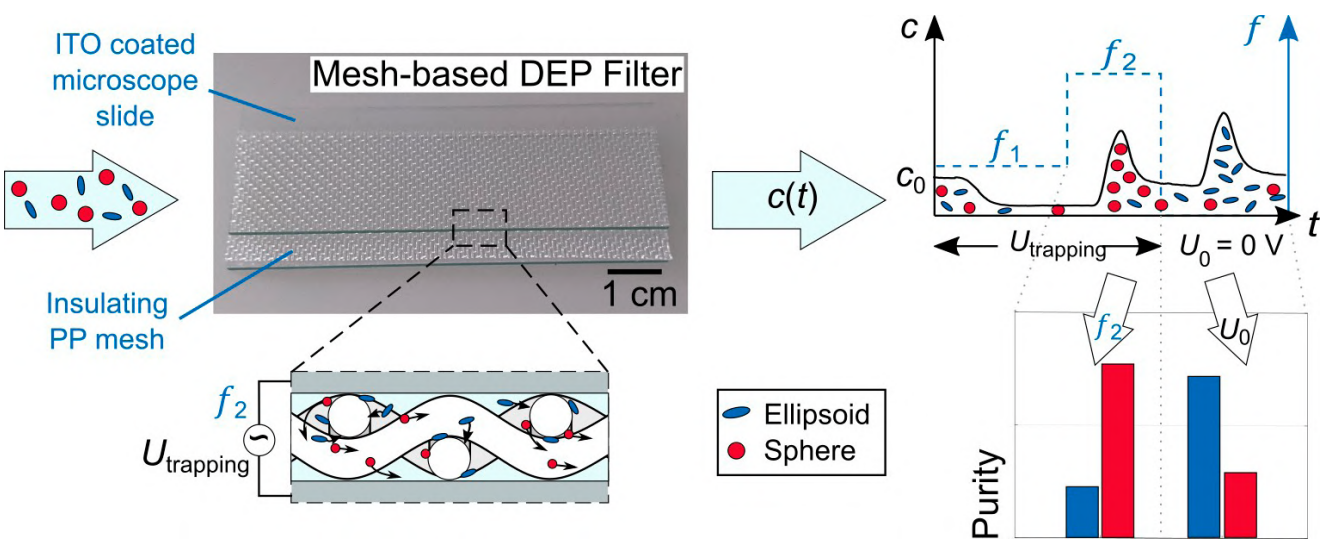
Selective remobilization of microparticles in a mesh-based DEP filter at a high throughput

Weirauch, Laura (1); Giesler, Jasper (1); Baune, Michael (1); Pesch, Georg (1); Thöming, Jorg (1)
(1) University of Bremen

Keywords | dielectrophoresis; shape-selective; selective remobilization; frequency shift; high-throughput

The separation of suspended particles in the lower micrometre range according to their electrical properties, size, or shape is a challenging task especially at high throughput. In numerous of studies dielectrophoresis (DEP) has proven a high selectivity and versatility, which make it a promising separation technique in a variety of fields, such as cancer diagnostics or dust recycling. DEP is the movement of polarizable particles in an inhomogeneous electric field. The direction and strength of the DEP force is not only dependent on particle and medium properties, but can be controlled electrically by the voltage (*U*) and frequency (*f*) of the applied field. So far, however, only a few processes in the field of bioanalytics have made it to commercial use. One of the main challenges in this context seems to be achieving technically relevant throughputs while maintaining the high selectivity.

We present a novel approach of a mesh-based DEP filter. The homogeneous electric field between two flat electrodes is disturbed by an insulating mesh positioned between the electrodes. In the resulting inhomogeneous electric field, the DEP force acts towards or against the direction of field maxima located at the mesh, depending on the particle properties. The regular arrangement of the mesh fibers provides very controlled and repetitive conditions suited to selectively retain particles. Furthermore, the filter offers a high potential for upscaling by using low-cost and commercially available materials. In this DEP filter type, we apply a technique in which particles are trapped and subsequently selectively remobilized via a frequency shift. Selective remobilization was demonstrated on polystyrene particles of different surface properties (conductivities) and shapes. By using a filter of the size of a microscope slide, we achieved purities between 70 % – 97 % at flow rates of 2 mL/min. Our approach of stepwise remobilization of particles exceeds classical DEP methods in multistep selectivity under high-throughput conditions.



From clean room into vehicle cabin: Use of HEPA filter to remove ultra-fine

particles in vehicles

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(1) Ecole Centrale Nantes, (2) MANN+HUMMEL France, (3) MANN+HUMMEL GmbH

Keywords | Air quality, Ultra-fine particles, Filtration, Ventilation, Energy savings

The air quality inside a vehicle is of high interest for the health of humans, particularly for professionals like bus, taxi and truck drivers who spend most of their time on the road. Vehicles operate in rough environments or urban locations with high fine dust concentration. Drivers and vehicle occupants can be exposed to high level of pollution.

The proposed solution to improve the air quality in the vehicle is the Smart Cabin Air Filter system. It introduces a HEPA (High Efficiency Particulate Air) filter in the fresh air path, protected by a pre-filter, in addition to the traditional interior filter in the air conditioning unit. The study focuses on optimizing filter life time. The effect of this smart and sensor driven three- filter system on total energy consumption and driving range will also be part of upcoming studies.

The HEPA filter uses a high-end filtration technology coming from the clean room industry, which plays a major role to limit the concentration of ultra-fine particles in the cabin. In order to maintain its performance at a high level while limiting maintenance costs, its lifespan must be maximised. A system simulation tool is used to compare configurations on elaborated scenarios. The model used is calibrated with experimental data.

Two scenarios will be presented in detail and discussed. First scenario is to preserve the HEPA filter by using the recirculation mode in the cabin as much as possible. Still, a minimum flow rate of fresh air is required to avoid a carbon dioxide intoxication. This fresh air brings particles and pollution on the HEPA filter, which triggers its ageing.

A second scenario is bypassing the HEPA filter when it is not needed to achieve good air quality. This reduces the usage of the HEPA element. With this control, the HEPA filter is used only when the fine particle concentration gets too high in the cabin. The simulation results show that this strategy is helpful: the savings are estimated at 60% in terms of usage time, and 48% regarding the particle loading.

Numerical simulation of filtration of fine particles in the granular bed gas filters

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Keywords | granular bed filter, gas clean, particle separation, LBM-DEM

Granular gas filters are used to remove the gas entrained particles by passing through a granular filter media, where the particles are too small to be collected by the conventional device such as cyclones, typically for the particles less than 5 or 10 μm in diameter. Recently, these fine particles, i.e. PM2.5, raised a significant concern, due to the irreversible damage to the environment and human health. However, the separation of these fine particles is a significant challenge due to their low inertia and low diffusion. Therefore, a high-efficient technology of particle filtrations is required for the industrial systems.

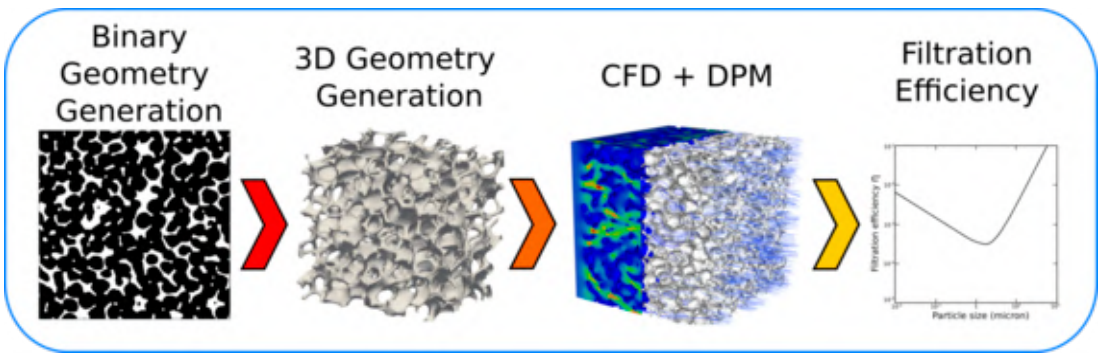
The separation efficiency of the fixed bed granular filter can be greater than 99%. Meanwhile, the granular bed filter can also process the hot gas with the temperature up to 800°C. However, during the filtration, the collected particles accumulated in the filter cake which increased the operation pressure, then failed the system operation. Under this condition, the operation should be stopped for cleaning at a regular time. To realize the continuous operation, some new technologies, such as the fluidized and moving granular beds, were developed, whereas their filtration efficiencies were still not as high as the fixed bed. To separate the gas-entrained fine particles in high efficiency and optimize the granular filter, therefore, it is essential to study the filtration mechanism of the fine particles with lower inertia in the granular bed.

In this study, the particle filtration in the porous media of the granular bed is simulated using the coupled Lattice Boltzmann method (LBM) and discrete element method (DEM). LBM is used to predict the gas phase flow in the porous media of the granular bed, and DEM is used to trace the motion of the fine particles. Using the LBM-DEM simulation, the mechanism of the filtration of the low-inertia particles was studied affacated by the structure of the porous media. The study predicts the separation efficiency, distribution of particles accumulated in the granular bed, and their effects to the evolution of the pressure drop and permeability of the porous media in the granular bed.

Particle capture in open-cell foams: filtration efficiency and comparison with granular beds

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Keywords | Particle filtration, Open-cell foams, CFD, DPM



Solid particulate in the liquid hydrocarbon feed of catalytic packed-bed reactors in refining processes is a well-known problem that causes exponentially increasing pressure drops, due to their trapping in the granular bed, that eventually lead to the reactor shut down. Traditional filters are inefficient in removing particles with dimensions in the range $1 < dp < 20$ µm, or induce a too high pressure drop. The deposition phenomena of such systems are analogous to those described by Yao et al. (1971) for water filtration in granular beds. When the solid density is much higher than the liquid one, the captation is mainly the results of inertial deposition and interception with the collector mechanisms.

The use of open-cell ceramic foams as filter medium is well established for processes involving molten metals. Their open cellular structure makes them suitable for deep-bed filtration and moreover their high porosity and specific surface results in lower pressure drops compared to other packing media, such as granular beds. However, the influence of their geometrical structure, topology on the characteristic length and filtration efficiency remains an open problem.

The aim of this work is to investigate the particles capture inside ceramics foams, to extract the key parameters and characteristic lengths affecting the filtration efficiency and compare them with more classical granular beds. The investigation is carried out performing numerical CFD simulations of the fluid flow inside foams geometries obtained either from x-ray tomography reconstruction or from digital generated model using an improved workflow based on Agostini et al. (2022), allowing the exploration of a large set of parameters. The solid particle is represented using lagrangian Descrete Particle Model (DPM) simulations, under the hypothesis of laminar regime and diluted systems and no influence of the particle on the fluid flow.

Yao, K.M., Habibian, M.T., O'Melia, C.R., 1971. Water and wastewater filtration. concepts and applications. Environmental science & technology 5, 1105–1112.

Agostini, E., Boccardo, G. and Marchisio, D., 2022, "An open-source workflow for open-cell foams modelling: geometry generation and CFD simulations for momentum and mass transport.", Chemical Engineering Science: 117583.

Particle filtration with innovative submerged micro- and ultrafiltration membrane elements

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(1) Technische Hochschule Köln

The main requirements for submerged membrane elements are a high packing density and a low energy demand during operation. To

reach these goals a new type of an aerated spiral-wound membrane element for micro-and ultrafitration with an innovative open-channel feed spacer was developped and investigated.

Experimental tests and CFD simulations allowed the determination of an optimal air bubble size and therefore the optimal channel height of the corrugated feed spacer with regard to the air purge and the membrane element manufactoring process. Using this spacers, the self-fabricated elements have a stable construction and can be backwashed to a sufficient degree which allows a chemical cleaning from the feed as well as from the permeate side without the need to remove the element.

Applying these elements, field tests for particle and activated sludge filtration were conducted. The main subject of the experimental work was to optimize the filtration techniques aiming to minimize the energy demand as well as the purge air volume. Cleaning procedures were also investigated and improved.

For particle filtration, the depostion of the particles was estimated based on force equillibrium as a function of the volume flow, particle size and crossflow velocity. With the chosen specific purge air flow rates of up to 0.2 $m^3/(m^2h)$ relatively low crossflow velocities of up to 0.4 m/s were achieved. Low crossflow velocities are associated with low energy demand, but they also result in a partcle deposition on the membrane surface. The above-mentioned operating conditions result in a critical particle size of about 1 µm and a limiting permeate flux of 10 $L/(m^2h)$. In the case of back-washing or the filtration of larger particles permeate flux can be increased.

The required energy depends on the operation mode, the suspension and its concentration which affects the membrane permeance. For the filtration of activated sludge only about 150 Wh/m^3 were needed. For particle filtration the energy requirement could be reduced to 40 and 60 Wh/m^3. The results show, that the specific energy demand is significantly lower than for comparable submerged systems.

Bi-dimensional Fractionation of Rare Earth Compounds by Magnetic Field Controlled Chromatography

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(1) Eggenstein-Leopoldshafen

Keywords | Particle Separation, Magnetic Field Fractionation, Rare Earths, Magnetism, Chromatography

Various paramagnetic rare earth (RE) compounds are vital components determining the functionality of high-tech products. The market for RE-containing parts has increased strongly in recent years as a result of the rapid expansion of future technologies. Coincidently, manifold property dimensions and purity of such particle collectives are essential and quality- critical for the production and application of coatings, medical therapy or diagnostic systems, catalysts and electrochemical energy storage systems or of electronic assemblies, e.g. in the fields of magnet and battery technology. The journey towards increased raw material efficiency and sustainability of global supply stream management thus depends on highly productive and selective as well as environmentally friendly processes for the separation of these valuable particles from ores and complex end-of-life products.

Unfavorably, many prevalent RE extraction processes rely on organic solvents as well as strong acids and bases. Additionally, most RE particle fractionation methods provide only limited productivities and selectivities, particularly in the size range of ultra-fine systems (< 10 µm) because of the complex superposition of forces acting on the particles. The fractionation of RE particles at industrial scales thus remains a relevant research task.

In this work, we used magnetic field controlled chromatography for a selective, efficient, and easily scalable fractionation of paramagnetic particle systems. The technique is based on the competition between magnetic and hydrodynamic forces.

Magnetic interactions evolve between the particles and a magnetizable matrix as a result of the application of an external magnetic field, see Figure 1; hydrodynamic forces are induced at will by adjusting the flow rate through the chromatography column. Diverse particle collectives containing RE metals (e.g. Dy_2O_3, Ho_2O_3 and Nd_2O_3) and luminescent RE doped phosphors (e.g. $LaPO_4:Eu^{3+},Tb^{3+}$ and $Y_2O_3:Eu^{3+}$) and their mixtures were fractionated bi- dimensionally, specifically regarding particle size and magnetic susceptibility. High selectivities of $/tfracd_{25}d_{75} > 0.65$ were attained while concurrently providing easy scalability. Furthermore, we demonstrated the feasibility of a process transfer to a continuously operating Simulated Moving Bed (SMB) chromatography system with low energy, space, and solvent

consumption, enabling space-time yields potentially relevant for industrial application.

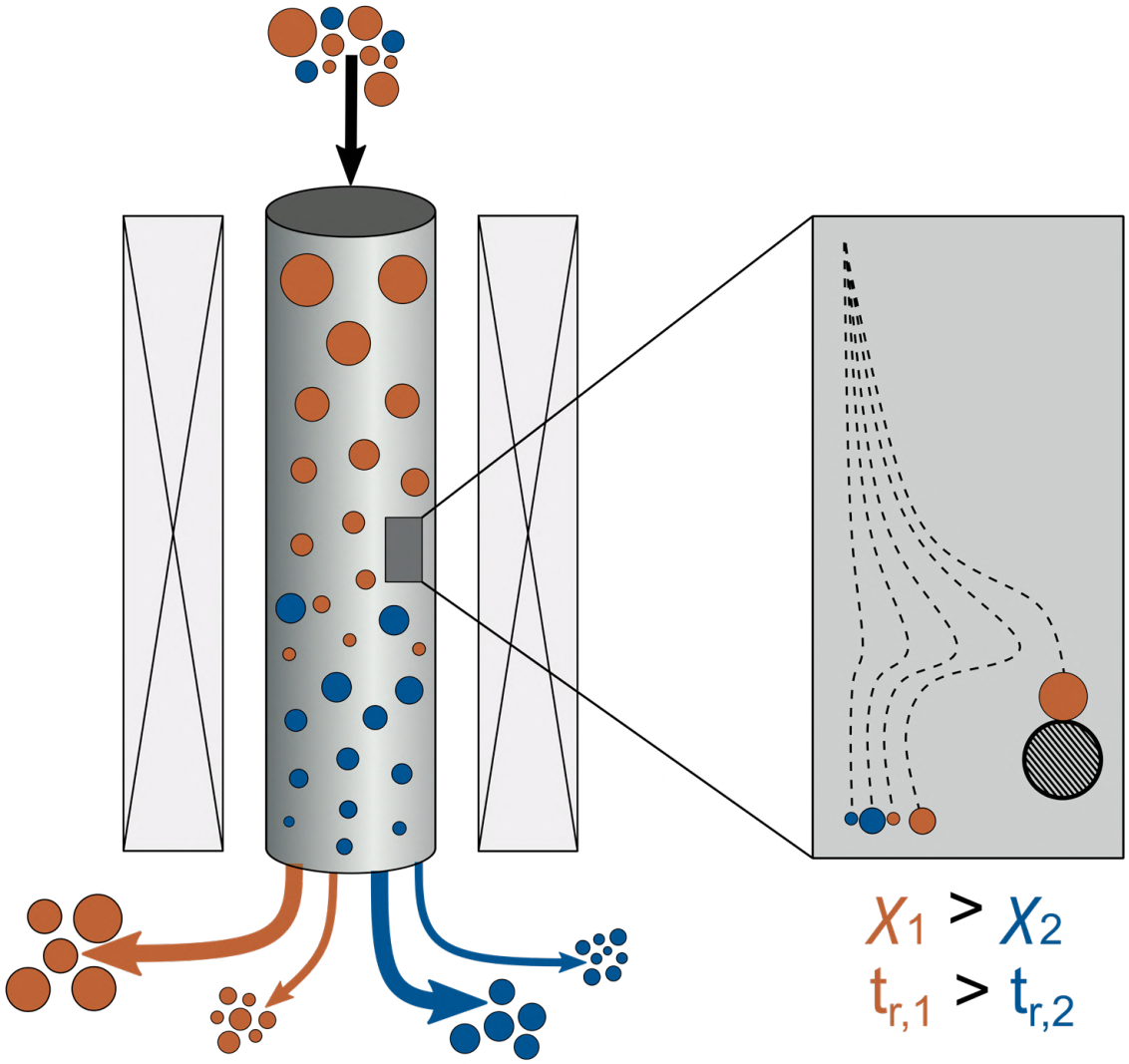


FIGURE 1: Schematic principle of magnetic field controlled chromatography technique. $t_{r,i}$ are the characteristic retention times, $/chi_i$ indicate the specific magnetic susceptibilities.

Nanoparticle depresants in fine particle separation – the effect of colloidal silica in calcium mineral flotation

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Keywords | Froth Flotation, Nanoparticles, Adsorption, Bubble Attachment, Particle Interactions

The micro particle separation of calcium minerals by means of selective froth flotation is a challenging task. The difficulty arises because of the similar surface properties of the minerals, and thereby similar responses to various known families of flotation collectors (selectively adsorbing surfactants). Within the scope of this study colloidal silica is investigated as a potential selectively interacting nanoparticle depressant in the flotation process of calcium minerals. The effect of the colloidal silica and its interactions with the reagent system were investigated by varying its modification and specific surface area/particle size. Single-mineral microflotation of scheelite (calcium tungstate), fluorite (calcium fluoride), calcite (calcium carbonate) and apatite (calcium fluoro phosphate) were used to determine if colloidal silica would

have any effect on the minerals. First results show that colloidal silica prevents calcite from floating while scheelite, fluorite and apatite are unaffected by the presence of the reagent, regardless of the dosage.

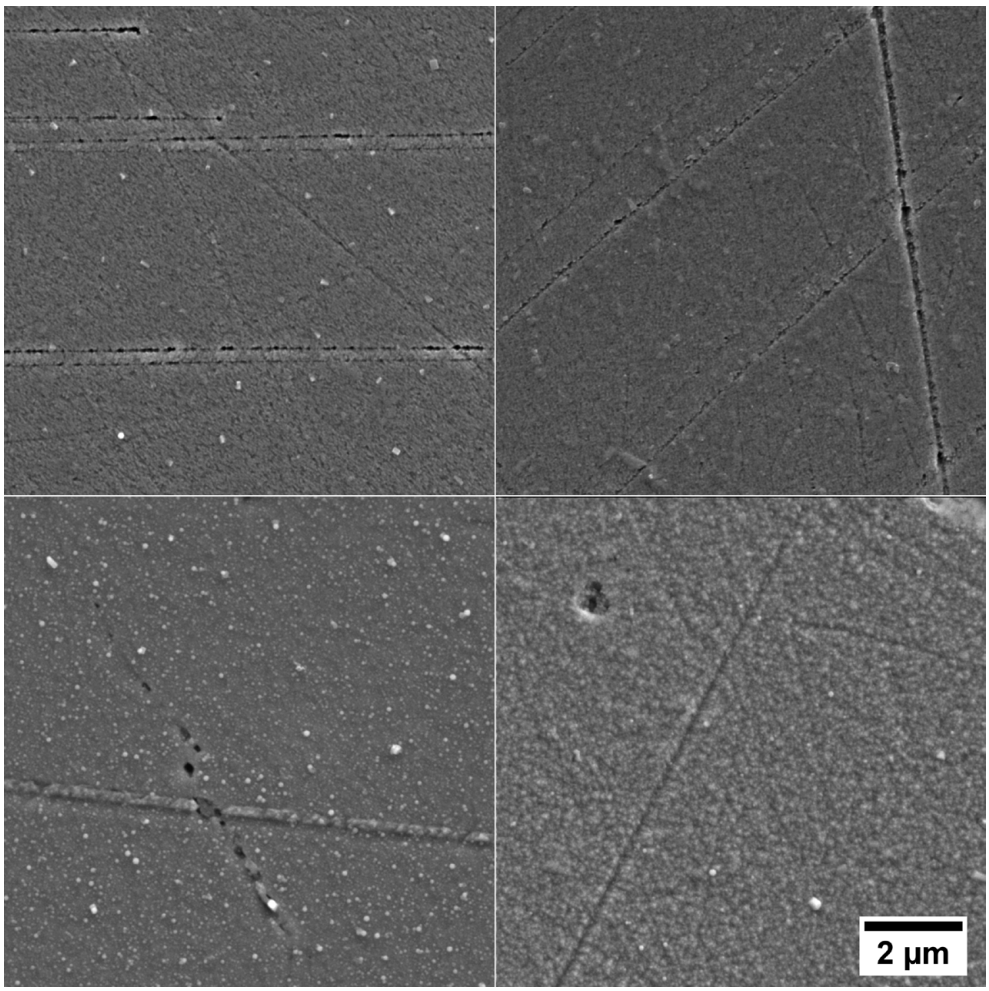


FIGURE 1: The figure shows SEM images of calcite (left) and scheelite (right) surfaces before (top) and after (bottom) conditioning with a colloidal silica dispersion with nanoparticles adsorbed on the calcite surface (bottom, left) but less pronounced on the scheelite surface (bottom, right).

First batch flotation tests have shown significant differences between the three modifications in terms of the significant effect on the selectivity. The same finding was made when varying the specific surface area/particle size. Further surface chemistry studies will provide more insight on the depression mechanisms of colloidal silica in calcium mineral flotation.

Investigating the influence of multiple particle properties on the separation of ultrafine particles via enhanced froth flotation

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Keywords | Ultrafine particles, multidimensional separation, flotation, particle characterization, partition curves

One of the most important techniques to separate valuable (mostly mineral) particles from other particulate phases (often unwanted so called gangue) is froth flotation. It is an efficient process for particles with sizes ranging from 10 µm to 200 µm and its main separation feature is the difference in particle wettabilities. Nowadays, electronic devices contain large amounts of fine composite particles, that need to be crushed for sufficient liberation, resulting in ultrafine particles (< 10 µm), whereby new challenges arise. Therefore, existing flotation techniques need to be adapted and improved in order to have an efficient separation. For that reason, this project, which is part of the

German research foundation priority programme DFG-SPP 2045 “MehrDimPart”, aims at developing a method for the separation of ultrafine particles based on multiple particle properties, such as size, morphology or surface energy.

A particle system consisting of ultrafine size fractions of glass particles as the floatable material and magnetite as the non- floatable material is used in this study. The hydrophobicity of the glass particles is modified via an esterification reaction using alcohols with differing chain lengths, resulting in particles with defined wettability states with water contact angles ranging from 40° to 100°. Information on the particle size and shape is obtained via a combination of laser diffraction and microscopic analysis. The technique of flow cytometry is introduced for multidimensional particle characterization, as it allows for simultaneous size and shape analysis. All flotation tests are carried out in batch mode using a novel flotation apparatus, specifically designed for the separation of ultrafines by combining advantages from machine-type froth flotation and column flotation. The separation process is evaluated using multidimensional partition curves, where the impact of specific shape and size descriptors, e.g. aspect ratio, roundness or ferret diameter, becomes evident as it affects the separation and the underlying micro processes. This investigation will help to further understand how certain particle properties influence flotation, as well as other separation processes. In this way, the separation of ultrafine particles will become more efficient, which will play an important role especially in the recycling of secondary materials.

Highly specific and multidimensional separation of fine particle systems with technical relevance: Understanding of the micro processes

Benker, Bernd (1); Cierpka, Christian (2); Fritsching, Udo (3); Hussong, Jeanette (4); Kruggel-Emden, Harald (5); Nirschl, Hermann (6); Pesch, Georg (7); Peuker, Urs Alexander (8); Weber, Alfred (9)
(1) Cutec Research Center, TU Clausthal, (2) Institute of Technical Thermodynamics, TU Ilmenau, (3) Institute of Particle Technology, Univ. Bremen, (4) Institute of Fluid Mechanics and Aerodynamics, TU Darmstadt, (5) Institute of Particle Technology and Processing, TU Berlin, (6) Institute of Particle Technology and Applied Mechanics, KIT, (7) Institute of Chemical Process Engineering, Univ. Bremen, (8) Institute of Particle Technology and Processing, TU BA Freiberg, (9) Institute of Particle Technology, TU Clausthal

Keywords | multi-dimensional particle properties, multidimensional separation function, flow field fractionation, separation at interfaces, flotation

Highly specific and multidimensional separation of fine particle systems with technical relevance: Understanding of the micro processes
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For improving highly specific and multidimensional separations of fine particle systems, an excellent understanding of the micro processes is essential. In these micro-processes, different particle characteristics are addressed by external fields. In contrast to the classical separation according to a single characteristic, the interactions between the micro processes pose a major challenge in the case of multi-parameter separation.

In different approaches of flow field fractionation of particles, for instance serial arrangements of flow constrictions (Hussong & Kruggel-Emden), superpositions of acoustic and electric fields (Fritsching), and of two surface acoustic waves (Cierpka) use the interplay between inertial forces and drag forces to separate particles by size, shape, and density. The interaction of air bubbles with particles is employed in ultrasonic-assisted microbubble flotation (Peuker), nanobubble-induced centrifugal field flotation (Benker/Weber) and in the combination of fractionation in the foam and hydrophilic selective flocculation (Rudolph) to separate particles according to their surface properties, size and shape. By using stationary magnetic fields (Nirschl) and alternating electric fields (Pesch), particles are also separated according to their magnetic and dielectric properties as well as size and shape. In all projects, also supported by a central unit within the SPP MehrDimPart (Peuker), in-depth characterization methods were developed to assess multidimensionality as a basis for creating multidimensional separation functions.

In summary, different micro processes have been realized experimentally and captured in simulations, which show ways for the multidimensional separation and fractionation of fine particle systems at technically relevant throughputs.

Modelling the motion of dust particles in electrostatic travelling wave fields

Yu, Yue (1); Hadler, Kathryn (1); Cilliers, Jan (1); Starr, Stanley (1); Wang, Yanghua (1)
(1) Imperial college london

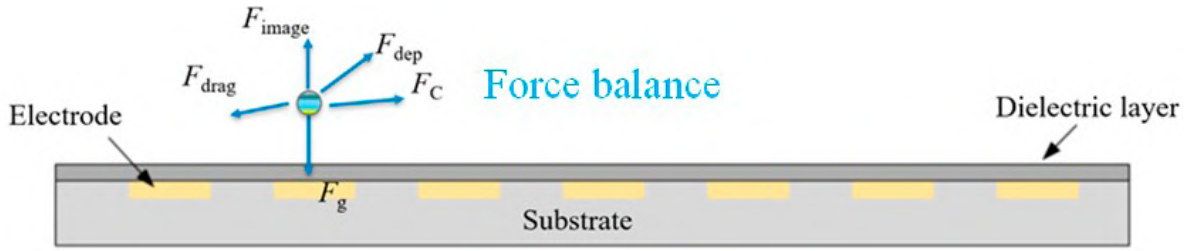
Keywords | Particle separation; Electrostatic traveling wave; Numerical simulation; Perturbation method; Runge-kutta method; Space resource utilisation; dust mitigation

Electrostatic travelling waves can be used to transport particles across surfaces. This technology has received particular attention for dust mitigation on solar panels (Mazumder et al., 2019)and for the manipulation of dust particles on the Moon as part of Space Resource Utilisation (SRU) processes (Adachi et al., 2017). Electrostatic travelling waves systems are particularly well-suited to the space environment due to the lack of moving parts and absence of moisture. The speed and motion of the particles across a travelling wave system depends on their physical characteristics, such as particle size, the design of the system, including electrode spacing, and the electrical field produced, for example voltage and frequency. In this study, we consider the motion of particles of different size with the aim of exploiting differences in motion and speed such that particles can be separated by size.

Numerical simulations and analytical perturbation methods are used to help understand particle motion in electrostatic traveling wave field. The electric field is calculated using fixed boundary conditions based on the Fourier expansion method, which shows more detail near the surface of electrodes. A more stable method, implicit Runge-Kutta method with fixed step, is chosen to solve motion equations. We investigate the effect of frequency, particle size and electrode width and gap on the particle moving characteristics, such as particle velocity, moving height and motion modes. A novel map of particle motion modes is presented, which shows the sensitivity of particle motion to different parameters. Analytical results of a particle moving in one specific mode are compared with numerical results, showing good agreement. Guidance on the design of electrostatic travelling wave systems for different applications are given based on the results, with a particular focus on particle size classification.

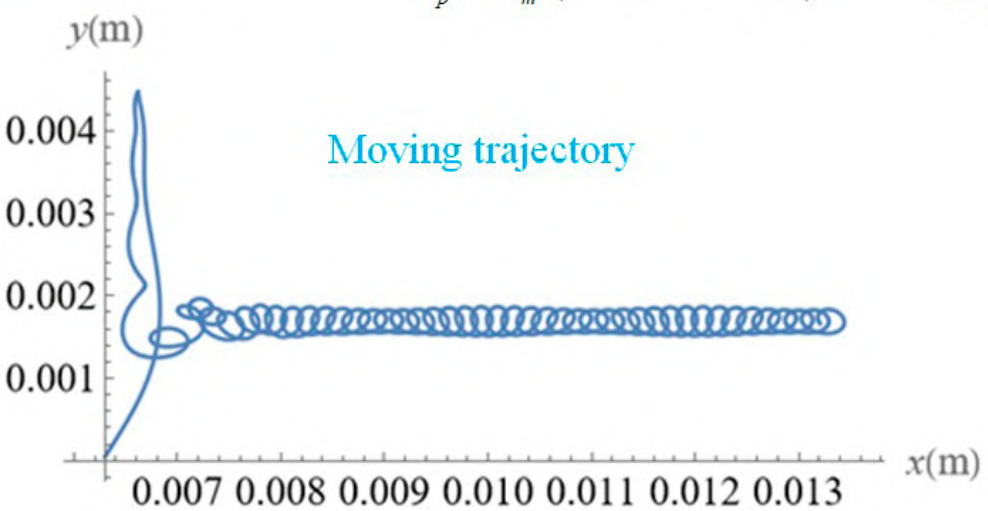
ADACHI, M., MOROKA, H., KAWAMOTO, H., WAKABAYASHI, S. & HOSHINO, T. 2017. Particle-size sorting system of lunar regolith using electrostatic traveling wave. Journal of Electrostatics, 89, 69-76.

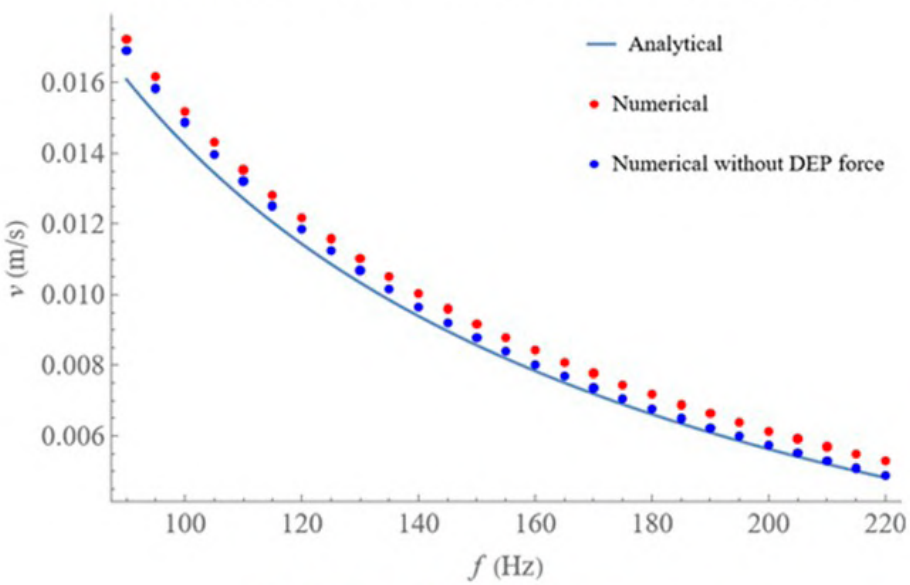
MAZUMDER, M., ELINGER, C., O'CONNOR, K., et al., . Industrial Production and Field Evaluation of Transparent Electrodynamic Screen (EDS) Film for Water-Free Cleaning of Solar Collectors. In: IEEE (ed.) 2019 IEEE 46th Photovoltaic Specialists Conference (PVSC). IEEE.



Moving equation

$$\begin{cases} m\dfrac{d^2x}{dt^2}+6\pi\eta R\dfrac{dx}{dt}=E_x\cdot q+4\pi R^3\varepsilon_0\varepsilon_m\dfrac{\varepsilon_p-\varepsilon_m}{\varepsilon_p+2\varepsilon_m}\left(E_x\dfrac{\partial E_x}{\partial x}+E_z\dfrac{\partial E_z}{\partial x}\right) \\ m\dfrac{d^2z}{dt^2}+6\pi\eta R\dfrac{dz}{dt}=E_z\cdot q+4\pi R^3\varepsilon_0\varepsilon_m\dfrac{\varepsilon_p-\varepsilon_m}{\varepsilon_p+2\varepsilon_m}\left(E_x\dfrac{\partial E_x}{\partial z}+E_z\dfrac{\partial E_z}{\partial z}\right)-mg-\dfrac{q^2}{4\pi\varepsilon_0(2z)^2} \end{cases}$$





Relationship between average speed and frequency

Numerical study of the transport and retention of particles in the slurry through the porous media

Sun, Dan (1)
(1) National Institute of Clean and Low Carbon Energy

Keywords | particle transport, separation, filter cake, porous media, CFD-DEM

In the porous media, the particle migration is confined, and these particles are eventually retained by the structure. As a result, the structure and topology of the porous media are altered, which, in turn, varies the fluid dynamics of both the fluid and particles. Clogging issues occur due to the accumulation of the particles and cause the damage to the productivity by the increment of the permeability of the porous media. Hence, the filter cake affected by the retention of fine particles is also in highly concerned to be investigated.

In this study, particle filtration in the porous media is simulated using a Eulerian-Lagrangian simulation method, Computational Fluid Dynamics (CFD)-discrete element method (DEM). The fluid dynamics of the liquid is simulated using CFD method. DEM is used to trace the particles under the Lagrangian reference frame. The discrete particle trajectories are predicted by integrating the force balance on particles.

To study the filtration of particles in the porous media, the particles are carried by the fluid in the slurry and injected into the porous media. The retention of the particles are studied with their effect to the filter cake permeability as well as the fate and conversion of the flow path.

FLASH COMMUNICATIONS

Reduction of the rate of pressure drop increase in surface filters for dust separation through additive dosing or coarse dust recirculation

Schmidt, Eberhard (1)
(1) Institute of Particle Technology, University of Wuppertal

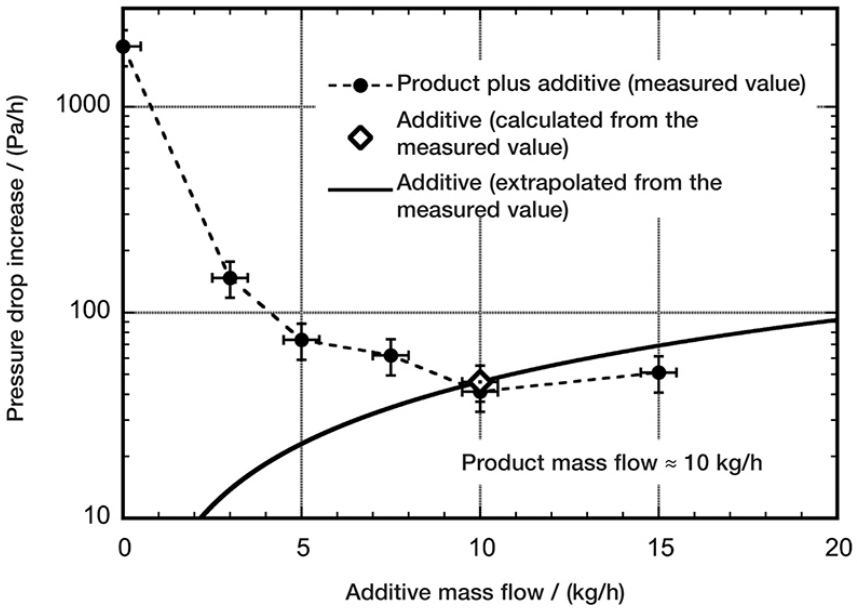
Keywords | Dust separation, surface filter, additive dosing, dust recirculation.

Surface filters are widely used throughout industry to separate particles from gases. The increase in the pressure drop over time is a decisive parameter with regard to the operating performance. Dosing with a coarse-grained additive or recirculating coarse materials from the dust bunker may reduce the pressure drop increase by more than one order of magnitude. This can be shown by using an example on a technically relevant scale.

The application example used here makes it clear that a filter system's operating performance can be considerably improved by specifically manipulating the composition of the raw gas and in this case, it was even possible to economically use a specific design (bag filter) that was already installed. However, it has not yet been generally clarified which additives are particularly suitable for conditioning the different raw gases. The same applies to the required quality of the recirculated material that will be processed, especially with regard to particle and agglomerate size distributions.

Initial model calculations indicate that the amount of dosed dust should approximately correspond to the amount of dust introduced with the raw gas in order to realise the lowest possible pressure drop increase. However, a deeper understanding of the interactions between all of these factors is still needed.

The objective of future work here is to develop a method for reducing the pressure drop rate increase and extending the cycle time of surface filters that are preferably regenerated through pulse jets, using specific recirculation processes involving the coarse dust from the filter system's dust collecting container and having them controlled by model calculations. This should result in the particle emissions into the purified gas being reduced, an increase in the service life of the filter elements and a reduction in operating costs.



Effect of an additive (stone dust) on the pressure loss development Δp/t with existing product (ammonium nitrate and ammonium sulphate) and a comparison against pure additive dosing.

Schmidt, Eberhard, Reducing the pressure drop rate increase in surface filters used for dust separation through additive dosing or recirculation, F&S Internat. Ed. No. 20/2020: 54 - 58.

Development of composite filter media for continuous cake filtration without gas throughput

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Keywords | Cake Filtration, Membrane-woven-composite, Dewatering, Fabrics, Membranes

Filter cakes resulting from the filtration of concentrated suspensions in continuously operating filters are often dried thermally afterwards to obtain the dry product. This is the most expensive process step. Mechanical dewatering can significantly reduce the moisture in the filter cake and thus the energy required for thermal drying. The result is significant cost savings. In gas differential pressure dewatering, the liquid present in the pore system of the filter cake is removed after overcoming the inlet capillary pressure. The emptied pores of the filter cake lead to an undesired gas throughput. This has economic and technical disadvantages, because the gas throughput has to be compensated by the vacuum pump. In this contribution, the investigations for the development of a filter media is presented, which can avoid this gas throughput. A combination of a woven filter cloth and a microporous membrane is used for this purpose. Due to the microporous properties of the membrane, the conditions for gas throughput-free cake filtration are given. The membrane capillary pressure is higher than usual differential pressures for filter cake dewatering. Therefore, liquid can be removed from the filter cake but not from the membrane pores. The result is a gas throughput-free cake filtration. The focus of this work is on the systematic investigation of a highly stressable connection between filter cloth and membrane. A joining process is to be designed so that the membrane and fabric are connected at their surface. For the initial experiments in lab-scale, various combinations of filter media and membranes are being investigated and functional samples are being produced. With these samples the strength of the connection and the filter media properties can be assessed afterwards.

Nonlinear Regression of Cake Filtration Experiments

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Keywords | cake filtration, nonlinear regression, residuals

Nonlinear parameter estimation of experimental cake filtration data of the form Vf = f(t) is presented as a potent alternative to the classical linear regression for cake filtration experiments on transformed data of t/Vf vs. Vf. In these regression methods, Vf is the volume of produced filtrate and t is the time that has elapsed since the measurement of first filtrate.

In can be shown that there is a significant error in the determination of the cake resistance values when using the linearized fit method compared to a much more exact result when nonlinear regression is employed, which may be larger than 20 %, depending on the particle system. This error can be traced to the negligence of the time offset which occurs to more or less great extent in every experiment. The cause of the time offset is shown not to arise because of bad time-keeping, but because of nonideal processes in the beginning stages of filtration.

The goodness-of-fit is shown to be best observed through the means of the residual plot, which may show any correlations between the regression's residuals and the independent variable, thereby hinting at superposed processes, such as a clogging of the filter medium, variable compression of the filter cake, sedimentation, etc. Fig. 1 shows the results of both linearized and nonlinear fits on the same cake filtration experiment at constant pressure.

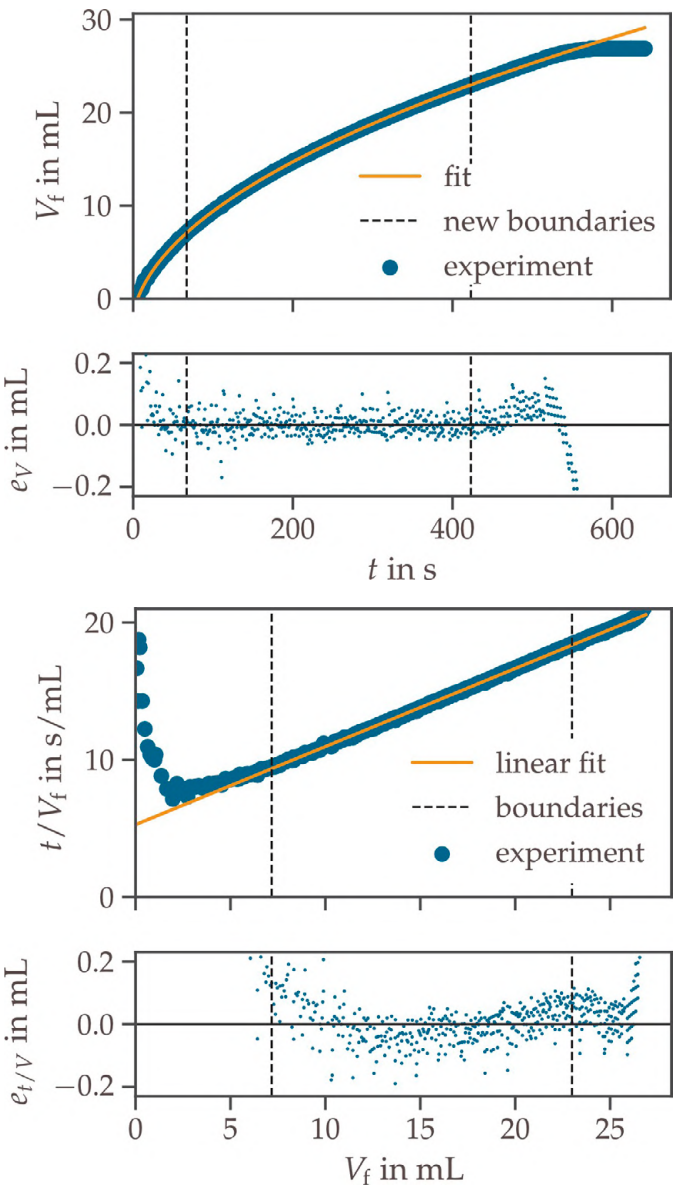


Fig. 1: Regression results for the same data set. Left: Nonlinear regression fit three fit parameters, including a time offset. Right: Linear regression with nonoptimal fit result, in which the time offset is neglected.

The increased accuracy of the nonlinear fit method can provide a deeper insight into the inner workings of the cake filtration process. Additional processes can be described by the superposition of model equations, which may further the insight into the cake filtration process overall.

A vast number of experiments serves as a foundation for this talk. Parts of the results have been published in a dissertation on the topic (Buchwald, 2022).

Buchwald, Thomas (2022) Nonlinear parameter estimation of experimental cake filtration data. Dissertation, TU Bergakademie Freiberg, 2021, https://nbn-resolving.org/urn:nbn:de:bsz:105-qucosa2-769740.

Monitoring and Optimization of Colloidal Fractionation in Tubular Centrifuges

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Keywords | Centrifugation, Fractionation, Mutlicomponent Suspensions, Online Monitoring, Process Optimization

Classification is an established process in solids processing technology to divide particulate products into defined fractions based on their size. However, complex particle systems with strict specifications for product-relevant properties often require a different approach. Thus, multidimensional fractionation is needed in which both geometric (particle size and shape) and material (density, interfacial properties) separation characteristics are considered. Preceding studies focused on the subordinate step of classification in semi-continuous tubular bowl centrifuges, which enable the separation of ultrafine particles due to high rotation speeds at high throughput rates. For multi-component inlet suspensions, however, the achievable selectivity in sorting different colloidal materials has not yet been investigated.

The experimental studies of this work include density fractionation experiments of selected fine particles which differ from each other in terms of their solid density. Their particle size is distributed over several classes in the lower micrometer and nanometer range. The PSD and solids volume fraction are determined by means of analytical centrifugation. Regarding the downstream evaluation of the fractionation result, UV-VIS spectroscopy is a promising method. It can provides valuable real- time analysis regarding the composition in the centrifuge overflow. For this purpose, spectral data of the product feed is compiled and correlated. Subsequently the regression is used for the quantitative analysis of the concentration after any given separation experiment. By adapting the operating conditions of the tube centrifuge, an optimization of the material separation will be investigated. The established online measurement methodology helps with this objective.

If accepted, this presentation may give new insights into the multidimensional fractionation on a pilot scale. Furthermore, the results will indicate a possible increase in process efficiency by real-time evaluation of the selectivity of material fractionation processes in tubular centrifuges.

7 AEROSOL PARTICLES

KEYNOTE LECTURES

Particle-Surface Impacts at Supersonic Speeds: From Aerosol Deposition to High Speed Flight Vehicle Damage Assessment

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(1) University of Minnesota, (2) Shanghai Jiaotong University

Keywords | aerosol deposition, supersonic nozzles

Aerosol particle-surface collisions at supersonic (Ma>1) speeds can be utilized in nozzle systems as means to produce near bulk density coatings (aerosol deposition), but can also be a process leading to damage of high speed flight vehicles encountering particles (e.g. Martian dust storms). While aerosol deposition systems have been optimized for a number of material systems and the transition from plastic deformation and adhesion to ablation upon collision is qualitatively understood, there are a number of features of high speed aerosol impacts which are not yet quantitatively understood. This presentation highlights some of our group's recent efforts to understand what governs particle impact speeds in nozzle deposition systems and the effects of impacts on both surfaces and particles. Specifically, through computational fluid dynamics and particle trajectory calculations with a drag model valid across a wide Ma, Kn range, we show that in any supersonic nozzle system there is a particle size for which the impact velocity is optimized; smaller particles will be decelerated by the bow shock at the deposition surface and larger particles are not fully accelerated in the nozzle.

Furthermore, we find that there is an optimum size for aerodynamic focusing of particles, enabling deposition of extremely small line widths with monodisperse particles of the appropriate size. Through molecular scale simulations comparing high speed deposition to traditional high temperature deposition approaches, we find that the experimentally observed loss of crystallinity in high speed deposited aerosol particles arises because of the strong thermal gradients resulting during deposition, i.e. when particles deposit there is conversion of translational kinetic energy to thermal energy. Finally, using phase Doppler particle analysis we confirm that in nozzle systems, it is possible to accelerate micropowder particles to velocities in excess of 600 m s-1, and individual impacts at these speeds can lead to damage in substrate materials, quantified by atomic force microscopy.

Enhanced light absorption and radiative forcing by black carbon agglomerates

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(1) ETH Zurich, (2) The Ohio State University

Keywords | black carbon, agglomerates, light absorption, radiative forcing, climate change

The climate models of the Intergovernmental Panel on Climate Change (IPCC) list CO2, CH4 and black carbon (BC), as the most potent contributors to global warming based on their radiative forcing (*RF*) impact. Among them, the BC contribution comes with the highest uncertainty (~ 90 %). Examining closely these models, it becomes apparent that they might underpredict significantly the direct *RF* for BC, largely due to their assumed spherical BC morphology (Kelesidis & Pratsinis, 2021). In specific, the light absorption and direct *RF* of BC agglomerates are enhanced by light scattering between their constituent primary particles (Kelesidis et al., 2020) as determined by the Rayleigh-Debye-Gans theory interfaced with discrete dipole approximation (Kelesidis & Pratsinis, 2019) and recent relations for the refractive index and lensing effect. The resulting light absorption agree very well with the observed absorption aerosol optical depth of BC. ECHAM-HAM simulations accounting for the realistic BC morphology and its coatings reveal high direct *RF* = 3 - 5 W/m2 in East, South Asia, sub-Sahara, western Africa and the Arabian peninsula (Fig. 1). These are in agreement with satellite and AERONET observations of RF and indicate a regional climate warming contribution by 2.4 - 4 oC, solely due to BC emissions.

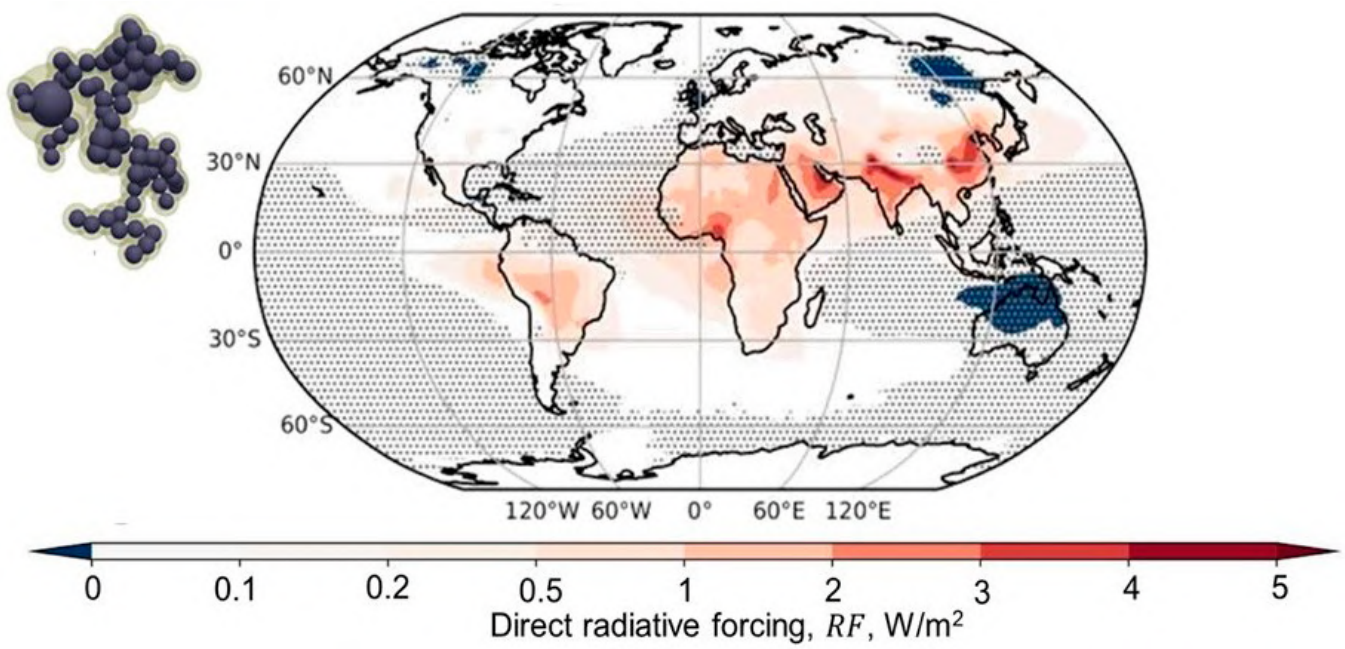


Figure 1. Global map of the direct *RF* of coated BC agglomerates estimated by ECHAM-HAM.

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Kelesidis, G. A., and Pratsinis, S. E. 2021. Determination of the volume fraction of soot accounting for its composition and morphology. Proc. Combust. Inst. 38:1189-1196, doi.org/10.1016/j.proci.2020.07.055.

Can we tune the particle properties by flame spray pyrolysis for high-end photocatalysis ? Challenges and oportunities

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Keywords | Flame Spray Pyrolysis, Artificial Photosynthesis, Photocatalysis, CO2 reduction, H2, Defects, Vacancies, Selectivity, Scale Up production

Flame Spray Pyrolysis (FSP) is an established aerosol technology for production of metal oxides at industrial level. Artificial Photosynthesis is considered among the most challenging high-end photocatalytic technologies, since it encompasses H2O splitting, H2 production and ultimately CO2 reduction towards added-value organics . Optimizing the FSP process to each- one of these steps, plus the selectivity of CO2 reduction towards a targeted product, pose great challenges on nano-physical chemistry, and FSP process engineering. Defect engineering vs. lattice integrity, surface configuration vs. selectivity, cocatalyst-particle synergy vs. inhibition, are among the challenges that the FSP process is owing to face and optimize.

Herein we will discuss these aspects for three case-studies on innovative FSP-made oxide nanomaterials ZrO2-x, Cu2O, NaTaO3. FSP reactor configurations will be discussed in tandem with the subtleties posed by the demand for multiparametric balancing of the particle properties towards competitive Artificial Photosynthesis.

Potential and limitations of CFD and DEM simulation in the design of orally

inhaled drug products

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Orally inhaled drug products (OIDPs) are pharmaceutical forms involving the entrainment of powder particles or droplets by an air stream generate by patient breathing or by a single inspiratory act. Many aspects, in the design of these products, require an intimate understanding of fluid and powder behavior as well as their complex and non-linear interplay in determining the aerosol physics. Recently, simulation tools based on computational fluid dynamics (CFD) and discrete element modeling (DEM) have attracted a growing interest in the OIDPs community, thanks to their explanatory and predictive capabilities. Some recent and promising applications of CFD-DEM computational tools to problems of aerosol generation and deposition in human lungs will be reviewed. The characteristic numbers and orders of magnitude for the main physical quantities will be discussed along with the available experimental techniques we can use to characterize the phenomena and to generate data to validate the simulations. Achievements and current limitations of the computational models will be illustrated.

ORAL COMMUNICATIONS

Quantitative method of mass assessment using tem grids for airborne micrometric particle exposure characterization

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Keywords | particle, mass metrics, microscopy

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Nanoparticles and submicrometric particles are often released from commercial consumer products, which could present risks over their fate to workers and the environment (Bressot et al., 2017; Svendsen et al., 2020). The work presented here aims to propose a new quantitative method to measure elemental mass concentrations via particle sampling and TEM-EDS (fig. 1). The principle is to collect airborne particles on a porous TEM grid, then add a certain mass of reference particles on it.

Results show that the absolute deviations between the theoretical elemental mass ratios and the experimental ratios remain lower than 8% (see table 1).

Using this technique, the size, shape, number, and composition of airborne particles collected on the TEM grid can be observed and analysed by TEM and EDS. Comparisons with the mass concentration deduce from calculation using mathematical models based on instrumentation results such as SMPS and APS, show the method consistency.

Compared with the previous methods, the present work proposes a specific characterization way for mass (concentration) of elements present in an unknown aerosol by collecting the tested aerosol and reference particles on one TEM grid and comparing the mass percentages of elements by EDS. Compared to the cumbersome on-line instruments, this new method appears to be very handy as only a short time of aerosol sampling in the workplace is usually needed.

Bressot, C., Manier, N., Pagnoux, C., Aguerre-Chariol, O., & Morgeneyer, M. (2017). Environmental release of engineered nanomaterials from commercial tiles under standardized abrasion conditions. Journal of hazardous materials, 322, 276-283.

Svendsen, C., Walker, L. A., Matzke, M., Lahive, E., Harrison, S., Crossley, A., Vázquez-Campos, S. (2020). Key principles and operational practices for improved nanotechnology environmental exposure assessment. Nature Nanotechnology, 15(9), 731-742.

Morphology of soot agglomerates from a diesel vehicle: speed and size effects

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Keywords | soot, vehicle, fractal dimension, prefactor, primary particles

The environmental impact of soot emissions from vehicles is manifested in its effect on the Earth's radioactive balance. This pollutant is the main absorber of visible solar radiation, and it is considered to be the second cause of global warming after CO2 (Ramanathan V. Carmichael V., 2008). Large and compact soot agglomerates have a higher single-scattering albedo than small and irregular ones (Kandilian, R., et al., 2015). Among morphological studies, most of them focus on the effect of the particle size on its optical properties, but not so many on the irregularity of agglomerates. In this work, in addition to the size (diameter of gyration and electrical mobility diameter), the fractal dimension, prefactor and number of primary particles constituting the agglomerates are also analyzed to fully characterize the particle irregularity. In this work, the morphological properties of the particles emitted by a Peugeot 3008 diesel vehicle are analyzed. The simulated vehicle speeds are within the ranges of the four stages of the WLTP certification cycle, using sixth gear for 120 km/h and 100 km/h (both for extra-high speed range), fifth gear for 70 km/h (medium speed range) and third gear at 50 km/h (low speed range), although, contrary to the WLTP cycles, the tests were made under stationary regime. Also, hot and cold idle tests were both performed. For each vehicle speed, the particles emitted were classified by size (50 nm, 85 nm, 140 nm, 215 nm, and 350 nm) with a Scanning Mobility Particle Sizer (SMPS), and collected with an electrostatic sampler (ESPnano sampler, with 3 mm copper grids). Figure 1a shows the consistency between the diameter of gyration results obtained from Transmission Electron Microscopy (TEM) images with electric mobility diameters, and Figure 1b the relation between fractal dimension and diameter of gyration. In this work, a relationship between both diameters and the number of primary particles forming the agglomerates is proposed.

Single Pass Tunnel Testing for Recirculating Virus Aerosol Control Technologies

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Keywords | aerosol control technologies; virus removal and inactivation; single-pass wind tunnel; airborne virus removal tests

A number of recirculating flow aerosol control technologies have been commercialized to mitigate aerosol-transmitted viral infections. Many of these technologies incorporate filters for particle collection and some may also incorporate technologies for virus inactivation, yet only tested to variable extents. Given the wide variety of commercially available aerosol control technologies to consumers, it is extremely important to develop standardized methods to characterize their performance in bioaerosol removal and inactivation, such that technologies can be compared on an "equivalent-test" basis. However, no standard procedures have been established for evaluating the effectiveness of bioaerosol removal and inactivation for recirculating aerosol control technologies. In this study, we propose the use of a single-pass tunnel for assessing the performance of bioaerosol control technologies. Single-pass wind tunnels can be completely sealed with well-controlled velocity and particle concentration profiles. Here, we specifically describe the construction of a single-pass wind tunnel and apply it to three recirculating aerosol control technologies, incorporating filters, electrostatic precipitators, and UV-C with a porcine respiratory coronavirus (PRCV) challenge aerosol, generated via pneumatic nebulization of a high titer (~10^7 TCID50 mL-1) viral suspension. Following guidelines similar to those used in the ASHRAE 52-2 test procedure for HVAC filters, in single-pass wind tunnel tests, the velocity and particle uniformity are first monitored across the cross-section of the tunnel. We show that the viable particle size distribution follows the volumetric size distribution of the nebulized virus-laden suspension, and that this distribution can be tuned to be similar in shape to the observed distribution from human respiratory activities. Following tunnel and virus aerosol characterization, for each tested technology, using triplicate tests, the single-pass log reduction based on RT-qPCR and viable virus titration is determined for each technology, simultaneously collecting virus aerosol particles upstream and downstream of the control technology. The tested technologies in this study have titration-based single-pass log reductions of virus viability in the 1.5-4.0 range. Overall, design and testing suggest that the single-pass wind tunnel approach may be a tractable method to examine the efficacy of a wide variety of aerosol control technologies in removing and inactivating viruses in aerosols.

Analyzing electrospray-generated particles by mobility classified mass

spectrometry

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While the analysis of sub-10nm particles is in many cases challenging by classical aerosol methods, mass spectrometry arises as a new opportunity of particle characterization in this size range, if particle charge levels are sufficient.

Electrosprays in the cone jet mode can be employed to produce sub-10nm particles with precisely tunable size and narrow size distributions at droplet generation rates of more than 109 per second for high conductivity liquids. The limited number of chemical particle compositions and the high charge level of the generated particles allow a most detailed analysis of the generated particles by mobility classified mass spectrometry. Particles up to around 10nm in diameter and masses up to 1MDa can be analyzed by this technology [1,2]. The high accuracy of the mass information (resolution 20.000) allows the determination of the chemical composition [2,3] and, in combination with mobility data, insight into structural features of ultra-small particles.

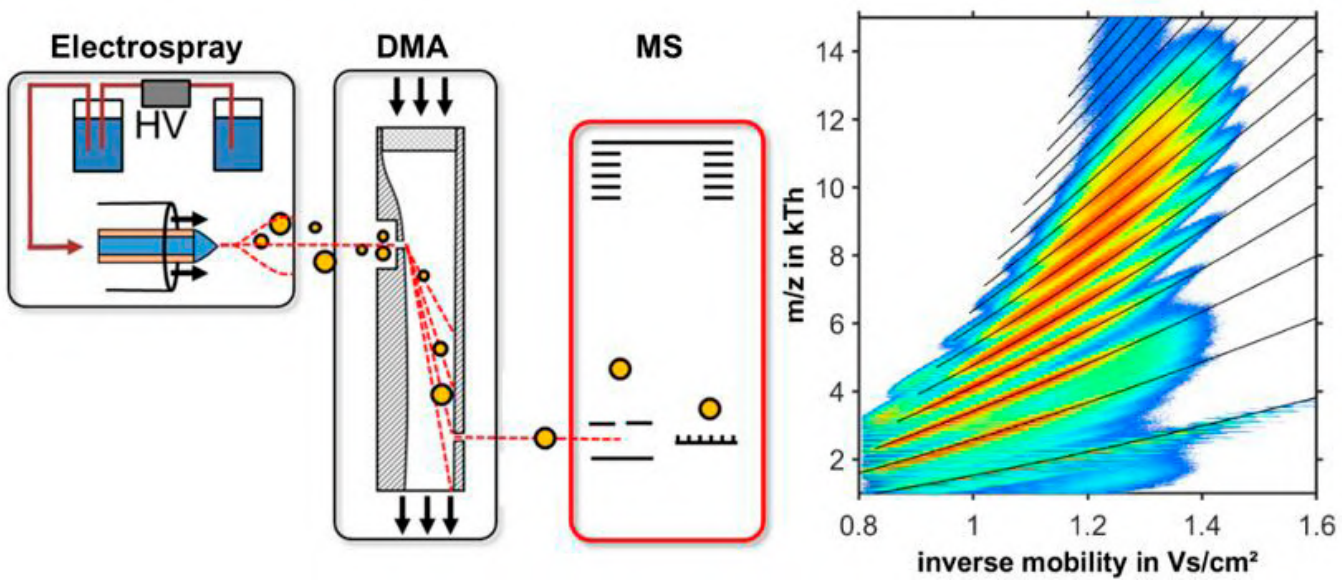


Figure: Schematic setup of the electrospray-DMA-MS setup and a heat map diagram for silver formate particles as generated by the electrospray. The black lines indicate tracks, which correspond to different charge states.

We demonstrate capabilities of the method for noble metal salt particles, which serve as starting point in an electrospray- pyrolysis process. We show a proper pathway for extraction of particle size distributions from mobility classified mass spectra, the dependence of particle size range accessible by DMA-MS as function of the particle material and the quality of information on the particle's chemical composition as function of particle size.

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Novel method for identifying evaporating, condensing, and reflecting atoms at the argon liquid surface in molecular dynamics simulations

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Keywords | Molecular dynamics, evaporation/condensation, refection, identification, liquid cluster.

We propose a novel approach for identifying evaporating, condensing, and reflecting atoms from the atomistic trajectories obtained from the molecular dynamics simulations. Rather than defining the liquid-vapour interface with the liquid and vapour boundary planes, the liquid phase in this approach is defined as atomic cluster, in which each atom is within the certain cut-off distance from one or more of the atoms of the cluster. The evaporation and condensation events are registered as the atoms leave or enter the liquid phase cluster, and the atomistic information is collected for each event. The reflection events are also registered when the vapour atom enters the liquid phase cluster and then leaves it back to the vapour phase after a short period of time. The liquid-vapour equilibrium molecular dynamics simulation of argon at 85 K temperature showed that the velocity distribution of surface-normal component of evaporation/condensing molecules deviates from the Maxwellian distribution of those molecules that cross the imaginary vapour plane parallel to the liquid surface. The number of reflecting molecules registered with the new approach is considerably lower, which consequently leads to approximately 47% greater value of condensation coefficient compared to the value evaluated with two-boundary interface method.

Overall, the proposed method improves over the shortcomings of the commonly used two-boundary interface method, such as the reflections of vapour atoms from the vapour phase molecules that take place far from liquid phase or the evaluated lower condensation coefficients values due to these types of reflections events. Furthermore, this approach could be used to investigate phase chance processes in simulations, in which the liquid drop is more complex than simple plane film, for example, in aerosol molecular dynamics simulations, where the evaporation/condensation take place simultaneously on many liquid drops/clusters.

Estimation of Shape Factors for fractal aggregates using Hydrodynamics Simulation

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Keywords | Dynamic shape factor, particle morphology, fractal

Introduction

The dynamics of aerosol aggregates, often described by fractal geometry, strongly depends upon their morphology (Wu and Colbeck, 2007) in addition to size. The shape factors account for the modifying effects of morphologies on the transport coefficients derived from the theories of regular spherical particles. In a recent study, the mobility characteristics of different

types of aggregates were examined over a wide range of fractal dimensions (d_f) using Stokesian Dynamics approach (Amalaruban et al., 2022). Herein we apply these results for estimating their dynamic shape factors.

Methodology

The dynamic shape factor $(/chi)$ is defined as the ratio of drag force on non-spherical particle (f) to the drag force on particle's volume-equivalent sphere (f_v),

$$/chi = /fracff_v = /fracR_mR_v \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where R_m and R_v are the mobility and volume-equivalent radii respectively. $R_v = N^{/frac13}a$, where N is number of monomers in an aggregate and a is monomer radius. As R_m is experimentally determined, we express $/chi$ in terms of R_m by eliminating R_v. Based on large number of simulations, Amalaruban et al. (2022) related R_m to the radius of gyration R_g through the formula $R_m = /betaR_g$, wherein the pre-factor $/beta$ was found to have monotonic

dependence on d_f. R_g is related to N via the mass scaling law $N = k_f(R_g/a)^{d_f}$ where k_f is a pre-factor. Upon combining these, Eq (1) may be recast as

$$/chi = /big(/frac/beta^{d_f}k_f/big)^{/frac13}/big(/fracR_ma/big)^{1-/fracd_f3} \ldots\ldots\ldots\ldots\ldots (2)$$

Results

As the results are valid in the continuum regime, we present the shape factors for large particles. For a typical cluster-cluster limited aggregate $(d_f = 1.76)$, the variation of shape factors with mobility radius for different monomer sizes are plotted in Fig.1.

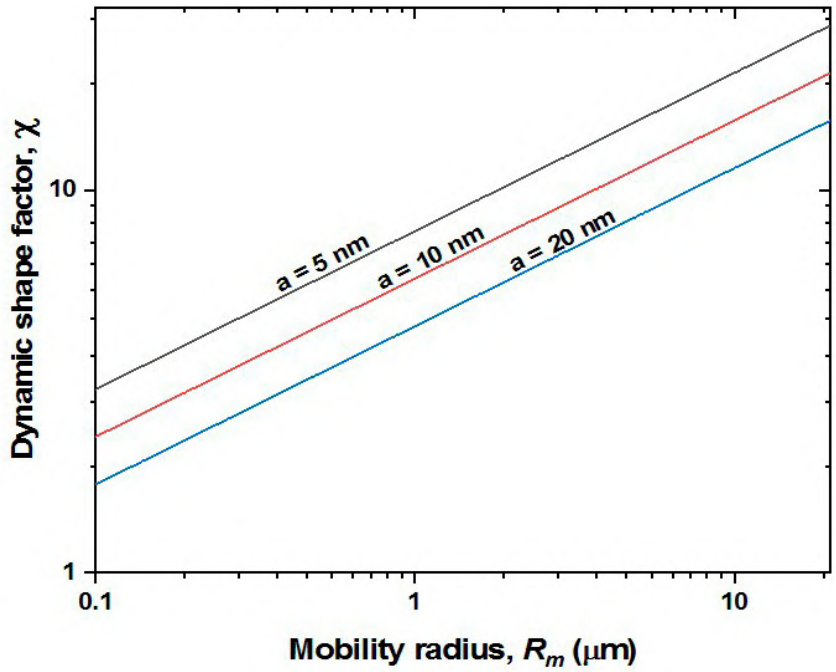


Fig. 1. Shape factor as a function of characteristic monomer radius for $d_f = 1.76$

The dynamic shape factor varies by a factor of 9 when the mobility radius changes from 0.1 μm to 20 μm. These size dependent shape factors can be implemented in the aerosol dynamics code in order to obtain more accurate information about the aerosol metrics.

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Dynamics of molecular collisions in air beyond the kinetic theory

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Keywords | molecular collisions, kinetic theory of gases, atomistic simulation, mean free path

Molecular dynamics (MD) unravel the fundamentals of major physicochemical processes dominating particle dynamics, thus facilitating understanding of natural phenomena and accelerating process scale-up and innovation (Mavrantzas & Pratsinis, 2019). Here MD simulations are used to explore the mechanics of molecular collisions in air in comparison with the kinetic theory (KT) of gases (Maxwell, 1860) which offers an elegant treatment of such collisions in gases, albeit under the assumption that molecules behave as hard spheres, and collisions are purely elastic. However, in gas synthesis of tiny nanoparticles (< 10 nm in diameter) at high temperatures or low pressures, one must accurately account for gas-particle collisions as self-preserving theory and asymptotic fractal-like dimensions cannot be attained (Kelesidis & Pratsinis, 2021). Hence the KT postulates are reassessed systematically with MD simulations where O2 & N2 are treated as true diatomic molecules.

The MD simulations are conducted in the NVT ensemble by employing a well-validated fully atomistic (FA) model. They are performed at 300 K and 1 atm and are thoroughly validated by comparing simulation predictions for the density, diffusion coefficient, and viscosity of air

with experimental and/or theoretical data.

When assuming air molecules as hard spheres, MD-calculated collision densities match perfectly those from KT. However, when the collision densities are computed with the FA model, these are significantly enhanced. A detailed analysis reveals that collisions from the FA model involve strong interactions between the colliding species. For example, many of these collisions are bimolecular; however, collisions between three and even four molecules are also observed. Moreover, colliding molecules can spend appreciable time together before departing in different directions. In contrast, in the simulations with the hard-sphere model, only two-body collisions are observed, and molecular encounters never occur. A new expression for the gas mean free path is obtained, more than 40% smaller than the 66 nm in all aerosol particle textbooks at these conditions.

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Kelesidis, G.A., and Pratsinis, S.E., 2021. Chem. Eng. J., 421, 129884.

Responsive nanostructured materials and devices against infections

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Keywords | nanoparticles, flame synthesis, aerosol deposition

Even though infections constitute the leading cause od deaths globally, there is only limited nano-related research against infections. Furthermore, because of the continuous use and abuse of antibiotics to fight infections, antimicrobial resistance in some bacterial strains (the so- called “superbugs”) has emerged. That constitutes the most serious public health threat today termed as “slow-motion catastrophe”. Therefore, there is an urgent societal need to provide innovative antimicrobial solutions as also highlighted by the WHO. Nanoscale materials offer advantages and solutions to this public health threat because they may exert antimicrobial action by multiple mechanisms rendering the emergence of antimicrobial resistance rather unlikely.

In this talk, I will highlight a few examples utilizing responsive nanomaterials against infections. This is explored using a nanomanufacturing process with proven scalability and reproducibility, flame aerosol technology [1], to assist rapid technology transfer to industry. We employ flame direct nanoparticle deposition on substrates and combine nanoparticle production and functional layer deposition in a single-step with close attention to product nanoparticle properties and assembly of devices [2]. For example, utilizing this technology, it is possible to develop nanomaterials as biosensors for physiological parameters (e.g. pH, H2O2) [3-4] relevant to bacterial infections or for food safety, as nano-enabled coatings on medical devices to eradicate bacterial biofilms [5-6], or even as drug nanocarriers for biologics (e.g. peptides, proteins) for localized treatments [7].

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Flame aerosol fabrication of robust SERS chemical sensors for food safety diagnostics

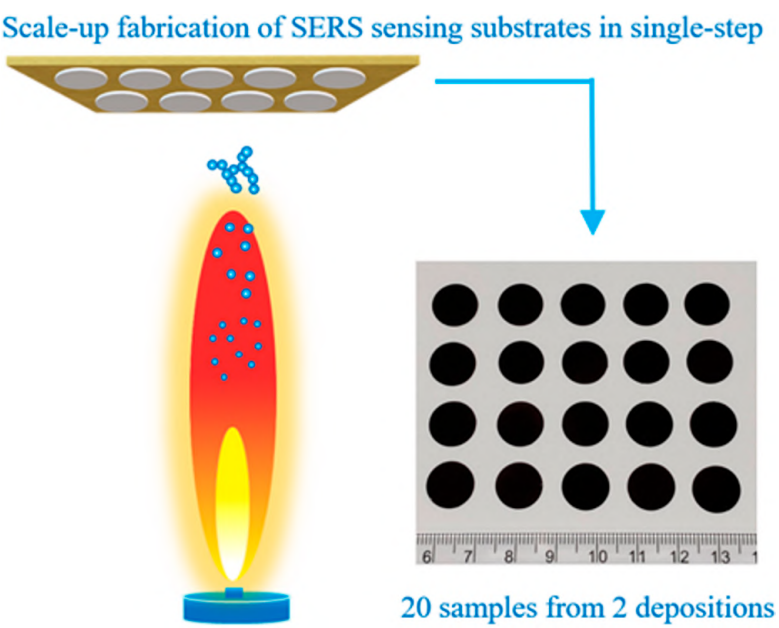
Li, Haipeng (1); Merkl, Padryk (1); Sommertune, Jens (2); Thersleff, Thomas (3); Sotiriou, Georgios (1)
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Keywords | Flame aerosol fabrication, surface-enhanced Raman scattering (SERS), chemical sensors, pesticide residues

Pesticides are widely used in modern agriculture to protect crops and food products from pests, diseases, and weeds. Human health problems are caused by pesticide residues through food contamination. For example, in 2018 around half of the food sold on the EU market contained pesticides, and 67% of fruits had pesticides (Authority 2020). Luckily, when the pesticide residue level is compliant with the legal limit, it is acceptable to human beings. Therefore, monitoring pesticide residues in food products are important for food safety and human healthcare.

Surface-enhanced Raman scattering (SERS) is a powerful surface sensing technique for monitoring pesticide residues, due to its advantages of label-free, fingerprint recognition, high sensitivity, rapid detection, and user-friendliness (Langer 2020). However, the commercialization of such SERS chemical sensors in food safety diagnostics is still limited by their sensing substrates due to high-cost and poor batch-to-batch reproducibility.

To address this challenge, we examine the economic and massive manufacturing of SERS chemical sensors using a highly scalable and reproducible flame aerosol technology, flame spray pyrolysis (FSP). The fabricated SERS sensing substrates have demonstrated good surface uniformity, high sensitivity, good batch-to-batch reproducibility, and good stability. The massive manufacturing of 150 samples can be achieved within 1.5 hours. The SERS substrates can be used to rapidly detect pesticide residues both on apple surface and in orange juice, offering a proof-of-concept of their promising application in food safety diagnostics (Li 2022).



Graphical abstract: Scale-up fabrication of SERS sensing substrates in one-step using flame aerosol technology. 10 samples were manufactured from one deposition within 40s.

Fundings from European Research Council (n° 758705), Swedish Foundation for Strategic Research (FFL18-0043) and Swedish Research Council (n° 2021-05494, 2016-05113) are kindly acknowledged.

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Li H., Merkl P., Sommertune J., Thersleff T., Sotiriou G.A., 2022. SERS hotspot engineering by aerosol self-assembly of plasmonic Ag nanoaggregates with tunable interparticle distance. Under review.

Silver nanoparticle based point of care diagnostic ammonia sensor

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Keywords | plasmonic nanoparticles, Ag, nanosilver, sensor

Ammonia is a biologically relevant analyte with strong links to metabolic health. Ammonia can exist in both its ionised form (NH4+), predominately found at physiological pH, and in the NH3 form. The term ammonia will be used to refer to the total content of both NH3 and NH4+. The removal of ammonia by the liver is an important regulation pathway for the control of ammonia levels in the body. In adult humans hyperammonaemia is defined as plasma ammonia levels >50 µM. The diffuse nature of hyperammonaemia, manifesting as symptoms from minor cognitive or behavioural changes to coma, makes an easy and accurate assessment of circulating ammonia levels essential (Häberle 2013). Enzymatic assays are often applied in well-equipped hospital clinical chemistry labs, however, due to the necessary equipment and specialised training their application in resource limited settings can be challenging. In the present study, the reaction of hypochlorite with plasmonic silver nanoparticles (AgNPs) is exploited. Addition of hypochlorite to the AgNPs causes their oxidative dissolution and thereby a strong decrease in optical plasmonic properties. However, in the presence of ammonia the hypochlorite reacts with ammonia and thereby does not dissolve the AgNPs retaining the plasmonic silver colour (Figure 1, insert). The AgNPs were applied as a dry powder but also deposited onto filter paper and used for ammonia concentration determination in a paper-based point of care format as shown in Figure 1. This project demonstrates the development of a paper-based point of care plasmonic silver diagnostic test for the naked eye detection of physiologically relevant ammonia levels in simulated serum.

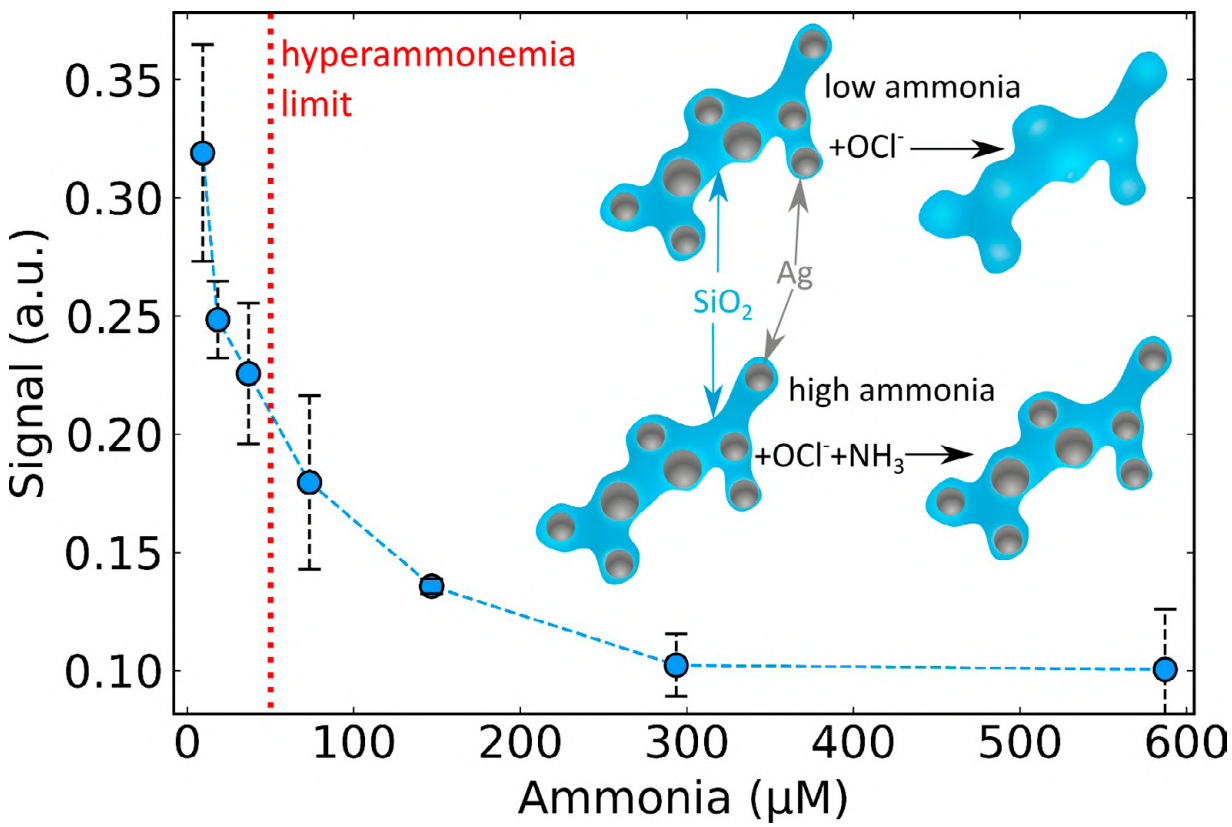


Figure 1: Ammonia sensor response of AgNPs deposited on paper. Dashed red line indicates 50 µM, the ammonia limit for hyperammonaemia in adults. Insert showing the mechanism behind the ammonia sensitivity.

This project has received funding from the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation program (ERC Grant agreement n° 758705). Funding from the Karolinska Institutet, the Swedish Research Council (2021-05494), and the Swedish Foundation for Strategic Research (FFL18-0043) is kindly acknowledged.

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Mass, size and shape of ultra-small particles: Pycnometry on the molecular level

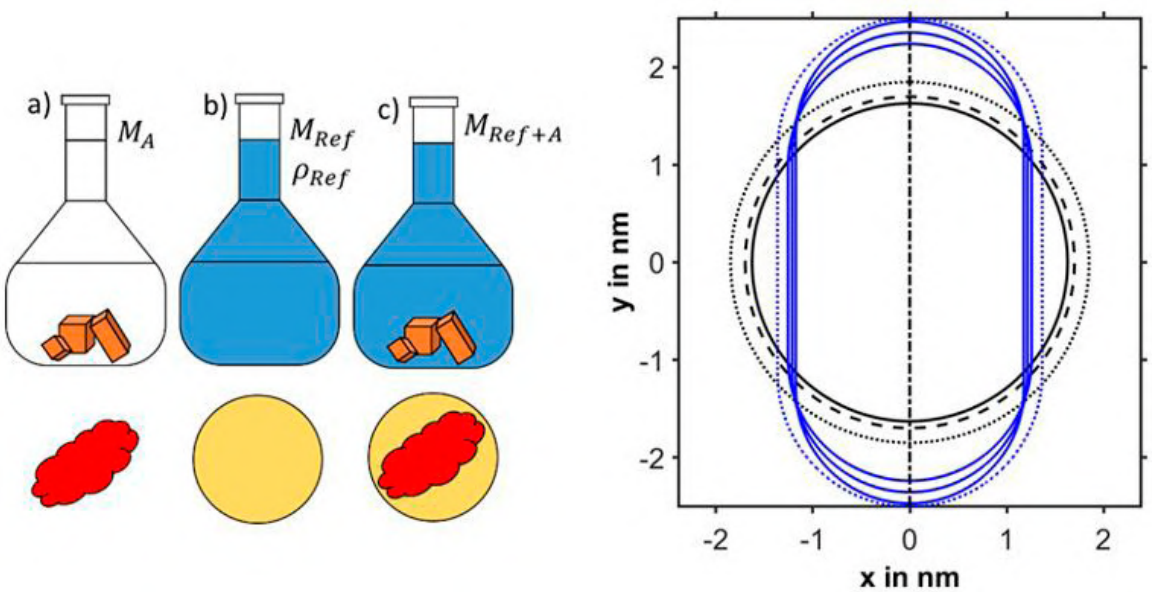
by mobility-classified mass spectrometry (DMA-MS)

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The multi-dimensional characterization of sub-5nm particles or macromolecules is quite a challenging topic. While modern SMPS systems are capable of measuring mobility diameter distributions down to 1nm, the correlation between the particle mobility and the particle geometry is ambiguous if the assumption of spherical particle shape is waved. The combination of a high-resolution DMA and mass spectrometry for analysis of the classified species has been successfully in the past for development of mobility theory and size analysis of clusters and small particles. Excellent agreement between theoretical predictions and measurement results is found for close to spherical particles [1]. However, for particles of unknown shape, the particle density must be known to derive information on the particle size from DMA-MS results.

Analyzing heterogeneous, spherical cluster ions by DMA-MS, allows inferring the particle density similar to classical pycnometry. The access to particle mass, density and mobility allows attributing a size and a shape factor to the particle under investigation. The high mobility resolution provided by SEADM's planar DMA (up to 100) and a mass resolution of 20000 for the mass analysis (Bruker qQTOF, Impact II) allows shape determination with high accuracy for ultra-small particles. Considering mobility peak resolution in data evaluation, allows additionally to give statements on the shape distribution.



Left: Pycnometer experiment and the cluster ion counterparts. Right: Properties, which can be attributed to a particle (here a lysozyme molecule) from a DMA-MS pycnometry experiment. Black solid line volume equivalent sphere, long dashed black line: mobility equivalent sphere, black short dashed line: collision surface of the of the mobility equivalent sphere. Blue solid lines: shape range attributed to the particle, assuming a cylinder with spherical endcaps as fundamental structure.

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Impact of Nonthermal Plasma Ionizer on Particulates from Combustion of Biomass

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Keywords | Biomass Boiler, Environment; Human Health; Nonthermal Plasma Air Ionizer; Particle size distribution; Particle number concentration

Topic: Impact of Nonthermal Plasma Ionizer on Particulates from Combustion of Biomass

Biomass is normally used for residential heating; it produces particulates that are harmful to human health and the environment. Though there are control techniques for large-scale applications, control of these emissions at the residential scale remains a problem. The aim

of this study is to investigate the usage of a novel non-thermal plasma air ionizer (NTPAI) technology for the control of particulates from small-scale (<25W) biomass boilers

In this study, a novel NTPAI was developed for particulates agglomeration arising from residential combustion of biomass. Firstly, the emissions from a working biomass boiler were characterized as functions of modes of operation. the particle size distributions (PSD) were determined as a function of operating conditions: cold start, de-ashing, warm start, steady-state, and shutdown modes. The PSD was measured using a Dekati "impactor", backed up by scanning electron microscopy (SEM). The NTPAI was evaluated at plasma powers of 0, 7.5, 15, 25, 35, and 45W for all biomass boiler modes. The NTPAI showed a significant impact on particle agglomeration as the percentage particle mass concentration of submicron and fine particles decreased, resulting in an increase in the percentage particle concentration of coarse and large particles sizes. Furthermore, the overall percentage reduction in mass concentration at these modes was 47, 50, 22, 40, and 52% at cold start, de- ashing, warm start, steady-state, and shutdown respectively at low NTPAI power. The impact of NTPAI was investigated at steady-state mode and showed an increase in particle number concentration by 75% among coarse and large particle sizes as ultrafine particles and fine particles were reduced.

The present study has demonstrated that the NTP air ionizer increases the particle size of the combustion biomass particulates thereby making them less harmful to humans and removable by current technologies, such as cyclones and electrostatic precipitators which cannot remove submicron particles.

Sand impact on snow albedo

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Keywords | snow covers, optical properties, sand, contamination, soot, mineralogy

Snow covers are greatly affected by the particles that are deposited on their surface (Flanner et al., 2021). Sand is one of the components of the Saharan haze and it interacts with radiation absorbing and scattering in the visible and infrared spectra (Balkanski et al., 2007) and its environmental and climatic impact depends on its composition, size, shape, and crystalline structure of the particles (Formenti et al., 2011). In this work, an experimental campaign was carried out in Sierra Nevada (Granada, Spain), in which the optical effect of different contaminating particles on the snow covers was measured using a field spectroradiometric system composed of three upwelling spectroradiometers and three downwelling ones. Sand collected from a Mediterranean beach (Spain) was employed for contaminating the snow. It was analyzed with X-ray diffraction obtaining the mineralogical composition: silicon oxide (54.51%), calcium carbonate (34.25%), acetoguanamine (7.4%) and dolomite (3.85%). With this composition the refractive index of the sand was weigh-averaged with the Lorentz- Lorentz effective medium model, considering the refractive indices of the components although ignoring the acetoguanamine, because no absorption coefficient was found for this material. After measurements, snow samples were taken from the measurement point, filtered, and weighted, showing that the snow surface was previously contaminated with soot (0.5 ppm), due to the existence of a nearby road. Snow albedo was calculated with the model of Wiscombe and Warren (Wiscombe, W.J., Warren, S.G., 1980) and the optical properties for sand were calculated with the Mie theory (using a radius of 250 μm for the snow grain and 100 μm for the sand grain). Furthermore, optical properties of soot agglomerates were calculated with the with the Rayleigh-Debye-Gans approximation and using the correction for multiple scattering proposed by Mountain and Mullholland (Mountain, R.D., Mullholland, G.W., 1988). The experimental and modelled results of the snow albedo are shown in Figure 1, where it can be observed that contaminating with sand (2000 ppm) causes a decrease in the snow albedo in the visible range whereas in the infrared range (contrary to the effect of soot) it increases the albedo due to the reflectivity of this material.

Porous NiO Prepared by Flame Spray Pyrolysis for 80wt%Ni- CeO^2 Catalyst for CO^2 Methanation

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(1) Japan

Keywords | Ni catalyst, CeO2, CO2 methanation, Inverse catalyst

Higher Ni loading is a straightforward strategy for increasing the Ni surface area which is desired for catalysis applications. However, the

optimal loading is essential, because excessive loading induces the growth of Ni particles. To overcome the limitation, small CeO2 particles were impregnated on porous NiO nanoparticles prepared via flame spray pyrolysis. The specific surface area and total pore volume of NiO were 251 m2/g and 2.3 cm3/g, respectively. The high porosity and surface area of the NiO allowed the deposition of small CeO2 particles (~5 nm) by the impregnation of cerium acetate monohydrate.

The particles were reduced using 5%H2 at 500 oC for 1 h, which converted NiO to metallic Ni. During the reduction, the growth of Ni particles was hindered by CeO2 particles. Consequently, the Ni size was relatively small (~20 nm) despite the extremely high Ni content (80 wt%), as observed by scanning transmission electron microscopy. In contrast, incorporation of Ni using nickel acetate tetrahydrate into FSP-made CeO2 support resulted in the formation of inhomogeneous Ni particles (20~100 nm) after H2 reduction. H2 chemisorption measurement showed the surface area of Ni particles in the former catalyst was 13.7 m2/g, 2.4 times larger than in the latter catalyst. The former catalyst exhibited remarkable performance for CO2 methanation (47% CO2 conversion at 250 oC), 2 times higher than in the latter catalyst.

(Fujiwara, K. et al., J. Jpn. Pet. Inst. 2021, 64, 261-270.)

FLASH COMMUNICATIONS

A CFD-DEM model for predicting the influence of nasal hair on the air-particle dynamics in nasal cavity

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Keywords | CFD, DEM, Nasal Hair, Particle Deposition, Nasal Cavity

Computational fluid dynamics (CFD) has been widely employed for simulating the airflow and inhaled particle dynamics in the nasal cavity. Specifically, the multiphase flow model (i.e., the Euler-Lagrange model) is emerging as a powerful digital tool in otolaryngology, which significantly contributes to the advancement of fundamental understanding and the enhancement of clinical treatment outcomes for many upper airway diseases, including nasal airway obstruction, chronic rhinosinusitis, empty nose syndrome, and laryngotracheal stenosis. However, existing CFD research efforts are not able to simulate the fluid-structure interactions between the nasal hair and the air-particle flow. Instead, as a simplification, they neglected the presence of nasal hair. Thus, the error induced by this simplification on the prediction of inhaled airflow fields and particle deposition distribution is unknown. To fill the knowledge gap and create a more physiologically realistic computational nasal cavity model, this study developed a coupled CFD-discrete element method (DEM) model to simulate the nasal hair motion and quantify its influence on the inhaled airflow and particle flow dynamics in a virtual nasal cavity geometry. Specifically, utilizing the non-spherical particle model in the discrete element method (DEM), each nasal hair is treated as a flexible fiber, planted on the inner wall of the nasal cavity. Two simulation cases were performed using the CFD- DEM model, with and without the presence of nasal hairs. The inhalation flow rate through the nostrils is 3.96 L/min, mimicking the human breathing condition at rest. Monodispersed particles (10 μm in aerodynamic diameter) were inhaled with a mass flow rate of 1.6 mg/s. Simulation results indicate that the nasal hair can enhance the particle filtration by trapping 5.5% of the particles. The presence of nasal hairs has reduced the particle deposition in the nose by 1.5%. The more physiologically realistic CFD-DEM model with the capability of modeling the nasal hair motion can be employed as the next-generation in silico tool for modeling the biofluid dynamics in the nasal cavity.

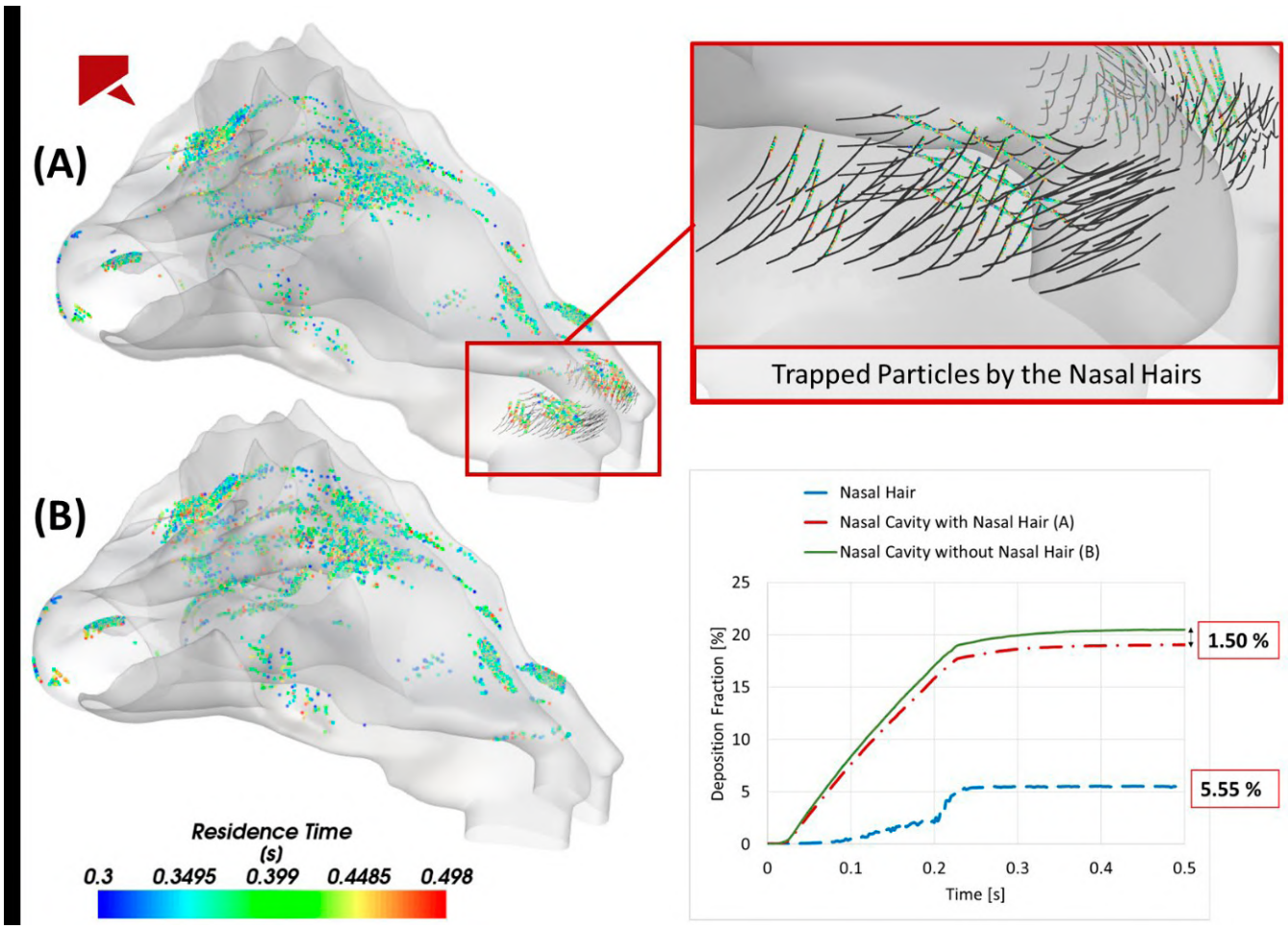


FIGURE 1. Comparison of particle deposition in the human nasal cavity with (A) and without (B) the presence of nasal hairs.

Engineering of flame-made plasmonic-semiconducting nanocatalysts: A study of the photo-induced carrier dynamics and interfacial electron transfer

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(1) University of Ioannina

Keywords | flame spray pyrolysis, hot electrons, plasmonics, photocatalysis

Plasmonic photocatalysts have been emerged as an innovative pathway towards the efficient harvesting of solar energy in order to amplify the performance of chemical processes of high technological importance, such as H2 production and CO2 reduction. Exploiting the plasmon-induced, highly energetic "hot" electrons and their sufficient injection in a semiconductor is the key-parameter for enhanced photo-activity (Brongersma et al., 2015). However, the interplay of the plasmon-driven mechanisms and interfacial charge transfer in such nanostructures is not fully understood. Furthermore, despite quantum size effect in semiconductors is well documented, its effect in the interfacial electron transfer and the photo-induced carrier dynamics in a plasmonic-semiconducting junction have not been thoroughly investigated.

In this context, Ag/TiO2 and Ag/NaTaO3 heterostructures of various compositions and particle size are developed using flame spray pyrolysis (FSP) technology (Pratsinis et al., 2002). Exploiting FSP flexibility, various setups have been employed for engineering the synthesis of TiO2 quantum dots and the smaller particle size reported for NaTaO3 nanostructures so far, as well as for controlling the Ag particle size (Fujiwara et al., 2014). Using electron paramagnetic resonance spectroscopy (EPR) and electrochemical impedance spectroscopy (EIS), the photo-generated carriers, hot electrons and interfacial electron transfer have been studied respectively. The observed "trapped" electrons in EPR indicates that quantum size effect and, thus the increased surface electron population in TiO2, is correlated to increased photocatalytic

yields. Furthermore, depending on the FSP setup where the Ag particles are formed and deposited on the semiconductor, the observed hot- electron population and photocurrent is directly affected, indicating an interplay between the size of the plasmonic and semiconducting particles and the electron stimulation and transfer in the junction.

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Development of reference materials for carbonaceous aerosol measurement

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Keywords | Carbonaceous aerosol, reference materials, aerosol generation

Carbonaceous aerosol, including organic carbon (OC) and elemental carbon (EC), has significant influence on human health, air quality and climate change. Accurate measurement of carbonaceous aerosol is essential to reduce the uncertainty of radiative forcing estimation and source apportionment, which could be beneficial to national and global control of air pollution and greenhouse effect.

Carbonaceous aerosol is typically measured by the thermal/optical analysis (TOA) method, in which the sample filter is heated in inert and oxidizing atmospheres to separate OC and EC. However, the accurate separation of OC and EC is difficult and can be influenced by multiple conditions, such as the charring of OC, the different temperature protocol and optical technique. Therefore, the development and application of reference materials (RM) for carbonaceous aerosol measurement, especially for the validation of OC/EC separation, is an important basis for further study of carbonaceous aerosol. Previous RMs were mainly based on ambient air sampling, such as RM 8785, which could not provide traceability of OC and EC concentration and the difference of OC/EC between two protocols could reach 60%. To develop traceable RMs with known OC/EC contents, our study applied an improved aerosol generation and mixing technique, which could provide uniform deposition of OC and EC on quartz filters. The traceability of the RMs was realized by the gravimetric method. To generate OC aerosol with similar pyrolytic property of real atmosphere, both water soluble organic carbon (WSOC) and water insoluble organic carbon (WIOC) were used. Moreover, different potential candidates for EC have been tested and the best type was identified as amorphous carbon. After generation, the filters were distributed to laboratories using different instruments and protocols. The homogeneity within the 47mm filter was validated, which could reach below 2%. The comparison of total carbon (TC), OC and EC results showed good correlations, with relative standard deviations within 10%. The results indicated that the newly developed RMs were acceptable for the calibration and validation of OC and EC, which could improve the accuracy of carbonaceous aerosol measurement.

8 NANOPARTICLES: PRODUCTION, CHARACT. AND APPLICATIONS

KEYNOTE LECTURES

Development and characterization of hydrophobic nanocellulose particles throught esterification reaction as additive in coating formulations

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Keywords | nanocellulose; cellulose nanofibers; alkyl ketene dimer; coating; water contact angle; Fourier transform infrared spectroscopy; thermogravimetric analysis; scanning electron microscopy

Most paper and board grades, especially for food packaging, need to be resistant to polar liquids such as water, aqueous solutions and suspensions. However, a sheet of paper composed only of cellulosic fibers is water-absorbent. Therefore, paper industry already uses some additives, such as alkyl ketene dimer (AKD), into pulp suspension (internal sizing) or coating (surface sizing) to improve paper resistance. However, AKD is unstable in water, and some of it may undergo hydrolysis in water to form unstable β-keto acids which provide less hydrophobicity [1].

Nanocellulose (NC)-based products provide renewable, technical, and sustainable benefits to packaging materials, enhancing paper properties and open the opportunity to development high performance food packaging [2]. Recent studies have pointed out that the combination between NC and AKD, as two independent layers, increase the hydrophobicity of paper surface but do not provide enough resistance to water [3].

Therefore, in this study esterification reactions will be conducted to develop a new modified NC by means of the incorporation AKD. Two different NC, produced by mechanical and chemical pretreatments followed by high-pressure homogenization, were studied. Both NC were freeze-dried to remove the water in the NC suspension, and, thus reducing the side-reactions between AKD and the water present in the NC structure.

Reactions with different ratios of lyophilized NC and AKD were carried out using ethyl acetate as solvent in the presence of a small amount of 1-methylimidazole used as an esterification promoter. After adding the catalyst, the suspensions were homogenized using an Ultra-Turrax micro-homogenizer and, finally, heated at high temperature (70 and 100ºC). Several methods, including Fourier transform infrared spectroscopy, thermogravimetric analysis, scanning electron microscopy and water contact angle determination, were used to characterize the hydrophobized-NC samples. Our results suggest that the modification of NC particles with AKD could significantly improve the hydrophobicity of coating formulations.

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Unique approach for manufacturing Li-ion battery electrodes

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Unique approach for manufacturing Li-ion battery electrodes Suman Pokhrel1,2, Michael Gockeln3, Lutz Mädler1,2
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High power density, high efficiency and long cycle life are the basic properties of rechargeable lithium-ion batteries (LIBs) making them potential candidates as power sources for the electronic consumer market. Due to their high production cost, the extended use of these batteries in electromobility and stationary energy storage devices are still challenging. To overcome this drawback, innovations in process design rules and automation engineering of large-scale LIB production lines are important for cost efficiency. We present an alternative electrode fabrication using double-flame spray pyrolysis and layer compaction using lamination to outperform the conventional doctor blading technique and meet the industrial requirements of LIB cost efficiency. In this unique approach, the electrode fabrication procedure involves using two independent flame configurations where one flame produces the electrode material such as Li4Ti5O12 and the other independent flame produces carbon.

While two flames are inclined at 20°, two independent particle streams mix at a certain distance above the flame and deposit on the particle collector. The physicochemical properties of the particles used for electrodes are determined using XRD and N2 adsorption. The carbon content in the sample is investigated using TG analysis and Raman spectroscopy. The particles deposited on the collecting unit is transferred on a conductive substrate using lamination. The new electrode fabrication strategy avoids (1) use of organic solvents and binders for paste fabrication (2) significant economic burden. The data showed flame made Li4Ti5O12/C-composite anodes have enhanced specific discharge capacities compared to reference electrodes prepared by conventional slurry-based doctor blading. In addition, the variations in lamination pressure resulted in significant differences in electrochemical performance. While optimization of the material design including particle size, carbon quality, porosity and electrode design might improve the capacity retention in liquid-electrolyte LIBs during extensive cycling, the long-term research goal is to assemble a complete all-solid-state cell using electrodes manufactured via flame-lamination technique.

Drug nanoparticle seeded desupersaturation and dissolution tests for development of robust amorphous solid dispersions

Bilgili, Ecevit (1); Guner, Gulenay (1); Amjad, Ayesha (1); Berrios, Matthew (1); Kannan, Manisha (1)
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Keywords | drug nanoparticles, wet stirred media milling, amorphous solid dispersions, recrystallization, dissolution enhancement, poorly water-soluble drugs

Amorphous solid dispersions (ASDs), wherein a drug is molecularly dispersed in a polymer, have been used for enhancing the dissolution rate of poorly water-soluble drugs. Unfortunately, ASDs are thermodynamically metastable: residual nanocrystals may form during processing/storage and the drug may recrystallize during the dissolution of the ASD in a liquid medium. Although the potential impact of residual crystallinity on drug release from the ASDs has been studied recently (Moseson et al., 2020*), drug nanoparticles have not been used as seeds. We aim to utilize nano-sized seeds (nanoseeds) of a model poorly soluble drug, griseofulvin (GF), to emulate the impact of residual crystals on the supersaturation maintenance of GF with three polymers: Soluplus (Sol), Kollidon VA64 (VA64), and hydroxypropyl methyl cellulose (HPMC) along with an anionic surfactant. ASDs were prepared by spray-drying organic solutions of GF–polymer–surfactant.

Nanoseeds were produced by wet stirred media milling and used in the dissolution–desupersaturation tests. DLS, SEM, and XRPD/DSC were used for characterization.

When added to a supersatured GF solution, obtained via a solvent-shift method, GF nanoseeds led to a higher extent of desupersaturation, via recrystallization of the dissolved drug, than the micron-sized GF seeds (Fig. 1). This could be attributed to the higher surface area and number concentration of the nanoseeds as compared with the micron-sized seeds. The use of micron-sized crystals underestimates the impact of residual nanocrystals, which could be quite misleading when formulators attempt to develop robust drug ASDs. Sol was found to be the only polymer that prevents GF nucleation in the absence of seeds; whereas HPMC and VA64 had limited inhibition capability for GF nucleation–crystal growth. None of the polymers could prevent GF recrystallization when nanoseeds were present; a higher seed loading promoted GF recrystallization (Fig. 1). These results agree with those from the dissolution tests. Overall, this study suggests that wet- milled drug nanoparticles can serve as surrogate for residual nanocrystals in ASDs.

*Moseson, D.E., Parker, A.S., Beaudoin, S.P., Taylor, L.S., 2020. Amorphous solid dispersions containing residual crystallinity: influence of seed properties and polymer adsorption on dissolution performance, European Journal of Pharmaceutical Sciences, 146: 105276.

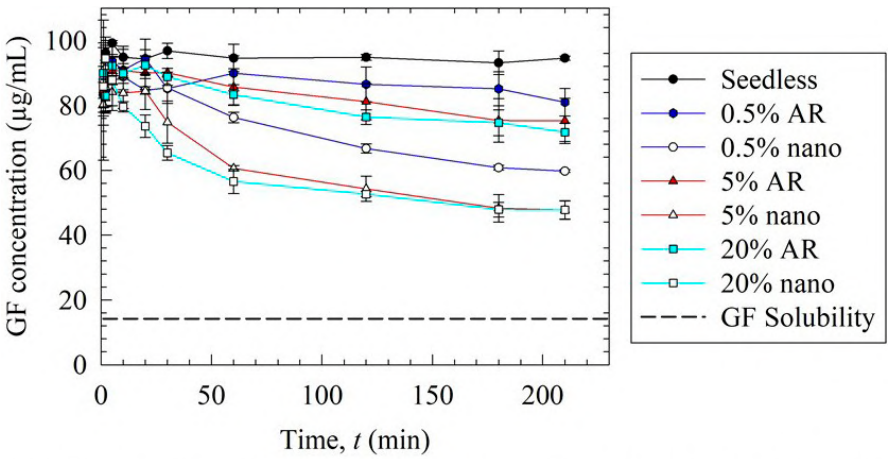


Fig. 1. Effects of seed size and loading during a desupersaturation test (solvent-shift method) performed on an ASD formulation with 1:3 mass ratio of GF:Sol. Drug nanoseeds were obtained from wet stirred media milling, while the micron-sized GF particles were used as-received (AR) seeds.

ORAL COMMUNICATIONS

Sintering rate of nickel nanoparticles by molecular dynamics

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(1) Carleton University, (2) The University of Melbourne

Keywords | Nickel Nanoparticles, Crystallinity, Sintering Rate, Molecular Dynamics

Nickel nanoparticles (Ni NPs) are widely used in batteries, catalysts, and filters. Properties of Ni NPs strongly depend on their crystal structure and morphology quantified by the state (i.e., solid, transient, or liquid phase) of primary particles (PPs), hard-agglomerate (aggregate) and PP size. The growth rate of PPs during gas-phase synthesis is determined by their sintering rate, which is sensitive to the state of PPs. At low temperatures and for large PPs, diffusion of atoms in PPs controls solid state sintering (**Zachariah & Carrier, 1999**). However, with increasing temperature or decreasing PP size, the state of particles changes and viscous flow sintering becomes dominant (**Friedlander & Wu, 1994**).

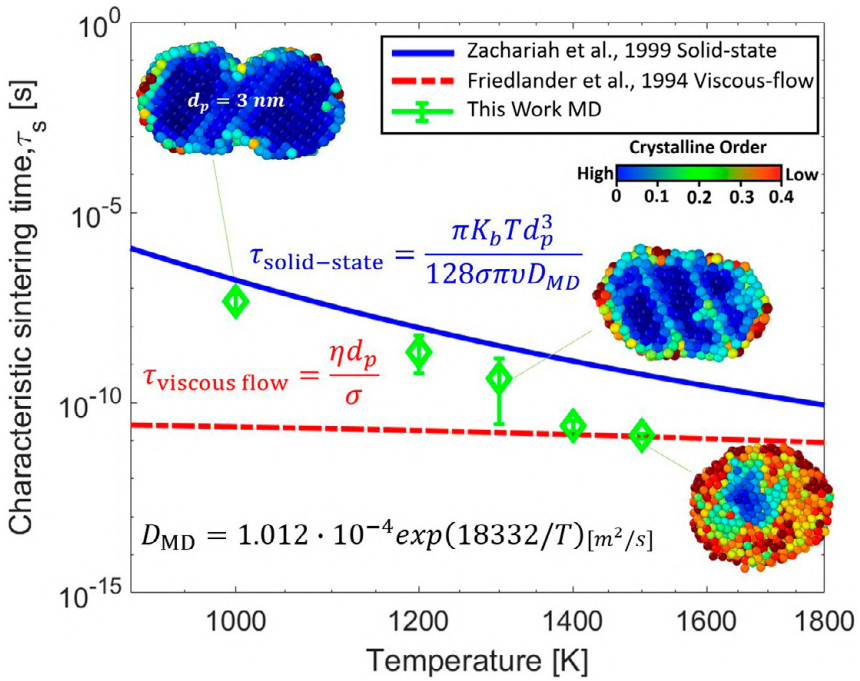


Figure 1: Characteristic sintering time of a Ni dimer with dp = 3 nm, as a function of temperature simulated by MD (diamonds) and predicted by viscous flow sintering (dashed line) and solid state diffusion (solid line) using MD-derived solid- state diffusion coefficient, D.

Using molecular dynamics (MD), the crystallinity and sintering of Ni NP dimers are investigated. Accuracy of the employed force field is validated by reproducing measured melting temperature of Ni and its X-ray diffraction patterns (**Donegan et al., 2012**). For large PP diameters (dp ≥ 5 nm) at low temperatures (T < 1400 K), MD-derived characteristic sintering times based on the evolution of dimer specific surface area are in excellent agreement with characteristic sintering times predicted by solid state diffusion theory using MD-derived diffusion coefficients (see Fig. 1). With decreasing dp or increasing T, the state of PPs quantified by their disorder parameter changes due to progressive melting starting from their surface that results in significantly lower characteristic sintering times compared to those predicted by solid state diffusion. A sintering rate for Ni NPs has been extracted that is valid for all particle states. The performance of this rate coupled with a monodisperse model (**Kholghy & Schumann, 2021**), in predicting the evolution of Ni agglomerate morphology quantified by its mobility and primary particle diameters during gas phase sintering in a flow reactor (**Tsyganov et al., 2007**) is demonstrated.

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Microstructural stability enhancement of high-temperature composite phase change materials using nanoparticles

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Keywords | Surface tension, Solar salt, Nanoparticles, Composite phase change materials, Thermal energy storage

Composite phase change materials (CPMCs) consist of at least two components, a phase change material (PCM) for storing thermal energy, and structural material for shape stabilisation. CPCMs for thermal energy storage (TES) have attracted significant interest in recent decades due to some salient futures including high energy density, good high-temperature stability, and availability of PCMs for a wide working temperature range. CPCMs, however, often have a PCM load below ~60% to prevent leakage when PCM is at liquid state, particularly for high-temperature applications. This work explores one potential measure to address this challenge by adding nanoparticles to the PCM. The hypotheses lie in the modification of the PCM viscosity and wettability, which could significantly reduce the leakage and enhance the CPCM microstructural stability. However, most reported experimental studies on inorganic salt-based suspensions are limited to heat capacity and thermal conductivity measurements, with less viscosity and little on wettability.

We investigated the effect of the addition of nanoparticles (0-2.5 wt. %) on the surface tension of Solar Salt (SS) on a MgO{1 0 0} surface. At 0.5 wt%, the surface tension is found to increase by 2.45 %, and 11.8% with 2.5 wt. %. Our work also shows that it decreases linearly with an increase in temperature, but in the presence of the nanoparticles, decreases up to 300 ◦C, followed by an increase up to 400 ◦C and then a decrease at 450 ◦C. Close to the melting point, the contact angle increases with increasing nanoparticle addition, and a fully wetting condition is reached at 390◦C, independently of concentration.

With the above understanding, we prepared CPCMs using SS as the PCM and MgO as the structural material, with and without 1.0 wt % SiO2. The prepared CPCMs were kept at 390 ◦C 5h. The CPCM with nanoparticles showed almost no leakage, an improved density (by 8.9%), porosity (by 18.9%), and reduced pore size. Thermal diffusivity increased by 6.5%. These show the relationship between nanoparticles addition and CPCM performance due to surface tension and wettability modification. Thus, an implication on the CPCM fabrication due to microstructural changes.

Characterisation of magnesium hydroxide from highly concentrated MgCl2 solutions

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Over the past years, a growing interest has been attracted by the peculiar characteristics of nanoparticles. Nanoparticles have been used in a wide variety of fields, e.g. medical and biomedical, catalysis, polymer science and wastewater treatment. In this context, Mg(OH)2 nanoparticles have been exploited as smoke suppressing flame retardant filler in polymeric matrices. Mg(OH)2 can be extracted from minerals or recovered from Mg-containing solutions, as seawater brine and saltworks bitterns, via precipitation. In the precipitation operation, a high reactant mixing degree must be guaranteed in order to produce nanometer-sized particles. In the literature, T-mixers have been typically employed in precipitation to ensure low mixing times, e.g. ranging from 1 to 10 ms (Battaglia et al., 2022).

In the present work, magnesium chloride and sodium hydroxide solutions were employed to produce Mg(OH)2 in a 2 mm circular cross-sectional T-mixer. Five Mg2+ concentrations were investigated mimicking the magnesium content in seawater brine and bitterns (from 3 to 24 g/l). NaOH solutions in stoichiometric amounts were employed as precipitants.

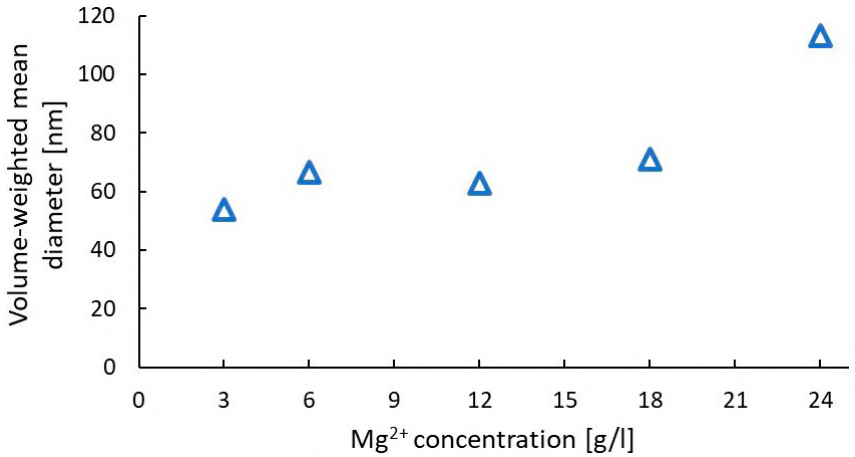
Particle size distributions (PSDs) were measured using a Zetasizer Nano ZSP analyzer with ultrasound treatment and the addition of a dispersant. Volume-weighted mean diameters of the obtained PSDs ranged between 50 nm and 120 nm, as reported in Figure 1, thus demonstrating the possibility of producing Mg(OH)2 nanoparticles from concentrated Mg2+ solutions employing T-mixers.

This project has received funding from the European Union's Horizon 2020 Research and Innovation Programme under Grant Agreement No. 869467 (SEArcularMINE). This output reflects only the author's view. The European Health and Digital Executive Agency (HaDEA) and the European Commission cannot be held responsible for any use that may be made of the information contained therein.

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Figure 1 Mg(OH)2 particles volume-weighted mean diameters as functions of Mg2+ concentrations



Classification and characterization of gold and silver nanoparticles by size-exclusion chromatography

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Keywords | Nanoparticles, Chromatography, Classification, Characterization

Size-exclusion chromatography (SEC) is a well-established and scalable chromatographic technique used for the determination of molecular weight distributions of polymers as well as for the separation of molecules and polymers according to their hydrodynamic size in solution. It is also a highly promising technique for property classification of various nanoparticle systems (Süß et al., 2018; Süß et al., 2020).

We herein report the classification and characterization of 11-mercaptoundecanoic acid stabilized gold and silver nanoparticles by SEC, where (attractive) interactions between nanoparticles and the stationary phase have been strictly avoided. In SEC, the separation is solely governed by the size-dependent diffusion of nanoparticles into the pores of the stationary phase. The left panel of Figure 1 shows serial injections of nine gold nanoparticle dispersions with different sizes (5 nm – 80 nm). The separation is clearly dominated by a size-exclusion mechanism since retention volumes increase with decreasing particle size. Furthermore, baseline separation is achieved even for multimodal nanoparticle mixtures as shown in the right panel of Figure 1. Remarkably, multimodal particle size distributions can be characterized accurately over a broad size range with the aid of a calibration curve, which is determined from the hydrodynamic diameters and respective retention volumes. The transfer of these results to silver nanoparticles is straightforward.

Our results demonstrate the large potential of size-exclusion chromatography for the classification and characterization of various types of nanoparticles on the analytical and preparative scale.

Figures

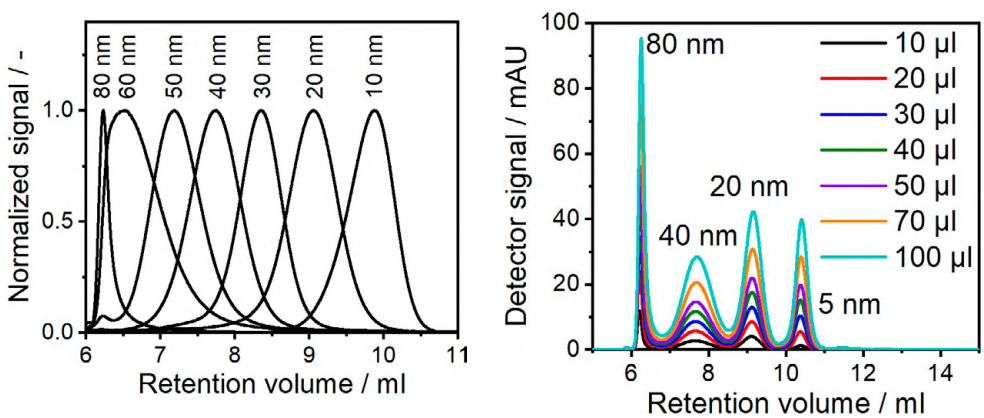


Figure 1: Chromatograms of serial injections of gold nanoparticles with different sizes (left). Chromatograms of a mixture of different gold dispersions for various injection volumes (right).

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Size and composition controlled synthesis of silver gold alloy nanoparticles

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Keywords | Particle Synthesis, Nanoalloys, Nanoparticles, Particle Characterization, Silver gold alloy nanoparticles, Product design, Metal nanoparticles

Research in the field of plasmonic nanoparticles (NPs) has received increasing attention in recent years. Particles in this size range show interesting electrical, catalytic and especially optical properties. Due to these properties, plasmonic NPs find application in a variety of research areas, including nanosensing (Han et al., 2001) and nanocatalysis (Ellert et al., 2014). Silver (Ag) / gold (Au) alloy NPs (ANPs) are of special interest, not only due to their use in above-mentioned applications, but also as a model system to study the formation of alloy NPs, as both elements are fully miscible. For a targeted product design, the precise control of the size, shape and chemical composition of the produced NPs is imperative.

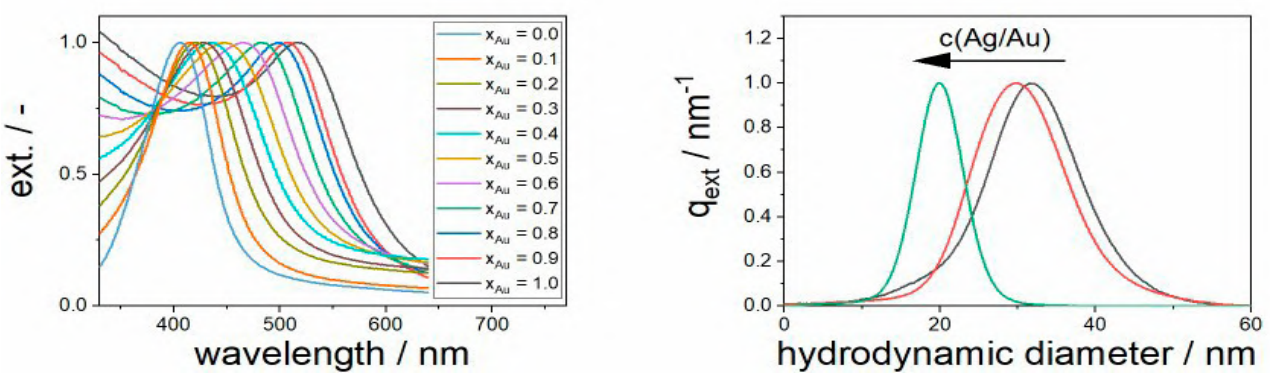


Fig. 1: Normalized extinction spectra of Ag-Au ANPs with varying compositions at constant particle size (left) and particle size distributions of Ag-Au ANPs with a molar gold content of 50 % (right).

In this work, we demonstrate how the chemical composition of the produced Ag-Au ANPs can be precisely adjusted through the precursor salt ratio (fig. 1). Further, we show how the size of the resulting particles can be altered via different mechanisms, including the synthesis conditions, in particular the strength of the reducing agent, and seeded growth. Furthermore, the 2D distributions in terms of size and composition is evaluated, which presents a measure for the quality of the resulting particles. Finally, the particles are comprehensively characterized via multiple methods, including size exclusion chromatography (SEC), transmission electron microscopy (TEM), multi-wavelength analytical ultracentrifugation (MWL-AUC) and extinction spectroscopy.

Current research goes towards the complete exclusion of human experimenters via automated synthesis platforms to increase reproducibility across different experiments (Salaheldin et al., 2017) and enable high throughput experiments.

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Liquid chromatographic separation of carbon dots

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Keywords | Carbon Dots, Separation, Chromatography

Carbon dots (CDs) are fascinating nanoparticles, which combine high fluorescence quantum yields (QY) with good biocompatibility. In literature it was already proposed that CDs consist of a carbon core and highly fluorescent fluorophores which are supposed to be attached to the surface of the CDs (Hinterberger et al., 2019).

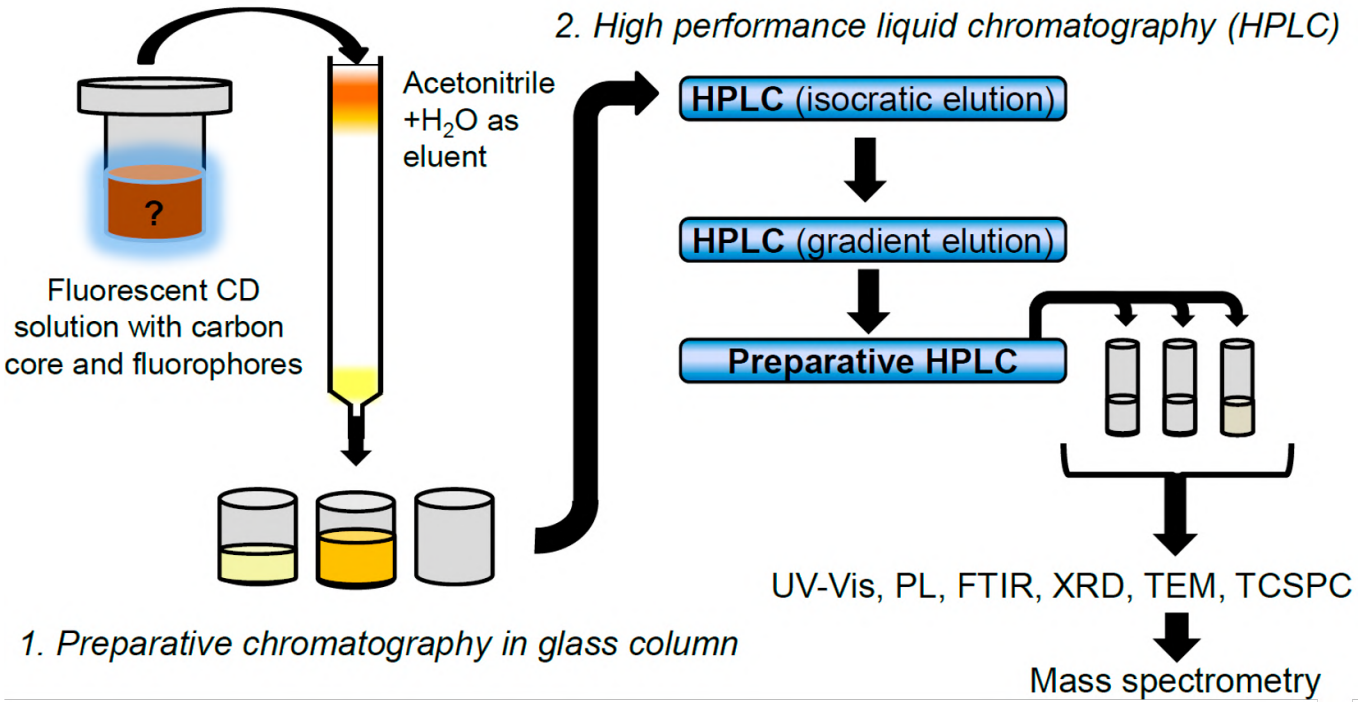
CDs syntheses yield multicomponent-mixtures since the precursors can undergo many different reaction pathways. Therefore, the exact structure of the CDs and the composition of the synthesized CD solution is still not understood. An efficient process for purification and also the separation of different CD species is required for deeper investigations of the CD's structure. Recently, we separated several species from products of solvothermal synthesis by isocratic normal pressure column chromatography. By spectroscopic analysis of the optical and structural properties of the product fractions, we showed that the CD solutions contain several fluorescent molecules and particles (Hinterberger et al., 2019).

For more precise separation of the different product species we then applied step gradient elution and transferred the separation process to normal-phase High Performance Liquid Chromatography (HPLC). Thus, we were able to improve the separation significantly and could associate the product fractions of a step-gradient HPLC elution with hydrophobic carbon cores, free molecular fluorophores and fluorophore polymers or carbon dots with linked fluorophores (Michaud et al., 2021). The step gradient separation allows us to elucidate the composition of the product mixtures resulting from different synthesis conditions. Thus, the influence of reaction temperature, time and solvent on the composition and optical properties of the products can be investigated in detail. We found the reported QY from CD mixtures might be overestimated as they result from non-separated CD mixtures. Here, we present QY for the different separated fractions finding that the highest values result from molecular fluorophores.

Acknowledgement: This work was funded by the Deutsche Forschungsgemeinschaft (DFG) – project number 182849149 – CRC 953: "Synthetic Carbon Allotropes".

Hinterberger, V., Damm, C., Haines, P., Guldi, D.M., Peukert, W., 2019. Nanoscale, 11: 8464-8474.

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The use of advanced fluorescence imaging for evaluation of the stabilizer choice and its effect on cytotoxicity, macrophage uptake, and bioactivity of curcumin nanocrystals

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Keywords | nanocrystal, curcumin, fluorescent imaging, confocal microscopy, immunogenicity, cytotoxicity, stability

Pharmaceutic nanocrystals find wide use in both oral and intravenous drug formulations. While the oral nanocrystalline drugs strongly profit from the increased dissolution rate and improved bioavailability, intravenously administered nanocrystals can be used for targeted drug delivery. When applied intravenously, a nanocrystal stabilization in form of a surfactant/amphiphile or a polymer coating is necessary to withstand the challenging environment of the human body. The eligible stabilizer should provide colloidal stability of nanocrystals and may further affect the dissolution rate of the condensed drug-made core. Moreover, the stabilizer can be also used for covalent coupling of ligands allowing for active targeting of various pathologies.

When considering the nanocrystals of poorly soluble drugs as solid nanoparticles, their biocompatibility and immunogenicity must not be overlooked. However, it is not an easy chore to evaluate the characteristics of the dissolving particles with a methodology optimized for non-dissolving systems. The most significant difficulties lie in a presence of both dissolved and solid forms of the same chemical entity, which significantly complicate the detection of the particles in the biological samples.

This work presents the application of advanced fluorescent imaging of live cells and their interaction with various types of nanocrystals of curcumin. Curcumin is a naturally occurring substance of many biological activities (anti-inflammatory, anticancer, antioxidant, and other). Its medical application is however hindered by the poor water solubility and low bioavailability. Curcumin is characterized by unique fluorescent properties which strongly depend on its form (crystalline or dissolved) and surrounding environment (water, ethanol, or lipophilic cellular membranes). The developed methodology based on the application of various excitation lasers, emission filters, and photobleaching allows for to detection of the crystalline particles inside the cells and can be therefore used to evaluate the immunogenicity, cytotoxicity, and nanocrystal stability in the media. Such findings are fundamental for a deeper understanding of the effect of stabilizers on the quality and bioactivity of the nanocrystalline formulations.

Systematic study of wet milling: Effect of surfactants on the stability, breakage kinetics, and dissolution profile of drug nanocrystals

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(1) University of Chemistry and Technology, Prague, (2) University of Chemistry and Technology, Prague

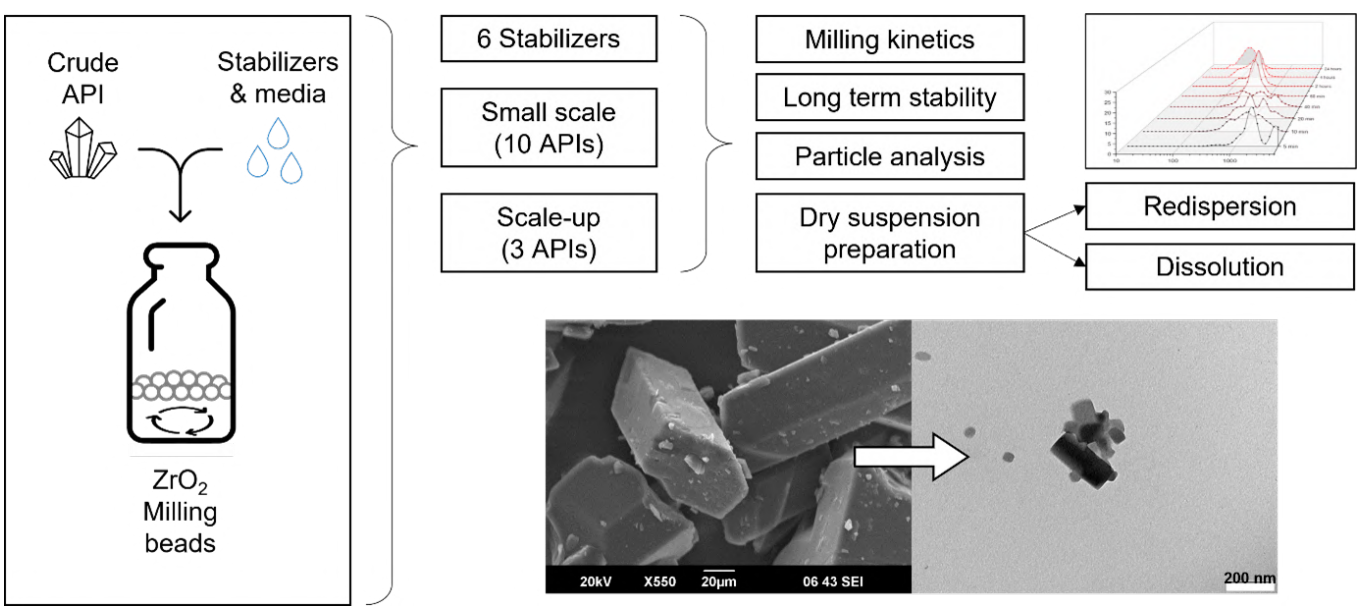
Keywords | wet milling, particle analysis, morphology, dissolution

Top-down nanocrystal preparation has been for a long time considered a viable and widely used method for improving the properties of APIs (active pharmaceutical ingredients), such as their low solubility or bioavailability. While a number of APIs are currently marketed in the form of nanocrystals or nanosuspensions, only little is known about which drugs are suitable for top-down nanomilling and why, as well as the existence of parameters that would define their suitability.

This study attempts to find the determining parameters of wet milling on a set of APIs and stabilizers and applies this knowledge to nanocrystalline formulations of APIs. The effects of API morphology, solubility, stabilization types, and milling parameters were studied, and stability was observed over a period of 8 weeks. Combination of sterical and electrostatic stabilization was found to be the best route for nanosizing a wide spectrum of drugs. This conclusion was underlined by the excellent performance of the combination of HPMC and SDS (hydroxypropyl methylcellulose and sodium dodecyl sulphate) in all tests performed. A considerable influence of API morphology on milling kinetics was also observed.

To further apply the knowledge obtained in the first part of this study, methods of preparation of dry suspensions & its resuspension capabilities were studied to allow for easier and more stable storage of nanocrystals. The dissolution characteristics of nanocrystals were also studied, showing that dissolution speed can be easily varied by modifying the method of dry suspension preparation.

The results from both parts of this study provide insight into nanocrystalline API formulations prepared via wet milling, both in the context of preparation, namely selection of APIs and stabilizers, and in the context of formulation properties, tailored dissolution characteristics, storage stability improvements, and others.



Impacting the Onset of Micro-explosions in Nanoparticle Formation by Flame Spray Pyrolysis by means of the local Process and Flow Conditions

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Keywords | Flame Spray Pyrolysis, Gas-Phase Synthesis, Micro-explosion, Metal oxides, Spray Combustion

Metal-oxide nanoparticles are of high interest in applications such as batteries, biomedicine and magnetic materials. A one- step process presents flame spray pyrolysis (FSP) that enables the tailored synthesis of nanoparticles made of a wide range of metal elements. The scale-up potential of FSP is high and a deep understanding of the process fundamentals will enable the synthesis of more novel materials. The spray formation is considered to play a key role as very short process time scales set the initial conditions for nanoparticle formation. Besides, the presence of drop micro-explosions has been evidenced in FSP that is linked to the formation of homogeneous nanoparticles (Stodt 2022). The role of these micro-explosions on the particle formation, however, is unclear.

In this work, the influence of atomization and flow conditions on the formation of bimodal drop size distributions (DSD), that are product of micro-explosions, are investigated by phase-Doppler anemometry. The experiments are carried out with varying Reynolds- and Mach-numbers and momentum-ratios of the precursor and dispersion gas in a twin-fluid burner. The gas-to-liquid mass ratio and the fuel/oxidizer ratio were hold constant to enable comparisons between the experiments. The evolution of the DSD along the height above the burner (HAB) is shown in Fig.1a visualizing the formation of a bimodal DSD. The results reveal that the temporary evolution of the bimodal DSD significantly depend on the magnitude of the Reynolds- number and, hence, on the turbulent mixing. The primary particle sizes have been analyzed by BET. As displayed in Fig.1b, the nanoparticle size clearly decreases with an increasing Reynolds number which might be attributed to a reduced high- temperature residence time of the particles in the flame.

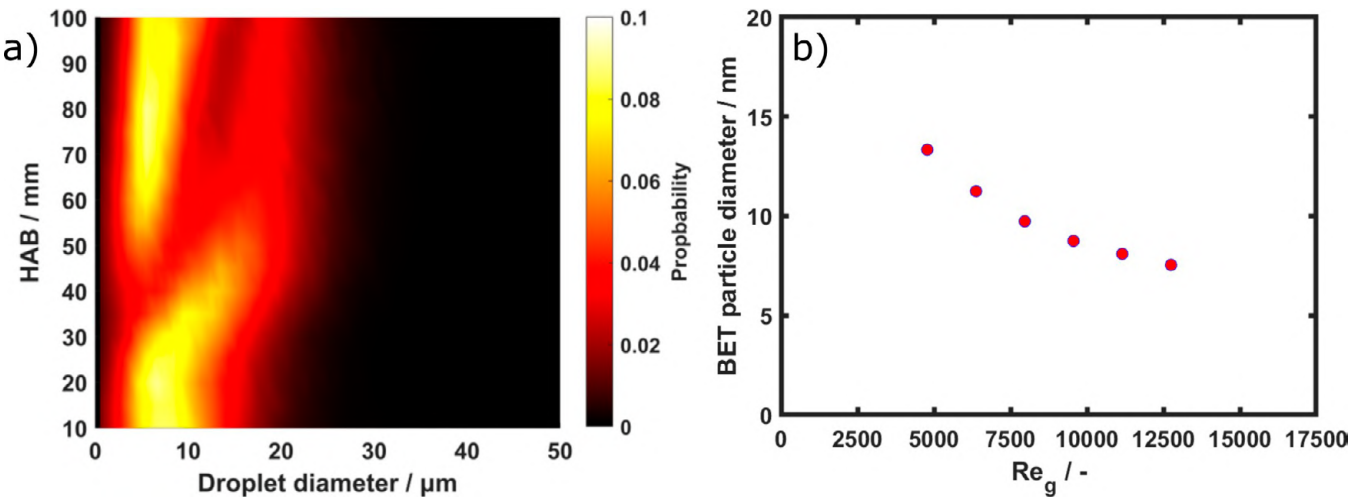


Figure 1: a) Evolution of DSD histogram along the centreline of the spray for a precursor feedrate of 3mLmin-1 and a dispersion gas flow rate of 15Lmin-1; b) Primary nanoparticle diameter as a function of the Reynolds-number.

The financial support of this project through the German-Research-Foundation within the priority program SPP1980SpraySyn (KI1396/6-2 and FR912/42-2) is gratefully acknowledged.

Stodt. et al., Microexplosions of multicomponent drops in spray flames, Combust. Flame, 240(2022)112043

Fundamental Understandings of Colloidal TiO2 Nanoparticles: Relationships between Surface Ligand Structure and Dispersibility

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Keywords | Nanoparticle, Dispersibility, Organic ligands

Nanoparticle-ligand complexes, which are nanoparticles coated with organic molecules, are expected to be applied in various fields for their processability and size-dependent properties. Nanoparticles are prone to aggregation due to van der Waals force between nanoparticles and thus enhancing dispersibility in solvents is an important issue in handling them. A common strategy for stabilizing dispersion of colloids in nonpolar solvents is to use ligands having hydrocarbon chains, which are expected to have steric repulsive forces (Talapin *et al.*, 2016). However, the length of the *n*-alkyl chain of the ligand required to enhance dispersibility has been a subject of controversy, because several experimental facts inconsistent with classical colloid model have been reported in recent years (Kraus *et al.*, 2018).

In this study, sub-10 nm TiO2 nanoparticles are synthesized by a reported solvothermal method (Do *et al.*, 2009). To evaluate the relationships between surface ligand structure and their dispersibility in organic solvents, TiO2 nanoparticles are capped with *n*-alkylphosphonates having various chain lengths by the ligand exchange strategy. The figure shows the key feature of the dispersibility of TiO2 nanoparticles capped with *n*-alkylphosphonates in toluene. It was found that both too long and too short ligands are detrimental to dispersibility.

To further investigate this solution property, the dispersibility of colloidal TiO2 at various solution temperatures were analyzed. Kinetic and thermodynamic validation through a series of experiments provided a better understanding of the underlying surface chemistry between ligand *n*-alkyl chain length and dispersibility. Detailed experimental results will be discussed in the presentation.

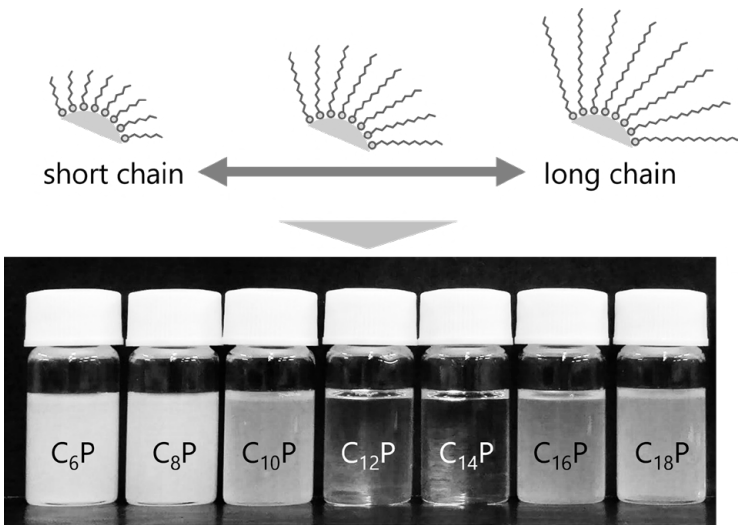


Fig. Schematic illustration and photographs of colloidal TiO2 nanoparticles capped with various *n*-alkylphosphonates in toluene.

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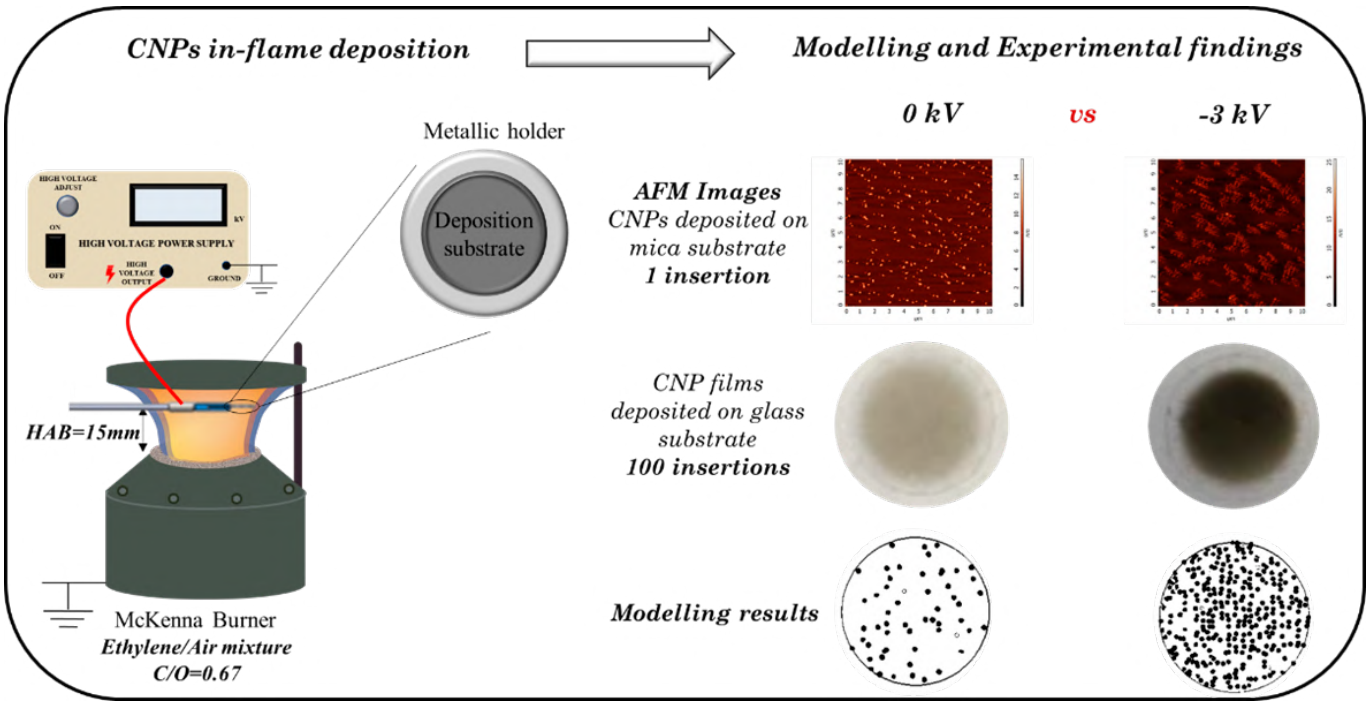
Talapin, D. V. et al., Nat. Mater. 2016, 15, 141–153.

Role of electric fields in the in-flame deposition of Carbon NanoParticle films

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Keywords | Carbon NanoParticles; thin film; flame synthesis; electrophoretic phenomena



Carbon NanoParticles (CNPs) generated in a flames at higher temperature- around 1800 K- can be deposited over a cooled substrate using thermophoresis as the controlling deposition mechanism. Thermophoresis is proved to be a reliable process for deposition of CNPs and formation of thin films and by changing the flame parameters and the harvesting point, it is possible to obtain films with different properties: by increasing the number of insertions in flame, the film thickness can be adjusted (De Falco et al. 2017). However, thermophoretic forces are complex to tune, as these are dependent on the temperature gradient in the boundary layer near to the substrate. Moreover, the structures of the deposited films are diffusion-controlled with low particles Peclet number and porous bulk structure (Castillo et al, 2014).

This work, as part of the PRIN project 2017PJ5XXX: "MAGIC DUST", proposes the possibility of applying electric fields in flame to activate electrophoretic phenomena. In this way, it is possible to change the harvesting rate and adjust the properties of the CNP films, without modifying flame conditions. To this aim, a new experimental setup has been designed and operated. It consists of an ethylene/air premixed laminar flame with C/O=0.67 at atmospheric pressure stabilized on a McKenna burner. The CNP film was generated on circular substrates blocked on a device consisting of a probe alternatively inserted inside the flame. The substrate is kept at a fixed electric potential (from 0kV to -3kV) while the burner is grounded. CNP film properties have been analyzed by AFM, light absorption and "sessil drop" method. The experiments clearly show that the application of electric field gives rise to a faster deposition and to the formation of more compact structures with higher detachment. In parallel, a numerical model for particle harvesting has been developed. The model results indicate that the deposition rate is 6 times faster passing from 0 kV to -3kV and CNPs trajectories are more perpendicular to the deposition substrate. These findings correspond to a higher Peclet number, with a transition to a ballistic-dominated deposition mechanism, leading to compact deposits.

Synthesis and catalytic applications of Lanthanum Aluminate Perovskites (LaAlO3)

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Keywords | Perovskite, lanthanum aluminate, synthesis methods, catalytic applications.

Lanthanum aluminate perovskite has an excellent stability at high temperatures, low toxicity and also offers a wide versatility to substitution of La3+ and Al3+, thus allowing it to be applied as a catalyst, nano-adsorbent amongst other equally important uses. As such, LaAlO3 perovskites have recently gained importance. This work covers strategies that have been developed over the past 10 years to synthesize some representative members of the LaAlO3 family in order to provide the researcher with a rational guide to understand the synthetic challenges, advantages and limitations associated with the preparation of these materials, and to highlight their most recent catalytic applications.

Extensive research has been performed to optimize the production of LaAlO3 perovskites using a variety of chemical routes. Methods with a lower synthesis temperature are preferred as they lead to small particle sizes and high surface areas. Recently, a pure LaAlO3 phase with an average crystallite size (~ 29 nm) has been obtained by way of a sol-gel reaction using natural reagents in which the calcination temperature was reduced to 600 °C. It is apparent that chemical co- precipitation using aqueous Na2CO3 solution as a basic precipitant is a simple method for the synthesis of pure LaAlO3 perovskites at pH (7 to 8). The calcination temperature (400 °C) is the lowest process temperature used to date for complete LaAlO3 formation using the thermovapourous method in water, although with large particle size (100 – 700 nm). However, the citrate sol-gel method is still the method of choice for researchers to prepare these materials due to high homogeneity and purity of the final material. Although the preparation methods published to date have proved successful for obtaining some representative members of the LaAlO3 perovskite family, they are not entirely satisfactory since they present several inherent shortcomings, such as the use of environmentally unfriendly and costly precursors and solvents.

Acknowledgments

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Green Synthesis of Hollow Structures Through the Decomposition of Azo Compounds Incorporated Inside Polymer Particles

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Keywords | Template-free, Hollow particle, Decomposition reaction, Azo compound

Hollow polymer particles have been applied in various fields owing to their high specific surface area and inner volume. The hollow regions in such particles are generally synthesized using a template. However, chemical agents, such as hydrofluoric acid, must be used to remove the templates, which is associated with a high environmental load. To address this problem, we performed a study aimed at establishing a method to synthesize hollow polymer particles without a template. First, azo compounds were dissolved in a styrene monomer phase, and soap-free emulsion polymerization was performed to produce polystyrene particles. The azo compounds were incorporated into the polystyrene particles from the monomer phase at a polymerization temperature greater than the melting point of the azo compounds. In addition, the polymerization temperature should be less than the 10-h half-life temperature of the azo compounds to prevent the decomposition reaction. Finally, the polystyrene particles were heated at a temperature greater than the 10-h half-life temperature of the azo compounds to emit nitrogen gas, and the azo compounds were decomposed to prepare the hollow regions in the polystyrene particles. This study provides a template-free and environmental-friendly method for the synthesis of hollow polymer particles with the size < 300 nm [1].

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Aminophosphine-based continuous liquid-phase synthesis of InP quantum dots in customized tubular flow reactor

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Keywords | Quantum dots; Indium phosphide; Flow synthesis

InP quantum dots (QDs) are typical III-V group semiconductor nanocrystals that feature large absorption coefficient, broad color tunability, and low toxicity, which render them promising alternatives to classic Cd/Pb-based QDs for applications in display, lighting, etc. [1]. Synthesis of InP based QDs is usually performed in glass flasks under high temperatures and inert atmosphere. However, flask-based batch synthesis

presented a number of problems, often mentioned were improper mixing, heating, reagent addition, poor reproducibility and difficulties in scale-up [2]. When aiming for industrial production, a continuous synthesis process offers several advantages compared to the scale-up of a batch reaction, such as process intensification, high reproducibility due to exact control of process conditions and low hazard potential due to the small volumes handled per unit. This makes up-scaling easier to implement and less dangerous.

Here we present a newly designed high temperature and continuous tubular flow reactor for the continuous and scalable synthesis of InP QDs with a safer aminophosphine precursor comparing to standard protocols involving highly pyrophoric and expensive (TMS)3P (Fig. 1). For homogeneous nucleation, the size of resulting InP QDs can be tuned by varying the temperature as indicated from the InP absorption peak shift at high temperature (above 300 °C). For heterogeneous nucleation, the QDs size is more tunable at relatively low temperature (180 °C).

In conclusion, our work is dedicated to develop a customized tubular flow reactor that enables the optimization of a wide range of process parameters (reaction temperature, residence time, concentration of precursors, etc.) and large amounts of high-quality material when compared to batch synthesis. Additionally, by changing the process parameters, the size of the InP QDs can be tuned to cover nearly the entire visible spectrum (480-650 nm) with good reproducibility.

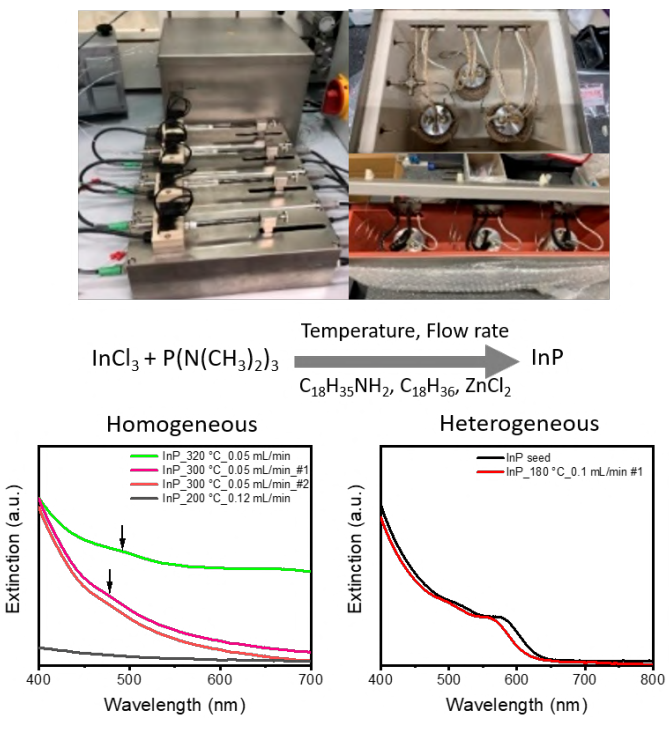


Fig. 1 Continuous flow synthesis of InP QDs in the tubular flow reactor.

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Synthesis And Characterization of Self-Assembled Nanostructured Films of TiO2-Carbon Nanoparticles

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Keywords | Flame synthesis; composite nanostructred films; TiO2 nanoparticles; flame formed carbon nanoparticles; scanning tunneling microscopy

In this work, nanostructured films of flame-formed carbon and TiO2 nanoparticles are produced through a simple two-step procedure

based on the flame synthesis of nanoparticles and the simultaneous deposition by thermophoresis into films. A carbonaceous granular film is produced by thermophoresis-driven self-assembling of carbon nanoparticles (CNPs) with diameters in the order of 20 nm. The flame reactor used for the synthesis of CNPs is a laminar premixed flame of ethylene and air operated in fuel-rich conditions. Then, the composite film is produced by the subsequent thermophoretic deposition of anatase TiO2 nanoparticles with a mean diameter of 3.5 nm. Anatase nanoparticles are produced in a flame-synthesis system based on a fuel-lean ethylene/air laminar premixed flame and titanium isopropoxide as titania precursor and deposited by thermophoresis on the surface of the film, where they intercalate between the carbon grains and diffuse within the pores. The electronic properties of TiO2-CNP films are characterized in relation to the morphology of films by measuring the electronic band gap using scanning tunneling spectroscopy (STS) technique. The electrical characterization of nanostructured film current-voltage characteristics is performed. A resistance switching phenomenon is observed, suggesting that TiO2-CNP films are promising candidates for application as resistive switching components.

Hybrid nanocatalytic materials for industrial-scale H2 production from HCOOH under ambient conditions

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Keywords | nanohybrids, SiO2, grafting, H2 catalysis, Formic Acid, Fe, Ru, cocatalyst, industrial scale

H2 production under mild, near ambient P, T, is of immediate scientific, economic and environmental interest. Formic Acid, HCOOH, is an H2-stroring molecule in liquid form. Thus, catalytic dehydrogenation of FA is currently an advanced H2- production approach, which trespasses the threshold towards industrial-scale implication. We will present innovative nanohybrids which allow large-volume, continuous production of H2, for several weeks, under ambient P, T. We will discuss comparatively a Fe-based and a Ru-based catalysts associated with SiO2 nanoparticles, that perform highly selective H2 production from HCOOH with zero CO. Mechanistic insights will be highlighted regarding he role of initial Hydride-Metal formation, the role/need-or-not of cocatalysts, the role of remnant H2O. The key role of [particle-catalyst] interface will be highlighted.

Targeted polydopamine nanoparticles transporting mycophenolic acid for innate immune response modulation in transplantation

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Keywords | Polydopamine nanoparticles, mycophenolic acid, innate immune response, allograft rejection

In recent decades, the development of more powerful immunosuppressive agents (IAs) has made it possible to effectively prevent transplant acute rejection. However, immunosuppressive medication must be chronically administered, and this increases patient susceptibility to infections and causes undesirable side effects that may lead to gradual deterioration of the transplanted organs (Vehara et al., 2019).

Herein, we report the use of nanoparticles (NPs) for the targeted administration of IAs to improve IA efficacy while reducing their side effects. More concretely, the aim pursued in this work was to synthesize and characterize polydopamine (PDA) NPs (190 nm) as a targeted vehicle of mycophenolic acid (MPA) to modulate the innate immune response mediated by phagocytic cells. To selectively lead the mitotic inhibitor to such cells, an antibody (Ab) against the CD11b receptor (α-chain of the integrin receptor CD11b/CD18) was adsorbed to PDA NPs. Once conjugated and after determining the incorporation efficiency of the immunosuppressant and the Ab (12-18 µg MPA and 11-12 µg Ab/mg NPs), the MPA release profile from PDA NPs was in vitro analyzed. Our findings showed a sustained release of the IA, which followed pH-dependent kinetics.

Therefore, this PDA-based nanovehicle could help to prevent early MPA release into the extracellular medium, increasing the chances of the IA to be transported to the draining lymphoid, where the immune response takes place. Likewise, internalization of MPA-carrying PDA NPs in murine macrophages (J774.A1 cell line) was analyzed by confocal microscopy, observing that it was receptor-mediated. Finally, the immunosuppressive activity of the targeted PDA NPs (0.01-0.05 mg/mL) was studied in vitro with both J774.A1 cells and primary peritoneal

macrophages, finding that they significantly reduced cell viability (to 26.5-2% after 72h incubation). Thus, based on the promising results obtained, ongoing assays are being performed to further evaluate the in vivo toxicity and immunosuppressive activity of the targeted PDA NPs, which could represent an innovative tool to try to modulate the innate immune response in the context of allograft rejection.

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Cationic micro/nanofibrillated cellulose: assessment of the deconstruction effect of two distinct cationization methods

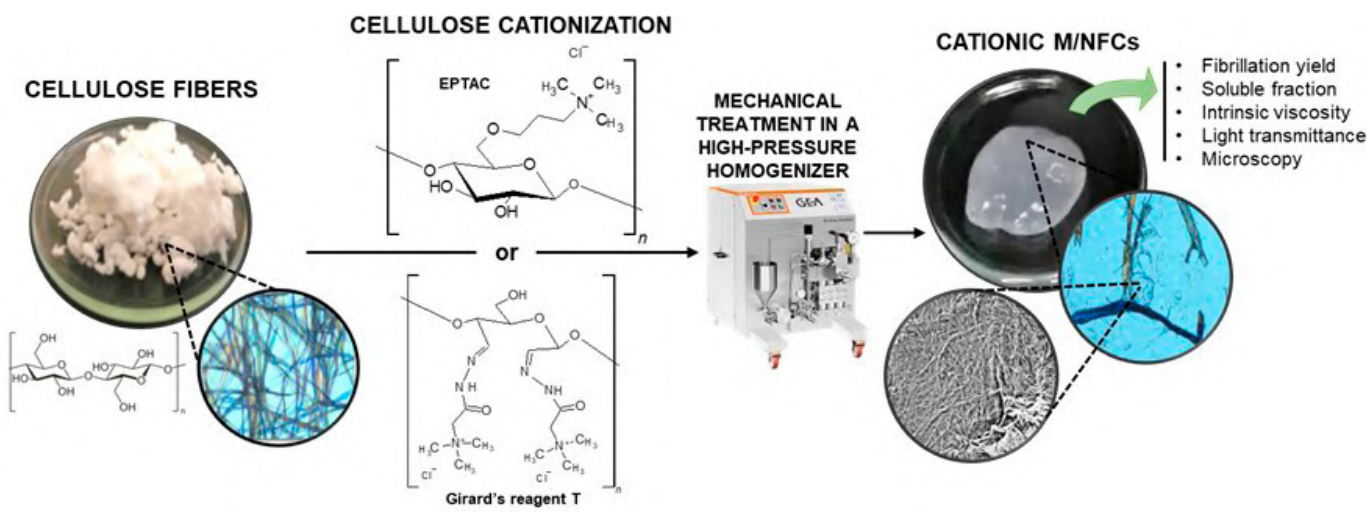
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Keywords | Cationic cellulose; Nanocellulose; Quaternary ammonium; Solubilization

Cellulose, primarily derived from lignocellulosic materials has been gaining increasing attention over the most recent decades as an alternative viable biomass feedstock for the development of new materials. Distinct mechanical and chemical methods have been developed over the years to deconstruct lignocellulosic materials into their hierarchical sub-structures to produce new cellulose-based products, such as micro/nanofibrillated celluloses (MNFCs). Among the different chemical pretreatments, cellulose cationization has already been shown to be an effective method for the production of cationic MNFCs (Aulin et al. 2010), with the benefit of presenting affinity towards negatively charged particles, allowing for a variety of applications (flocculants, adsorbents, biocides).

In the present work, quaternary ammonium cations were attached to cellulose fibers by two distinct approaches: 1) a direct reaction with 2,3-epoxypropyltrimethylammonium chloride (EPTAC) in an alkaline medium or 2) a dual step cationization, where the cellulose is first oxidized to dialdehyde cellulose with sodium periodate and then reacted with Girard's reagent T (Fig. 1). The cationic fibers, with diverse degrees of substitution, were further subjected to a mechanical treatment in a high- pressure homogenizer to produce cationic MNFCs (Pedrosa et al. 2022). The deconstruction potential of the two distinct cationization methods was compared for the first time and evaluated in terms of fibrillation yield (by gravimetric analysis), solubilized fraction (by membrane filtration), intrinsic viscosity measurements, light transmittance and microscopy.

From the obtained results, it was possible to observe that the dual step cationization resulted in significantly more degradation of the cellulosic structure than the direct cationization, leading to the formation of shorter fibrils, partial solubilization of the cellulose and less cohesive gels.



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Kinetic study of tempo-mediated oxidation and real-time monitoring strategy

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Keywords | Nanocellulose; Real-time monitoring system; Tempo-mediated oxidation

The present work shows the feasibility of an effective real-time monitoring system for TEMPO-mediated oxidation reactions while using the catalyzed reaction and measuring the NaOH consumption over time providing kinetic information as function of the process conditions. A bleached kraft eucalyptus pulp (BKEP) was subjected to TEMPO-mediated oxidation in different conditions of temperature (5 – 35 °C), oxidizer amount (5 – 15 mmol/g), and TEMPO catalyst amount (2 – 32 g/kg). This provided a three-dimension factorial plan able to provide the dependence between carboxyl content and time for different reaction conditions.

It was found that, the NaOH consumption exhibited a linear relationship with the carboxyl content of the fibers, regardless the process conditions, which clearly indicates that the evolution of the reaction could be directly monitored by means of considering the amount of NaOH/g of fiber. In addition, the effect of temperature and catalyst was elucidated, obtaining the kinetic constant of the reaction as function of temperature and catalyst. Further, as the temperature was varied during the experimental work, the activation energy was properly obtained through the Arrhenius equation. The system was assumed to be homogeneous, although heterogeneities are known due to the morphology of the reacting species (fibers). From the study of the temperature, kinetic constants ranging from 0.58 to 5.50/seconds were found, responding to a first-order reaction system. Further, the activation energy was quantified to be around 73.70 kJ/mol for an addition of 16 g/kg of TEMPO, which is in accordance with previously reported data (Dai et al, 2011; Sun et al, 2005). Regarding the study of the effect of TEMPO catalyst, kinetic constants ranging from 0.47 to 3.63/seconds were found, indicating that extra catalyst do not benefit the rate of reaction, while high temperature does (30 °C).

Finally, regarding the oxidizer amount, changes on the kinetic constant were observed, which clearly indicates that changes on the limiting reagent occurred. This last part of the study deserves further research in terms of heterogeneous catalytic systems, as changes on morphology may occur, exposing further primary alcohol groups and, thus, affecting the reagent concentration.

Mechanochemistry: a sustainable route to novel metal- supported ceria-based heterogeneous catalysts

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Keywords | mechanochemistry palladium ceria heterogeneous catalysts dry milling

The preparation of efficient heterogeneous catalysts is key in the improvement of current industrial chemical processes, either aimed at the synthesis of feedstock chemicals or at pollutants abatement from energy-intensive manufacturing processes. In addition, the sustainability of the whole process needs to be addressed, including the preparation method for the catalytic material of choice (Benavides et al., 2017). In this regard, the mechano-chemical synthesis is widely recognized as an interesting alternative due to its many environmental advantages (Ardila-Fierro and Hernandez, 2021). However, due to the many interwoven parameters involved in the milling process, the fine tuning of catalytic materials such as metal- supported heterogeneous catalysts was mainly discouraged, until recently (Danielis et al., 2018).

In the latest years, we have developed a novel solvent-free mild-energy milling method for the synthesis of metal-supported catalysts, where solution-based syntheses represent the state-of-the-art preparation route (Danielis et al., 2018). The mild milling intensity coupled with the use of a redox active support such as CeO2 allowed for the preparation of catalytic powders with a peculiar core-shell architecture, the latter containing both the metal and the support elements and resulting in enhanced catalytic performance. Here, we focus on the results obtained for Pd/CeO2 systems, where the milling process directly influences the particle morphology and, in turn, catalytic performance. The metal-support interaction promoted during milling resulted in a well-developed core-shell structure accompanied by remarkable CH4 oxidation activity, both in lean and rich conditions, and by selective activation routes of other key molecules such as CO2 and NOx.

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The Impact of Support Material of Cobalt-Based Catalysts Prepared by Double Flame Spray Pyrolysis on CO2 Methanation Dynamics

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Keywords | CO2 methanation, heterogeneous catalysis, transient experimentation, double flame spray pyrolysis, support effect

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+ these authors contributed equally to this work
Storage of fluctuating renewable energies and use of sequestered CO2 from combustion can be achieved by chemical storage using methanation

The development of catalysts requires a fundamental understanding of the dynamics of the kinetic processes taking place on the catalyst surface. To study such catalytic systems defined model catalysts are required. Double Flame Spray Pyrolysis was demonstrated earlier by Stahl et al. 2021 to be a suitable tool to synthesize such materials.

In the present study DFSP is used to synthesize supported cobalt catalysts for defined mixing of aerosol streams coming from two individual flames. This way nanoparticle formation of active metal and support is spatially separated to form interconnected hetero-aggregates. Using identical flame parameters hetero-aggregates can be prepared having an identical cobalt particle size distribution and degree of mixing on different support consisting of SiO2, TiO2 or SiO2-TiO2 mixtures. The Periodic Transient Kinetics (PTK) technique was used to study transient catalyst behavior by step changes of the feed gas composition and the analysis of the reactor response with high temporal resolution.

It was revealed that H2O adsorption strongly depends on the support with increasing sorption capacity from SiO2, via SiO2- TiO2 to TiO2. The storage of H2O on the support induces a sorption-enhancement effect and thereby promotes the transient CH4 formation rate for Co/TiO2 catalysts. Transient experiments before and after a steady-state operation period under CO2 methanation reveal an unchanging reaction mechanism, while changes in selectivity towards CH4 and CO are observed together with a certain degree of deactivation. Hence, the support material was identified to play a major role in activity, selectivity and deactivation behavior for supported Co catalysts in CO2 methanation.

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Influence of particulate properties on the performance of nano-silicon graphite composites for lithium-ion battery anodes

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Keywords | silicon nanoparticles, graphite, composite, lithium-ion battery, particle size, surface area, particle size

The success of electro mobility necessitates the enhancement of lithium-ion batteries to increase energy density and reduce costs. Therefore, new materials like silicon has attracted much attention in the past years. Silicon exhibits the highest gravimetric capacity among all anode materials and is an abundant and low cost material. A major challenge arising from the use of silicon is the severe volume change during cycling (up to 300 %), which can result in the pulverization of particles and inhomogeneities in electrode structure, as well as a growing solid electrolyte interface (SEI), which consumes lithium during cycling. In order to overcome these challenges, the use of silicon nanoparticles and the structuring of these nanoparticles with graphite have been proven to be beneficial.

In the present study a core shell structured nano-silicon graphite composite (Si@Gr) has been developed and the influence of its composition (silicon particle size, silicon content, carbon coating) on its structural, mechanical and electrical properties has been studied (Müller et al.).

At first, a nano-silicon suspension was prepared by nano-milling in a stirred media mill. In the next step, the suspension was used to create the core-shell structure in a fluidized bed granulator. The influences of silicon particle size (120, 160, 250 nm), silicon concentration (5, 10, 15 wt.%) and a pitch based carbon coating were studied in detail. A smaller particle size led on the one hand to an increased specific surface area, which is detrimental in terms of side reactions, which are accompanied by lithium losses. On the other hand, the electrochemical performance could be enhanced because the absolute volume change diminishes with reduced particle size and, thus, minimizes the detrimental effects. For increasing silicon contents, a linear increase of the specific surface area and rising instability of the composite particles, which led to increased detachment of the nanoparticles, could be detected. In order to stabilize the particles a carbon coating was conducted (Si@Gr/C), which not only stabilized the particle structure, but also decreased the specific surface area and improved the electrochemical performance.

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Sonochemical oxidation of highly conductive carbon blacks (CBs) of turbostratic structure and its effect on the performance of a proton exchange membrane fuel cell (PEMFC)

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During ink preparation prior to electrocatalyst layer deposition, Pt-functionalized CB particles and ionomer must be mixed and homogenized in the colloidal state. This is usually realized via power ultrasound. When ultrasound is used, apart from the physical effects induced by acoustic cavitation, further chemical processes such as dissociation of water molecules into radicals (sonolysis) could occur, followed by further chemical reactions.

To leverage our understanding of the effect of ultrasound treatment, physicochemical properties of CBs were investigated regarding electrical conductivity, particle size distribution, isoelectric point and oxygen-containing surface groups. Finally, the effect of ultrasonication on the surface of CB particles, ionomer and electrochemical active surface area was evaluated in terms of PEMFC performance under real operating conditions. It was observed that the effect of ultrasound intensity on the surface properties strongly depends on the surface structure of the CB used. High surface (~800-1400 m^2/g) CB preserved its initial basicity after high intensity sonication. In contrast, a significant effect of sonication on the surface properties can be observed for medium (~245 m^2/g) and low (~62 m^2/g) surface CBs. After high intensity sonication, both (initially basic) medium and low surface CBs exhibit an acidic surface. It is assumed that the generation of free radicals during sonication causes an oxidation-related formation of acidic, oxygen-containing surface groups on the CB surface. Furthermore, it is strongly suspected that due to the short lifetime (ns) of the radicals and the distinct fine pore system of high surface CB, radicals are not able to reach and oxidize the surface of the pore system. For medium surface CB, significant decrease in PEMFC performance was observed with increasing sonication intensity (see figure 1).

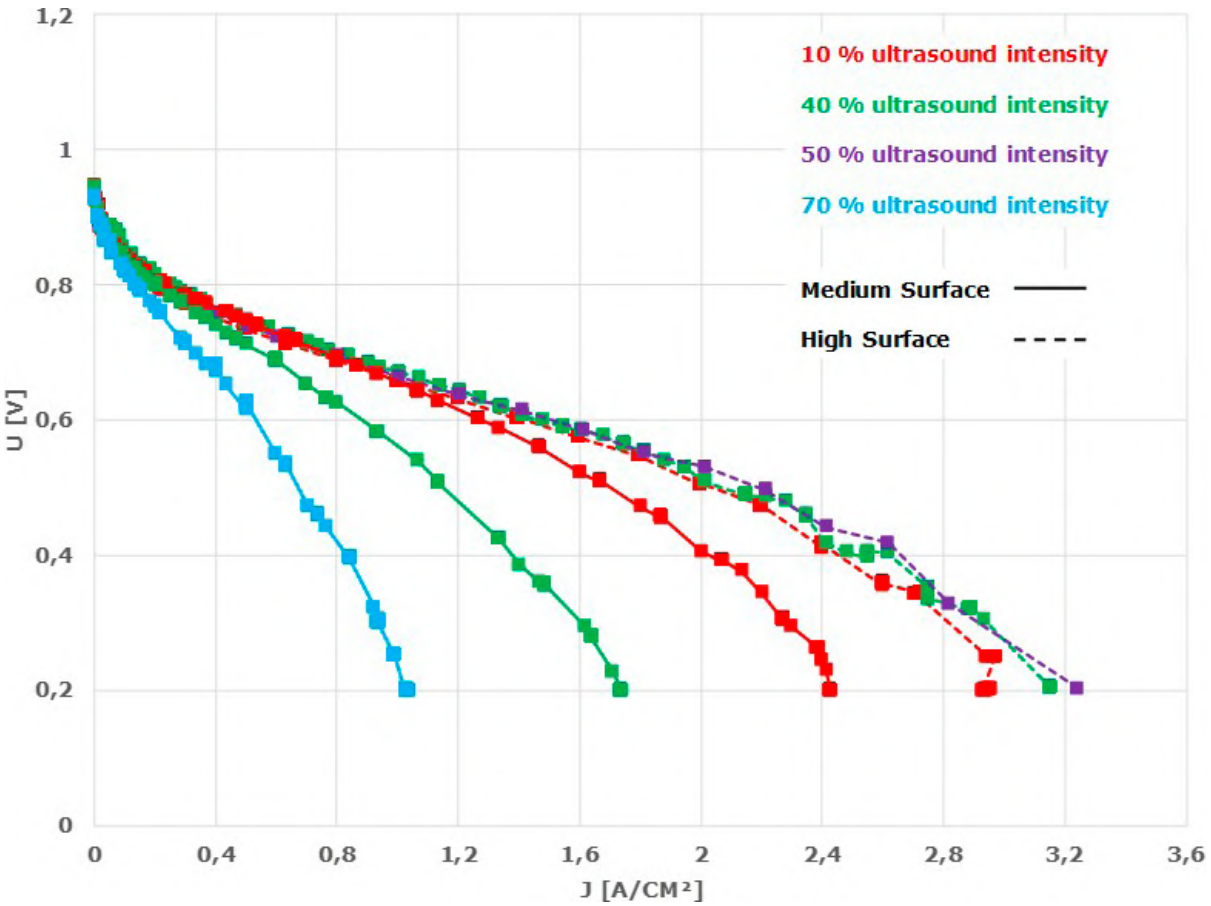


FIGURE 1: Effect of intensity variation of the ultrasonic treatment of ink slurries on polarization curves in a PEMFC

Presumably, the sonication-induced hydrophilic surface of the CB particles reduces the adsorption affinity of the hydrophobic ionomer backbone and consequently worsens their interactions in the cathode catalyst layer (CCL). Furthermore, a hydrophilic CCL retains product water, very likely resulting in flooding of the electrode and mass transfer limitations.

Nanostructured Materials for Renewable Energy Productions from the Hot-aerosol Synthesis

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Keywords | Flame Synthesis; Water Splitting; CO2 reduction; Electrolyser

Electrolysis of water and carbon dioxide to green hydrogen and carbon-contained fuels is a promising approach to achieve the net-zero carbon emission target. However, this approach primarily relies on the activity, stability, and cost of electrocatalysts. Generally, expensive and rare-earth materials such as Pt, Ir, or Au are utilized in small-scale measurements. For large-scale deployment of water or carbon oxide electrolysers, engineering active and stable electrocatalysts from earth- abundant elements are required. Here, we fabricated highly active electrocatalysts including Co3O4 and Bi2O3 fractals for hydrogen and formate productions, respectively, from flame spray pyrolysis. The electrocatalysts were in-depth characterized and tested for renewable hydrogen and formate productions using a Lab-scale size electrode. The electrocatalysts were then fabricated on gas diffusion layers such as carbon fiber papers or platinized titanium mesh for testing in industrial-revenant conditions. We demonstrate the possibility and potential of large-scale synthesis of electrocatalysts for renewable energy productions using a hot-aerosol synthesis approach.

Effect of conductive particles and substrates on enhanced methane production

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Keywords | Anaerobic Digestion, Direct Interspecies Electron Transfer, Conductive Particles, Methane Fermentation

Anaerobic digestion is a microbial process that produces renewable energy in the form of methane gas by treating various organic waste. Recently, it has been reported that addition of conductive materials (CMs) can help improve methanogenesis by promoting direct interspecies electron transfer (DIET) between exoelectrogenic bacteria and electron-accepting methanogens, but only with the presence of exoelectrogenic bacteria, i.e. *Geobacter* sp. (Barua et al., 2017). According to Rotaru (2014) ethanol as substrate could enrich the *Geobacter* sp. Therefore, using ethanol as substrate may be an important factor in cultivation for DIET to occur efficiently. Besides, the effect of DIET is considered to be dependent on the type of CMs including their physicochemical properties (Barua et al., 2017), yet research for direct comparison on effect of different CMs on DIET under the same experimental condition is still limited. Therefore, the objective of this study is to evaluate effect of methane production efficiency using various CMs in different substrate. 20 g/L granular activated carbon (GAC) powder, 1.0 g/L multi-walled carbon nanotube (MWCNT) and 0.6 g/L polyaniline (PANI) were used and evaluated in two sets of experiments. For comparison of substrate, sodium propionate was used in the experiment 1 as control, while ethanol was used in the experiment 2. Results shows significant enhancement of methane production rate in all the CMs conditions only in experiment 2, and the order was followed by GAC>MWCNT>PANI>Control (non-CM addition condition) (Fig.1). Enrichment of *Geobacteracea*, an exoelectrogenic bacteria was also observed only in experiment 2. Furthermore, the value of BET specific surface areas of CMs are in the same order as the methane production rate result, implying large surface area of CM with enriched Geobacteracea induced by ethanol cultivation could enhance methane production rate through DIET.

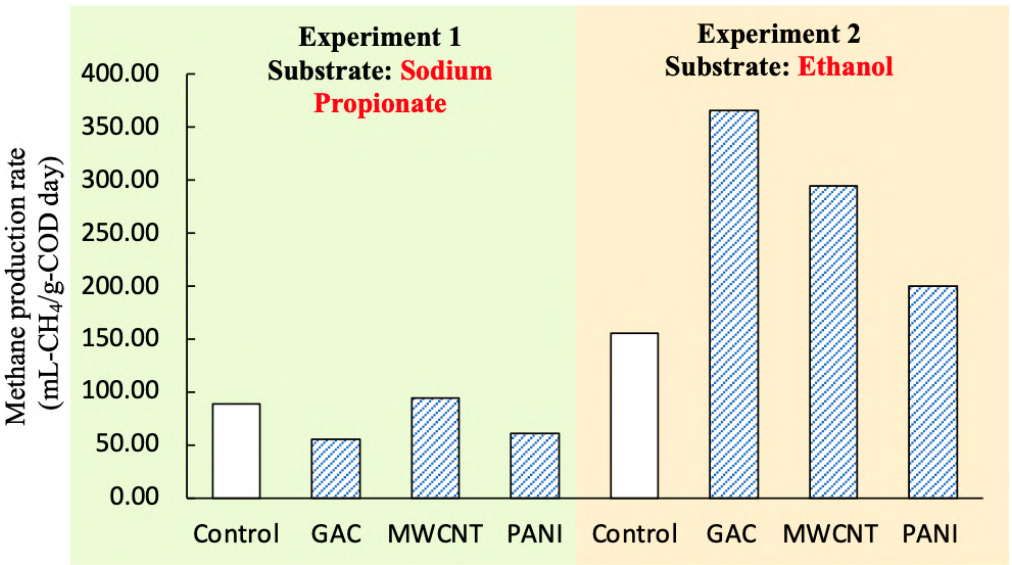


Figure 1. Methane production rate for each condition in both experiment

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Continuous synthesis of functionalized magnetic nanoparticles and application to solid-liquid separations

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Keywords | Magnetic nanoparticles (MNPs), continuous synthesis, hexavalent chromium, solid-liquid separations.

During the last decades, there has been growing interest in the use of magnetic nanoparticles (MNPs) due to their unique properties, worth to mention are their high specific surface area, easy functionalization, chemical stability and superparamagnetic behavior, which make them suitable materials in different fields including catalysis, data and energy storage, environmental remediation and biomedicine. This growing use of MNPs demands cost-effective methods for their synthesis that allow the continuous production of the particles and accurate control of process variables, such as residence time, mixing degree or temperature, to obtain small particle sizes and narrow size distributions. Microfluidics hold promise to achieve these requirements (García-Merino et al., 2021). This work reports a highly efficient continuous procedure for the synthesis of MNPs, consisting of a co-precipitation reaction followed by the surface functionalization of particles with amino groups, both steps taking place in continuous mode using a spiral shape microreactor (Circular section 3 mm, 400 mm length). The main properties of the particles were characterized with Fourier transform infrared spectroscopy, dynamic light scattering, transmission electron microscopy, Thermogravimetry and BET analysis. This way, a continuous production of MNPs has been achieved with reduced environmental effects due to the low energy and reactant consumption, minor particle agglomeration due to the microdevice geometry that enhances mixing, and higher reproducibility of the functionalization step than in batch processes. Furthermore, these MNPs have been applied in the solid-liquid separations field, for the capture of hexavalent chromium from aqueous solutions, selected as a model compound that has been widely studied (Rivero et al., 2004; Bringas et al., 2006). The capture process has been implemented in the previously described microreactor, and the results have been compared to those obtained in batch runs and with commercial ion exchange resins technology.

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Bi-Sn-Sb carbon-supported nanoparticles for the continuous electrochemical reduction of CO2 to formate with improved performance

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Keywords | Bi-Sn-Sb nanoparticles, CO2 electroreduction, formate, continuous operation, stability

Electrocatalytic reduction of CO2 to HCOO- or HCOOH represents a promising alternative to reduce the emissions of CO2 to the atmosphere. In this sense, HCOO- and HCOOH are raw materials with numerous and wide-ranging applications in industry. Despite the great advances developed in this field by using mainly Bi and Sn-based cathodes, it is required to develop new electrocatalytic materials with improved activity for the future implementation of the process at an industrial scale. In this sense, previous studies, carried out in DePRO's research group at the University of Cantabria, have employed Bi carbon-supported nanoparticles for the CO2 electroreduction to HCOO-, reaching product concentrations up to 18.02 g·L-1 with Faradaic efficiencies, rates, and energy consumptions of 45.1 %, 4.67 mmol·m-2·s-1 and 535 kWh·kmol-1, respectively. (Díaz-Sainz et al. 2019). In this context, the main goal of this communication is to study the performance of Bi-Sn-Sb carbon- supported nanoparticles for the continuous electrocatalytic reduction of CO2 to formate in a filter-press reactor to improve the stability of the electrodes, after the promising results achieved with Sb-based electrodes in an H-type electrochemical cell (Ávila-Bolívar et al. 2021). In this way, preliminary tests have been carried out using the same electrochemical reactor under similar operating conditions, working with a liquid electrolyte at the cathodic compartment of the filter press cell, achieving promising results in terms of concentrations (22.2 g·L-1), Faradaic Efficiencies (55.5 %), rates (5.75 mmol·m-2·s-1) and energy consumptions (510 kWh·kmol-1), demonstrating an improved performance of the electrodes, which represents a step forward in the development of the process for future implementation at industrial scale.

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Acknowledgments

The authors thank the financial support from the Spanish State Research Agency (AEI) through the projects PID2019-108136RB-C31, PID2019-108136RB-C32 and PID2020-112845RB-I00 (AEI /10.13039/501100011033).

Ordered Pt nanorod arrays grown on Au nanoseeds for proton exchange membrane fuel cells

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Keywords | Ordered electrodes, Pt nanorod, fuel cell

Owing to the distinctive surface properties and stability, three-dimensional (3D) ordered electrodes based on one- dimensional nanostructures are considered one of the potential strategies to realize large current density operation for proton exchange membrane fuel cells1, 2. In this work, gold nanoseeds are anchored on the gas diffusion layer surface via the physical sputtering method followed by Pt nanorod (NR) arrays in-situ grown on the surface to fabricate gas diffusion electrodes. Pt NRs, with a diameter of about 4 nm, are uniformly distributed within the catalyst layer forming a 3D ordered structure. The obtained electrode is directly tested as the cathode in air/H2 fuel cells, and the influence mechanisms of the Au seeds on the electrode power performance are carefully investigated. A maximum peak power density of 0.751 W/cm2 is recorded, which is 34.8% and 47.4% higher than that based on pure Pt NR array and commercial Pt/C nanoparticle electrodes, respectively. An accelerated degradation test is conducted to evaluate the electrode durability. The Au-induced Pt NR GDE also shows a lower performance degradation rate, which can be ascribed to the stable 1D nanostructure by deeper “roots” in the GDL surface provided by the Au sputtering.

An effective strategy for fabricating sustainable porous carbon spheres derived from Kraft lignin with controllable structures

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(1) Hiroshima University

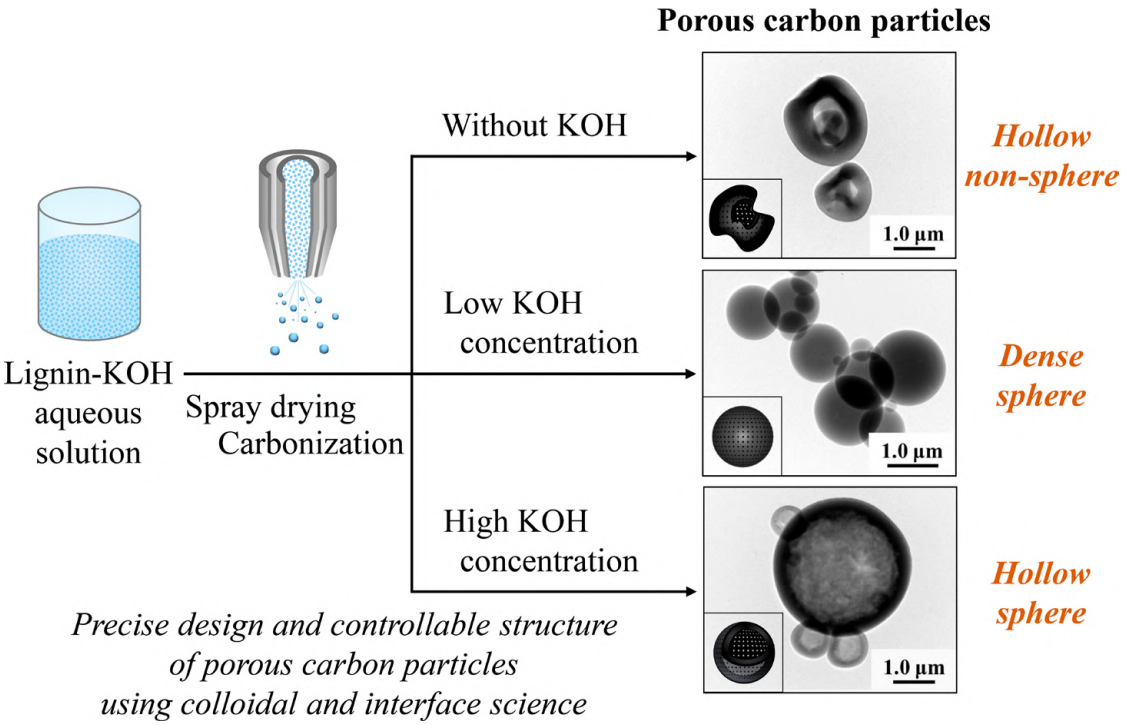
Keywords | Biomass; Porous carbon spheres; Spray drying method; Nanostructured particle; Sustainable material; Supercapacitor

Spherical carbon particles have attracted tremendous interests in recent years due to their intriguing structure-induced physicochemical properties and great potential for widespread applications. The rational synthesis of carbon-based materials from green, renewable, and cost-effective resources in accordance with sustainable development goals is of high interest and encouraged. In this regard, lignin has been considered as a potential sustainable source for the preparation of advanced porous carbonaceous materials due to its high carbon content (above 60 wt.%), abundant, low feedstock cost, and extensively crosslinked polyphenolic structure. However, their preparation with the precise design and controllable structure using a facile and scalable strategy remains a significant challenge. The motivation for this present study is to synthesize porous carbon spheres in a sustainable manner via a spray drying approach followed by a carbonization process, using Kraft lignin as the carbon precursor and potassium hydroxide (KOH) as the activation agent. As a result, the proposed method successfully controlled the structure of carbon particles from dense to hollow structure, and the surface textural properties can be easily tuned by adjusting the KOH concentration [1]. In addition, to obtain an in-depth understanding of the particle formation of carbon particles, a plausible mechanism is also investigated in this research, which provides systematic guidance for further fabrication of spherical carbon materials with appropriate architecture and composition. Furthermore, the high specific surface area (2424.8 m2 g-1) with micro-mesoporous structure of hollow carbon spheres (HCSs) were obtained at a low KOH-to-lignin mass ratio (below 1.5), which was in accordance with green chemistry principles. These HCSs have applications as electrode materials in supercapacitors for energy storage devices [2]. With the great achievements and continuous efforts in this important field, these results suggest that our approach adopted herein will open up opportunities for the development of advanced carbon materials and high value-added utilization of Kraft lignin as a promising material for potential applications.

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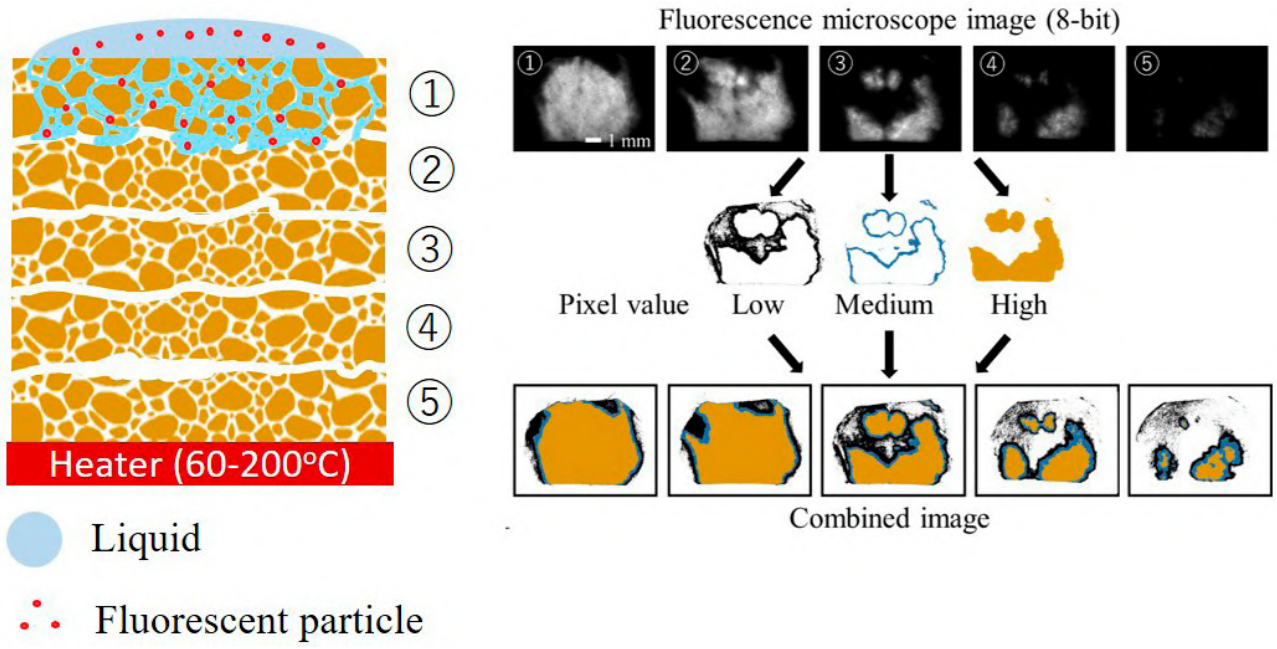


Observation of Glycerol-Water Mixtures Transport Through Heated Biomass Layers Using Colloidal Fluorescent Particles

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Keywords | Visualization, Liquid Transport, Porous, Fluorescence

Observation of Glycerol-Water Mixtures Transport Through Heated Biomass Layers Using Colloidal Fluorescent Particles



Visualization technique to monitor microliter liquid flow in porous media is essential to improve the quality of many products such as microfluidic paper-based analytical devices (μPADs), smart-fabric, and food packaging. While the visualization of in- plane liquid spreading is straightforward and has been performed extensively, (Schuszter et al., 2015; Cheng et al., 2017; Boscariol et al., 2018) visualization of trans-planar liquid penetration remains a challenge. The challenges stem from the complexity of the multilayer porous structure, the volatility of the liquid, the swelling of the fibers, and the small thickness of a single layer medium (Zhang et al., 2017; Chang and Kim, 2020). A number of studies have proposed imaging methods such as optical coherence tomography (Salesse et al., 2020), X-ray computed tomography (Zhang et al., 2017), nuclear magnetic resonance (NMR) (Bencsik et al., 2008), and magnetic resonance imaging (MRI) (Sederman and Gladden, 2001) to help visualize trans-planar liquid penetration. The implementation of such methods, however, requires the installation of complex instruments and the presence of trained personnel.

The present study proposes a method for visualization of microliter liquid transport using dispersed fluorescent particles as tracers. A mixture of colloidal fluorescent particles and glycerol (1:1, v/v) was added to a five-layered biomass sample where heating at various temperatures was applied at the bottom. Once the heating was completed, the five layers were disassembled and each layer was examined using the fluorescence microscope. In order to map the distribution of fluorescent particles, the resulting image was processed using ImageJ software. By using this method, we can determine the effects of forces such as gravity, capillary, viscosity resistance, surface tension, diffusion, and evaporation of the liquid. The heating temperature was found to affect the interaction between the liquid and the porous medium. The method also demonstrated a promising application for trace impurity detection in atmospheric conditions.

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Production of mixed Pd, Hf and Ta nanoparticles by atmospheric-pressure spark ablation

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Keywords | spark ablation, gas phase synthesis method, Pd-based nanoparticles

Palladium is considered one of the most effective hydrogen-sensing materials because it forms palladium hydride (PdHx) upon exposure to H2, thereby affecting its optical properties (Sousanis, 2021). Pd-based materials for optical H2 sensors, however, suffer from cracking, blistering and delamination due to expansion and contraction of the lattice during absorption/release of H2 gas. Cracks formed on Pd-based materials provide higher transmittance of light that can favor optical H2 sensing as it can suppress hysteresis (i.e., the difference in the optical response of the material when H2 pressure is increased or decreased) that deteriorate the performance of the sensors. Formation of cracks can be circumvented by alloying Pd with other metals or reducing the thickness of the sensing films. Alternatively, thin films comprised of nanoparticle building blocks have also been shown to circumvent hysteresis (Isaac et al., 2015).

An effective way of producing nanoparticle building blocks that can then be used as building blocks of nanomaterials is spark ablation: a gas phase synthesis method, where the spark plasma formed between two conductive electrodes, ablates material from them (Schwyn et al., 1988). The ablated materials form a vapor cloud, which quenched and carried away by a gas flow, forming nanoparticles upon nucleation and growth. Spark ablation can be used to produce nanoparticles with precise control over size and composition in a simple, inexpensive and environmental friendly method, since no chemical precursors are required and no waste is produced. Spark ablation method has been used to produce alloys (Isaac, 2015) and core-shell nanoparticles (Snellman et al., 2021).

Here we use spark ablation operated at atmospheric pressure to produce a range of Pd-based nanoparticles that can potentially be used for optical H2 sensing. More specifically, we produced Pd particles decorated with Ta or Hf nanoparticles as well as core-shell nanoparticles using the same elements. In addition, we have investigated the important role of electrode configuration in the production rate and chemical composition of Pd-Hf and Pd-Ta nanoparticles. The morphology and composition of the generated Pd-Hf and Pd-Ta nanoparticles were studied by Scanning Electron Microscopy, Scanning Transmission Electron Microscopy and Energy-Dispersive X-ray techniques.

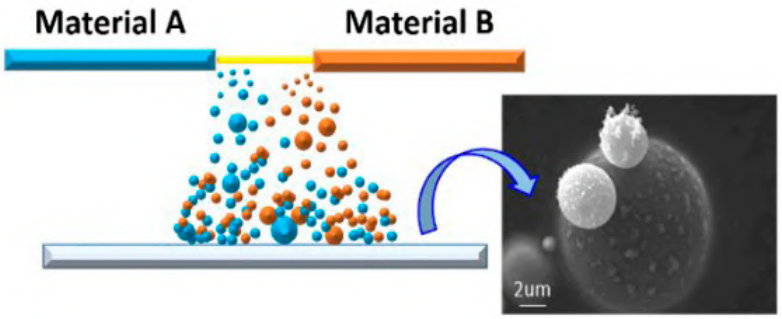


Figure 1. Spark ablation method for the production of Pd-decorated particles.

Large-scale synthesis of novel LaCoO3/graphene catalysts for the degradation of organic pollutants

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Keywords | Bisphenol A, gas-phase synthesis, graphene, heterogeneous catalysis, peroxymonosulfate.

The metal leaching in perovskite-based catalysts during wastewater treatment limited their industrial applications. Moreover, the robust and scalable synthesis of stable perovskite-based catalysts with a high specific surface area is extremely important for the practical application in wastewater treatment but has been rarely achieved so far.

In the current study, we developed a low-cost and scalable method to synthesize novel stable and environmentally-friendly LaCoO3/graphene nanocomposites for removing organic pollutants from wastewater. The novel LaCoO3/graphene catalyst exhibited an excellent efficiency of degradation towards bisphenol A, metoprolol, diclofenac, and carbamazepine, compared to the LaCoO3 catalyst in the peroxymonosulfate activation system. This enhanced removal efficiency of the LaCoO3/graphene nanocomposites compared to LaCoO3 nanoparticles is attributed to its abundant oxygen vacancies, synergistic effects between LaCoO3 and graphene advantages, and its relatively large surface area. More importantly, the LaCoO3/graphene nanocomposites exhibited good catalytic performance over a wide range of pH (from 3.0 to 9) and excellent reusability with consistent catalytic performance. The fabrication of LaCoO3/graphene composites prevents cobalt leaching and increases the content of Co2+ in the structure, thus leading to much higher catalytic activity than that of pure LaCoO3. Radical quenching experiment and electron paramagnetic resonance tests revealed that both radical pathways (SO4•–, •OH and O2•–) and non-radical pathways (1O2) contribute to pollutant degradation and 1O2 radical played a dominant role in the oxidation of pollutant.

The results demonstrate that the LaCoO3/graphene system could be potentially utilized towards peroxymonosulfate activation for environmental remediation.

FLASH COMMUNICATIONS

Branching Index: An alternative to characterize cellulose nano and microfibers by image analysis

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(1) Universidad Complutense de Madrid, (2) UC3M

Keywords | nanocellulose; morphology; image skeleton analysis; quality control; branching index

Cellulose micro/nanofibrils (CNFs) are produced from a wide variety of sources, using different types and intensities of treatments. This diversity makes necessary a proper characterization before its application beyond diameter or length. The morphology description using these parameters sometimes could be unreliable or meaningful, since CNFs need to be described in the network state they will be applied. Therefore, simple and quantitative methods are needed to characterize nanocellulose morphology that help to control the homogeneity of the products and the possible effect in the final applications.

We present a novel approach for CNF morphological characterization, the branching index (B.I.), based on the analysis of eroded CNF microscopy images [1]. The application of skeletonization to image objects results in another object which is an abstraction of the initial image (Fig. 1a). To measure B.I., images were first binarized using ImageJ (Fig. 1b), and then eroded until obtaining a fiber skeleton that contains both shape and topological structures of the original (Fig. 1c). The transformed object simplified down to lines and nodes can be more easily counted using different micrographs (Fig. 1d). The slope obtained from the linear regression of the curve nodes vs projected area was used as B.I. (Fig. 1e).

With this technique, we compare the ramifications of CNFs from different sources and treatments (Fig. 1f), describing quantitatively a lower impact of the sources in B.I, a significant effect of fiber peeling induced mainly by the mechanical pretreatments, the shortening of fibers with the reduction of nodes caused through enzymatic hydrolysis, and the formation of networks of shortened nanofibrils that is observed in CNFs after TEMPO-mediated oxidation with an exponential increase of B.I. As for the treatment severity we observe a slightly increase of B.I. at the same time the area projected is reduced. In addition, it was possible to differentiate between ramified fibers and networks through the disposition of the sample in the map nodes/area. The present method would be an easy tool to implement at industrial scale to monitor CNF quality and homogeneity.

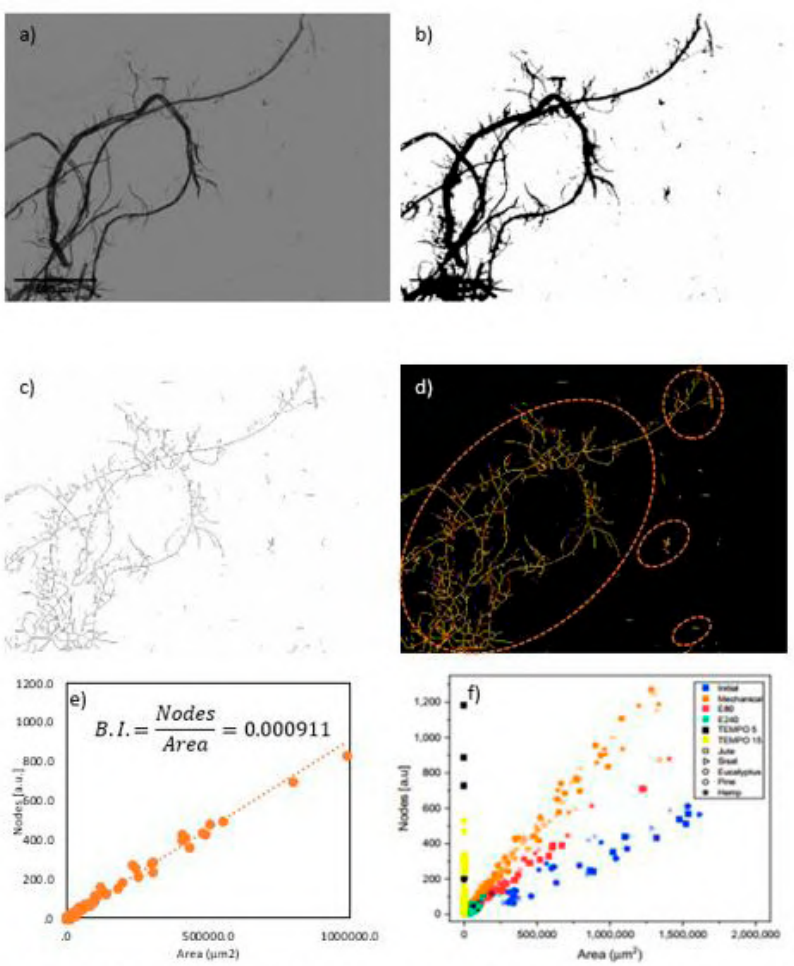


Figure 1. a) Initial image from optical microscope; b) Binarized image; c) Eroded image; d) Accounting of particles and nodes; e) Representation of nodes vs. projected area; f) Comparison of different CMF/CNFs samples

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Selection of nanoparticles as fillers in sustainable mixed matrix membranes

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Keywords | Mixed matrix membranes; biopolymers; nanofillers; sustainability

Membrane technology is already viewed as a strong clean efficient alternative and combination with other processes to decarbonize and clean chemical industries. The performance of membranes is usually undermined by the uncertainty of the stability of novel materials in the presence of impurities in real separation or reactor applications and the trade-off between their intrinsic properties, such as the permeability (production) and selectivity (separation factor) in gas separation or water uptake and ion exchange capacity or conductivity in fuel cells and batteries. The introduction of inorganic nanoparticles (zeolites, metal nanoparticles, ceramic oxides or graphene and other layered materials) in a polymer has been observed to enhance synergically the conductive or separation properties of the latter, even compared with commercially available membrane materials. The substitution of conventional polymers by biopolymers or low-cost biodegradable polymers in the formulation of membranes and film coatings is being studied in our group regarding their potential for CO2/CH4 separation (Casado-Coterillo et al. 2020), membrane-coated electrodes (Marcos-Madrazo et al. 2019) and antifouling properties (Torre- Celeizabal et al. 2022).

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High-throughput generation of aircraft-like soot

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Keywords | soot, aircraft emissions, flame synthesis, specific surface area, porosity

High-throughput, laboratory reactors for generation of aircraft-like soot are essential to quantify the impact of such emissions on public health and climate change due to the high costs and limited access to aircraft engines. Flame spray pyrolysis (Fig. 1: schematic) is used here to generate high soot concentrations, up to 255 mg/m3, three orders of magnitude higher than those typically obtained by widely-used soot generators, such as premixed flame reactors (Kelesidis et al., 2020). This enables routine characterization of the soot specific surface area (*SSA*) and pore size distribution by N2 adsorption (Fig. 1: lines). The geometric mean mobility diameter, *dm*, of soot agglomerates was systematically varied from 15 to 180 nm by varying the equivalence ratio (EQR) at constant fuel feed rate. The distribution of the primary particle diameter, *dp*, as well as the mass-mobility exponent, *Dfm*, measured here reveal that soot primary particles were sinter- bonded by surface growth (Kelesidis et al., 2017a), in agreement with power laws derived by discrete element modeling (Kelesidis et al., 2017b). Most importantly, soot made at EQR ≤ 1.34 has mainly small pores (< 2.5 nm; Fig. 1) and similar morphology, *SSA*, *dm* and *dp* with those

from high thrust aircraft emissions.

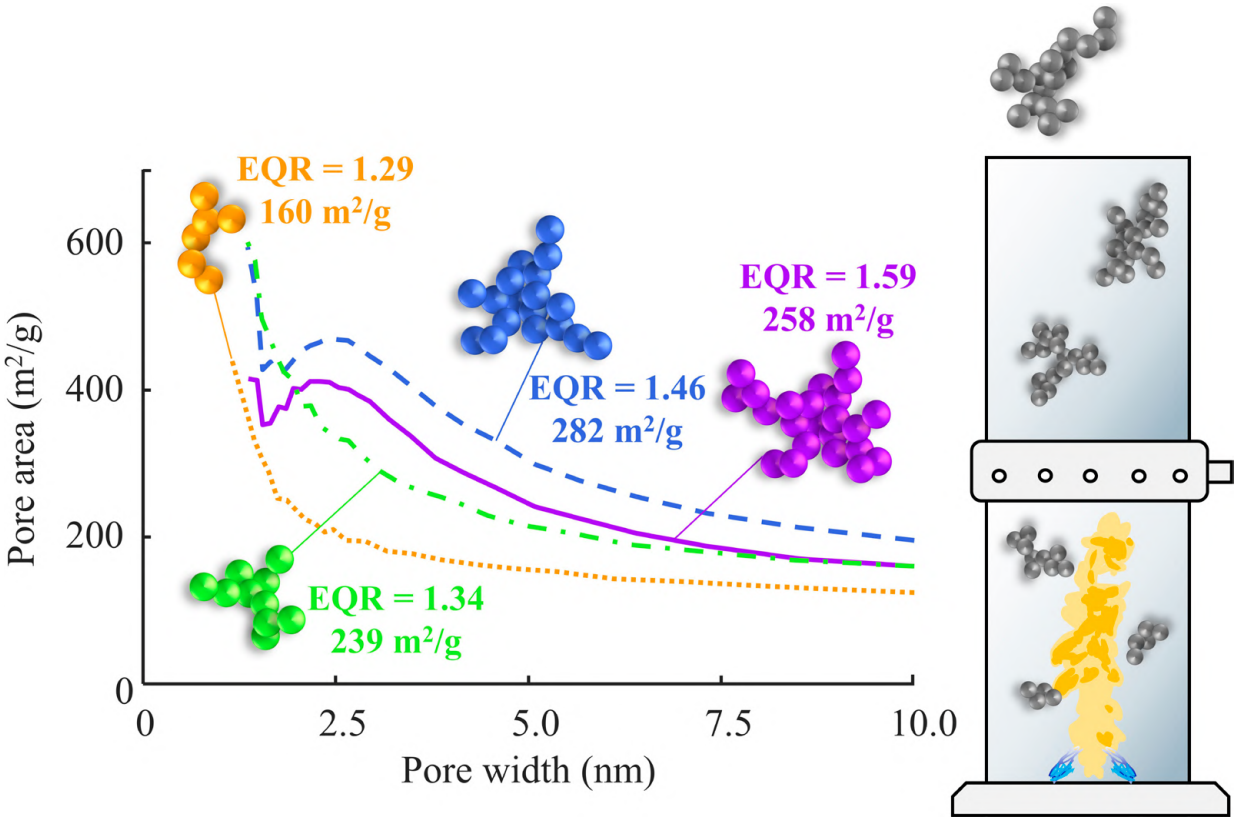


Figure 1. Pore surface area as a function of pore width of soot made here by flame spray pyrolysis of jet fuel (schematic) at EQR = 1.59 (solid line), 1.46 (broken line), 1.34 (dot-broken line) and 1.29 (dotted line).

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Application of cellulose microfibrils for stabilization of pickering emulsions in food industry

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Keywords | cellulose microfibers; Pickering emulsions; sunflower oil; tomato souce;rheology; emulsification index; food applications

Cellulose microfibrils (CMFs) can satisfy the requirements for a sustainable and environmentally friendly particle emulsifier (Lu et al., 2021). The main aim of the research was to study the application of CMFs from cotton linters as a stabilizer of Pickering emulsions. In this case, the nanocellulose-stabilized Pickering emulsions are prepared with different oil-in-water (O/W) ratios by mixing edible oil (olive or sunflower) and different concentrations of CMFs. The dispersion of the nanocellulose fibers in the water suspension, before their addition, has a significant effect on the final stabilization potential. Finally, the effects of using CMFs as stabilizer in food emulsions formed with tomato and olive oil are studied, which open a new application field in the food industry.

The results show that at low O/W ratios two phases are distinguished, the aqueous phase and the oil phase, which are completely separated due to the CMFs enclosed by the oil droplets as shown in **Figure 1**. From the 60/40 ratio, a higher oil content hinders the formation of small droplets and promotes coalescence.

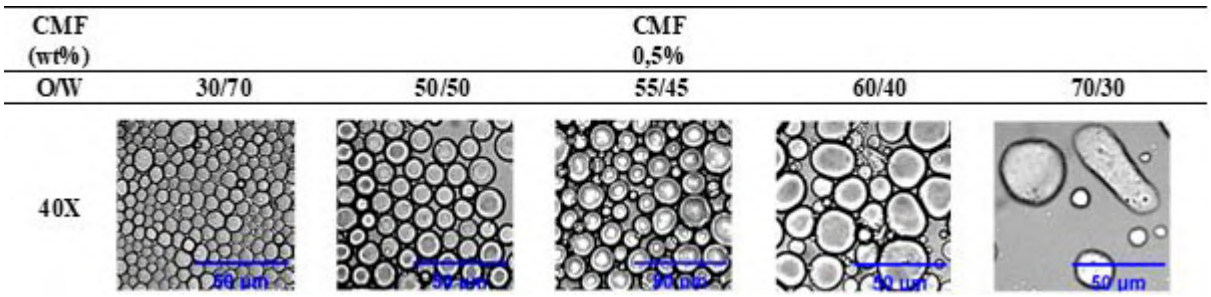


Figure 1. Optical microscopy images of emulsion droplets prepared with sunflower oil at 40X

Comparing Pickering emulsions prepared with different types of oil (olive and sunflower), it has been observed that the apparent viscosity of olive oil emulsions is slightly higher than sunflower oil emulsions. However, the average droplet diameter in both cases is similar, around 8 – 11 µm.

Finally, the results obtained show that the CMFs also can stabilize the oil/tomato sauce emulsions. The apparent viscosity of this system is higher than previous cases due to the presence of tomato constituents.

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Acknowledgment

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Rheological properties of micro-/nanofibrillated cellulose based coating formulations

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Keywords | Coatings, Micro-/nanofibrillated cellulose, Rheology

Micro-/nanofibrillated cellulose (M/NFCs) based coatings have emerged in papermaking due to their excellent strength, printing and barrier properties, but their usages in papermaking are still limited due to the lack of compatibility with other typical coating components. A systematic approach can overcome this issue thus enhancing the uniformity of coating formulations and resulting into better surface and barrier properties. In this regard, the rheological properties were measured in the present study to identify the best suitable concentration of starch-based M/NFCs coating formulations.

M/NFCs were produced using four different pretreatment methods: mechanical, enzymatical, TEMPO-mediated oxidation and cationic. Native corn starch was used as host component for M/NFCs coatings and cooked together for preparation of coating formulations with varying M/NFCs concentrations. Four different concentrations of M/NFCs were used to study the rheological properties while maintaining total solid concentration constant. Apparent viscosity for these formulations was determined using Haake, Model RS1, Germany at 50 °C. The formulations were sandwiched between a two plate-geometry, one of which was connected to a temperature control bath and the other to an instrument to apply the shear stress. The shear stress in the range of 0.5 to 300 Pa was applied, and flow curves were obtained using a Haake RheoWin 4.20.005 software.

It was observed that shear viscosity increased with increasing concentration of M/NFCs due to an increased entanglement of the fibers with concentration. The lowest shear viscosity and yield stress were observed for starch formulation, but both properties were increased with addition of M/NFCs in the formulation. While applying on the paper surface, it was also noticed that the lower concentrations with intermediate shear viscosity and yield stress of these M/NFCs formulations were suitable for achieving uniform coatings.

Electrochemical reactor for continuous CO2 valorization: synthesis and use of nickel carbon-supported nanoparticles to catalyze the oxygen evolution reaction

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Keywords | Ni-carbon supported nanoparticles, CO2 electroreduction, formate, continuous operation, oxygen evolution reaction

The electrocatalytic reduction of CO2 to formate is being considered as a promising strategy to store energy from intermittent sources of energy in the form of a value-added chemical. Great efforts have been addressed using diverse types of electrocatalysts in the cathode in form of different electrode configurations (Al-Tamreh et al., 2021). Commercial DSA/O2 or Pt-based-anodes have typically been employed as counter electrodes to carry out the oxygen evolution reaction (OER), but they are based on very expensive and scarce materials. Therefore, it is desirable to study new competitive electrocatalysts as anodes for the electroreduction of CO2 to formate for the future implementation of the process at an industrial scale. In this context, this communication aims at analyzing nickel-carbon-supported nanoparticles, which are synthesized and characterized, to carry out the OER for the electrocatalytic reduction of CO2 to formate in a continuous electrochemical reactor. Firstly, the OER activity of these materials is studied in a Rotative Disk Electrode by linear sweep voltammetry in terms of current per electrode surface area and per gram of the active metal. After the promising results obtained in the electrochemical characterization, anodes are fabricated by the deposition of nickel nanoparticles using airbrushing techniques. The configuration of the electrochemical reactor is similar to our previous approaches, using the same experimental setup and operating conditions (Díaz-Sainz et al., 2021). Employing a nickel loading of 0.75 mg·cm-2 and with a supply of the current density of 90 mA·cm-2, similar results are obtained in terms of formate concentration (7.5 g·L-1) and Faradaic Efficiency for formate (90 %), similar to those obtained previously in the research group using a DSA/O2, but employing less expensive and at the same time, more abundant particles as anodes.

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Acknowledgments

The financial support from the Spanish State Research Agency through the projects PID2019-108136RB-C31, and PID2020-112845RB-I00 (AEI /10.13039/501100011033) is gratefully acknowledged.

The “CCU+OX” coordinated project: development of processes for capture and electrochemical valorization of CO2 coupled to useful electro-oxidations

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(1) Universidad de Cantabria, Dpto. Ing. Química y Biomolecular, (2) Universidad de Alicante, Instituto de Electroquímica, (3) Universitat de Barcelona, Dept. Ciència de Materials i Química Física

Keywords | CO2, capture, optimization, membrane technology, electrochemical conversion, particle catalysts

The project “Development of processes for capture and electrochemical valorization of CO2 coupled to useful electro- oxidations (CCU+OX)”,

funded by the Spanish Ministry of Science and Innovation- State Research Agency (AEI), gathers three Spanish research groups with experience in chemical engineering (Universidad de Cantabria, UC), applied electrochemistry (Universidad de Alicante, UA), and materials science (Universitat de Barcelona, UB).

The CCU+OX coordinated project focusses on the development of new processes for CO2 capture and utilization under an approach of sustainability. For doing that, the project addresses optimization strategies for improving the separation of CO2 from gaseous waste streams, using materials with better performance, like biopolymer-based membranes. Moreover, the project also works on finding new catalytic materials and configurations for the electrochemical reduction of CO2 to obtain value-added products in an efficient way.

This communication will summarize the main research activities and progress made in the framework of the project, with special emphasis on the role that particles are playing as catalysts for the development of electrodes with improved performance. Traditionally, in studies on CO2 electroreduction, efforts have been focused so far on the cathodic compartment where CO2 is converted to the products of interest, simply leaving that in the anodic compartment a reaction like oxygen evolution (i.e. OER, oxidation of water to give molecular oxygen) can take place. In this sense, one of the most innovative aspects of the project is the exploration of more relevant alternative oxidative reactions to the OER that allow the co-valorization of CO2 and bio-based products. Particularly, the selective oxidation of glycerol, which is an important by- product from biodiesel production, is being studied in the project. Promising results of our on-going research will be presented on the use of anodes prepared with Pt-based nanoparticles for glycerol oxidation, when coupled to CO2 reduction in continuous flow-by reactors, which show that C3 oxidation compounds of very high value, like dihydroxyacetone (DHA), can be obtained as co-products in the anodic compartment.

Acknowledgements: This project is financially supported by the Spanish State Research Agency (PID2019-108136RB-C31, PID2019-108136RB-C32, PID2019-108136RB-C33 /AEI /10.13039/501100011033).

9 MODELLING AND SIMULATION

KEYNOTE LECTURES

News on modeling cohesive powders: from compression to tension

Luding, Stefan (1)
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Keywords | cohesive powders, compression, tension, contact models for particle simulations

News on modeling cohesive powders: from compression to tension

Recent experiments of cohesive powders allow to calibrate and validate DEM particle simulations in a variety of different states, from low to high deformation rates, and from low to high confining stress -- where both ends have to be extended towards tension (negative) as well as extremely high rates and stresses (tableting).

Measurement and prediction of cohesive powder behavior are found to be very challenging, and different element test devices can typically only access a narrow regime in the state space of deformation rates and confining stresses.

Experiments like shear testers are considered, covering mostly the low to intermediate regime, while pile formation, free surface drum flow sit on the lower ends of state space. To extend the regime, either more massive (geotechnical) testers or other devices are needed to access extreme situations, such as tableting or tensile testing. Studies to build a wide experimental database are ongoing, using reference materials but also industrially more relevant ones, to improve testers and eventually allow for calibration and validation of discrete models for fine, cohesive powders.

Particle simulations often rely on primary particles, which is very limiting in case of fine and thus cohesive powders, due to the enormous particle numbers. Therefore, the particles are often up-scaled to meso-scale, where a single particle/parcel represent many primary particles, reducing the total number needed to conveniently low numbers. While applications like flow/dosing can be well modeled, there remain problems related to the geometry, narrow gaps, related to contact behavior at very high rates and/or compression stresses, as well as under tension.

Various situations are discussed and reviewed, and some solution approaches are proposed for the different issues that are observed. For example, i) the cohesion between particles requires a serious, reliable upscaling rule or empirical calibration, using the good dimensionless quantities; ii) steady-state flow rheology requires valid constitutive relations in non- dimensional form that account for rate, stress, and other effects like creep or walls; iii) extremely high compression rates and stresses reveal non-linearities that are not contained in advanced contact models.

State-of-the-art modeling of computational granular dynamics for a simulation-based digital twin

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(1) Room 327, Faculty of Engineering Building 8

Keywords | simulation-based digital twin, discrete element method, signed distance function, FELMI

The fourth industrial revolution is declared, and the interest in developing digital twins for engineering systems is rapidly growing. The digital twin virtually represents a physical system. In the construction of the digital twin, modeling and simulation as well as data science has been paid attention to. This trend is not the exception in the powder industries, where the numerical modeling should be introduced positively. This is so-called the simulation-based digital twin. Accordingly, the digital twin requires efficient, stable, and accurate techniques to reproduce the

physical twin. Towards the construction of the digital twin for the powder industries, the author's group has developed numerical models and data science techniques. In order to simulate the actual phenomena of the particles efficiently, the coarse-grained discrete element method (DEM) [1] has been proposed, where the coarse-grained DEM can reduce the number of particles drastically. An implicit algorithm related to the drag force term [2] has been proposed to simulate the solid-fluid mixture systems stably. A new capillary force model [3] has been developed to simulate the wet-particle behavior accurately, where the toroidal approximation [4] is employed. Adequacy of these models has been proven through validation tests. Besides the modeling, a reduced order model (ROM) based on the proper orthogonal decomposition (POD) [5] has been developed very recently. The ROM has sufficient benefits to reproduce the actual phenomena without computing the DEM simulations. The above approaches will become promising in constructing the digital twin in powder industries.

This study has been financially supported by JSPS KAKENHI (17KK0110, 21H04870, and 21K19760)

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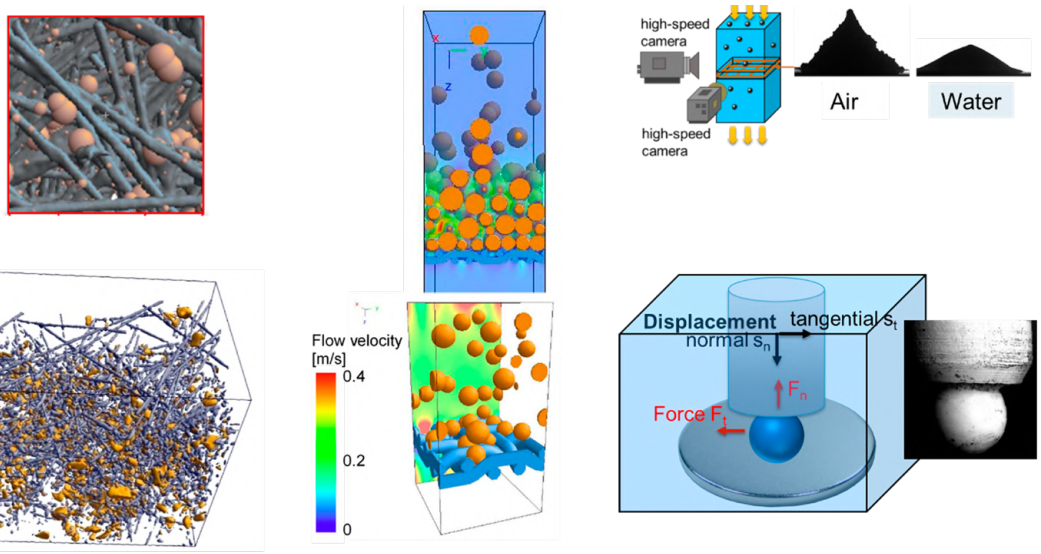
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CFD-DEM simulation of particle separation processes and experimental methods for the parameter estimation and validation

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(1) Technical University of Kaiserslautern

Keywords | CFD-DEM coupling, particle separation, contact model, experimental estimation of particle contact properties

In the last three decades, microscopical approaches for the simulation of particle-fluid separation have been developed as a powerful tool that can help to understand complex separation mechanisms of particles in fluid flow or filter media, support the model-based optimization of existing and the design of novel separation processes. While all the methods calculate single particle transport and deposition, they differ however in the resolution of the flow, consideration of physical effects, particle-fluid and particle-particle interactions, as well as the computational effort. This contribution first gives a short overview of different methods and their suitability and challenges for the description of the separation of fine particles are discussed.



The focus of the performed studies is on the detailed description of the particle separation in complex nonwoven structures and in a filter cake by considering micromechanisms and real filter medium microstructure. For low particle concentrations, the developed model (Kerner

et al.) is based on the Computational Fluid Dynamics (CFD) of the resolved flow in the nonwoven structure and Lagrangian particle tracking (Figure). In the case of high concentrations, the adhesive nonelastic particle interactions and dynamic viscous forces as well as bridging mechanisms by clogging of pores must be considered to predict the filter cake formation. These microprocesses were calculated by the resolved coupling of the Discrete Element Method (DEM) and CFD (Puderbach et al.), Figure. A particular challenge for the CFD-DEM simulation of aqueous suspensions is the estimation of contact parameters considering viscous forces. Many methods that are used for dry particles are not applicable in liquids. Therefore, some measurement methods for the parameter estimation and validation of the DEM model were developed and will be explained with examples of experiments.

Figure: Simulation of particle deposition in nonwoven (left) and cake formation (middle); experimental tests (right): heap formation and nano/tribo indentation in a liquid cell.

Kerner, M. et al.: Evaluation of eles

Virtual Formulation Laboratory for prediction and optimisation of manufacturability of advanced solids based formulations

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(1) University of Leicester

Keywords | upscaling, particle properties, bulk behaviour, flow, segregation, caking, compaction

Virtual Formulation Laboratory (VFL) is a software tool for prediction and optimisation of manufacturability and stability of advanced solids-based formulations. Four processes are considered: powder flow, mixing, compaction and storage. VFL predicts manufacturability problems quantified by suitable manufacturability indicators and accounts for a range of material types, particle structures and blend systems to enable the formulator to test the effects of formulation changes in virtual space and check for potential problems covering manufacturing difficulties experienced in production plants. This talk focusses on upscaling and linking particle properties to bulk powder behavior covering flow, segregation, caking and compaction.

Assessing the influence of surface energy heterogeneity on dry powder coating performance using DEM

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(1) Newcastle University, (2) Imperial College London

Keywords | Dry powder coating, surface energy heterogeneity, DEM

Dry powder coating is increasingly being employed in industry to improve manufacturability of powders due to its favourable benefits over the conventional solvent-based techniques. This surface modification process offers reduced energy consumption and zero emission of volatile organic solvents leading to improved process safety and less environmental impact. Moreover, it is well suited to moisture sensitivity materials, particularly pharmaceutical excipients in drug manufacturing. However, most pharmaceutical powders exhibit anisotropic surface energy as revealed by experimental characterisation using Finite Dilution Inverse Gas Chromatography (FD-IGC) (Karde *et al*., 2022). Currently, there is lack of studies on the influence of anisotropic surface energy on dry powder coating efficiency. In this study, different approaches are employed to implement surface energy heterogeneity for discrete host particles in the Discrete Element Model (DEM) via the bonding model and particle orientation. Surface energy distribution is applied on the heterogeneous surfaces of host particles based on deconvoluted FD-IGC experimental measurements. The influence of anisotropic surface energy on coating performance in an impeller-agitated system is investigated. Analysis of the evolution of the guest-host particle interactions shows that guest particles preferentially adhere to the higher energy sites, as indicated by the higher proportion of guest- host particle contacts compared to lower energy sites (Fig. 1b). Understanding such complex guest-host interactions provides useful insight on the role of heterogeneous surfaces towards achieving uniformly distributed coatings.

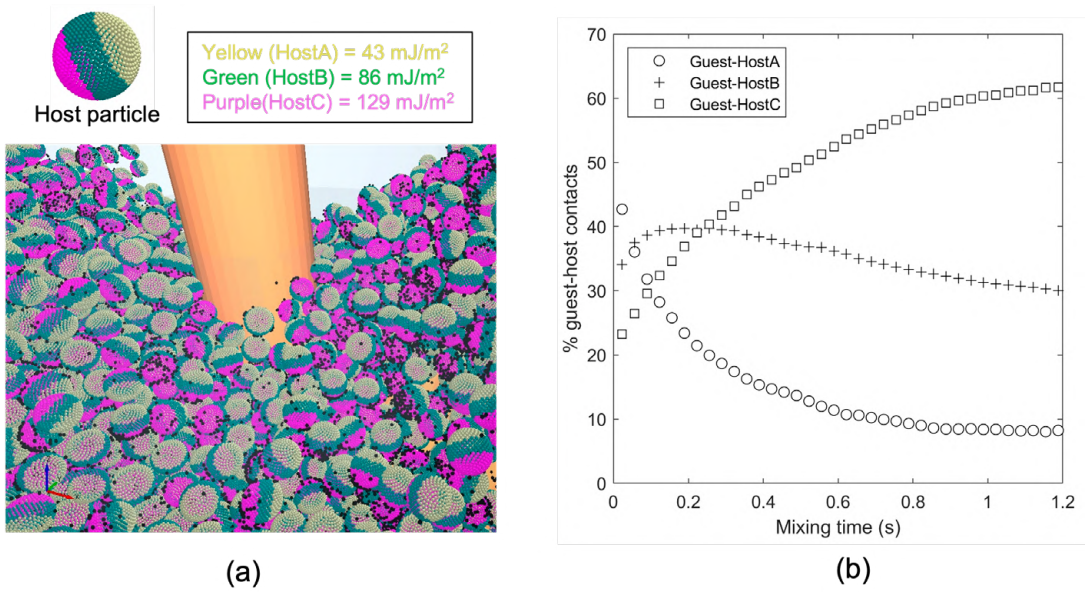


Fig. 1. (a) DEM snapshot of coated host particles with heterogenous surface energy and (b) evolution of the guest-host particle interactions in the system

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Simulation of Ni-Mn-Co Hydroxide Co-Precipitation in a Continuous Stirred-Tank Reactor by adopting CFD and Population Balance Modelling

Shiea, Mohsen (1); Querio, Andrea (1); Buffo, Antonio (1); Boccardo, Gianluca (1); Marchisio, Daniele (1)
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Keywords | Co-precipitation; Population balance modelling; Quadrature method of moments; Ni-Mn-Co Hydroxide; Lithium- ion battery

Recent trends in the lithium-ion market confirms the growing demand for cathode materials based on Ni-Mn-Co hydroxide precursors, which in turn highlight the importance of the ongoing research on the synthesis of these precursors. While the literature offers numerous experimental works that investigate the effect of synthesis conditions on precursor properties, e.g., particle size distribution (PSD), only few studies have been conducted to predict particle properties through simulation. Here, we present a modelling framework that couples the CFD with the population balance modelling to predict the PSD of Ni-Mn-Co hydroxide particles produced in the presence of ammonia. The modelling framework consists of several pieces: 1) the CFD that predicts the velocity and turbulent fields; 2) a chemical equilibrium solver that calculates the local supersaturation generated by transported total concentrations; 3) a population balance equation (PBE) that describes the formation and evolution of particles due to the nucleation, growth, aggregation and breakage; 4) the quadrature method of moments for solving the PBE; 5) transport equations for total concentrations to determine the local composition; 6) a micromixing model to consider the segregation of feed solutions. As a case study, a three-liter pilot-scale CSTR (depicted in Figure 1a) is simulated by implementing the framework in OpenFOAM software. Figure 1b shows the predicted steady-state PSD at the outlet of the reactor for some selected conditions. It should be noted that PSD predictions depend highly on the kinetic models employed for the co-precipitation process, however, such parameters can be identified by conducting appropriate experiments, and matching predictions with measurements. Once the kinetic parameters are known, the developed framework can serve as a promising predictive tool to simulate the co-precipitation of Ni-Mn-Co hydroxide, which is of great usefulness for the process optimization and scale-up.

Acknowledgement

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views of the European Commission/Research Executive Agency.

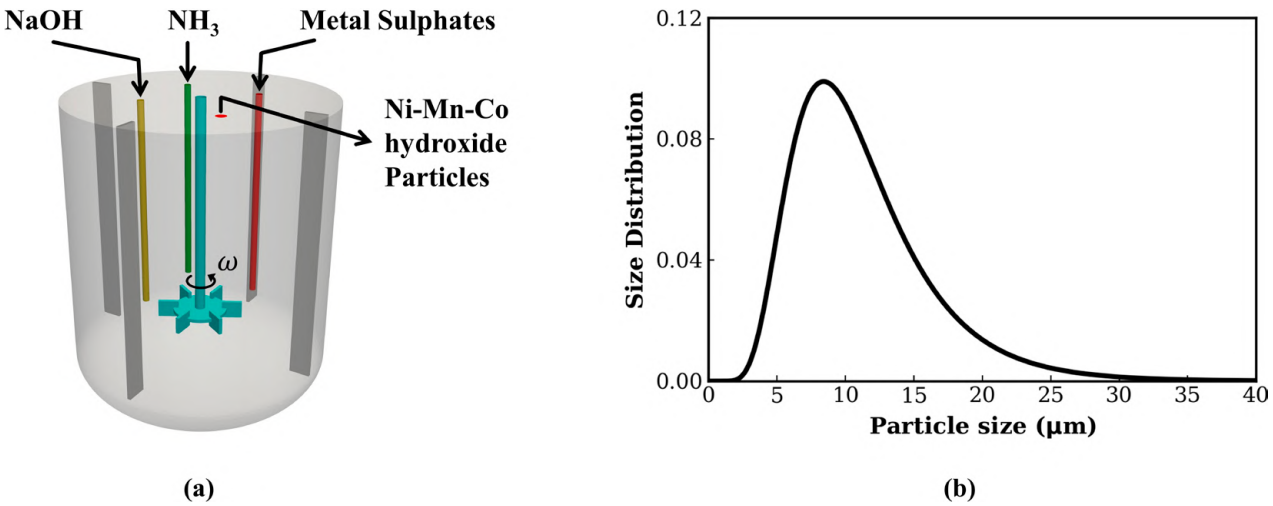


Figure1: (a) simulated CSTR; (b) steady-state reconstructed PSD at the reactor outlet

Numerical investigation of ironmaking blast furnace with simultaneous injection of hydrogen through shaft and hearth tuyeres

Li, Jing (1); Kuang, Shibo (1); Zou, Ruiping (1); Yu, Aibing (1)
(1) Monash University

Hydrogen blast furnaces (HBFs) are helpful to reduce the carbon footprint of the BF ironmaking process. However, there exists a hydrogen usage limit in an HBF. How to lift such a limit represents a focus of HBF R&D. This talk will present our continuous efforts in this direction. Aiming to increase the hydrogen usage limit, the simultaneous injection of hydrogen through both hearth and shaft tuyeres into a 380-m3 industrial BF is studied. This is done by a recently developed 3D BF process model. The model is validated under different conditions. On this basis, the effects of shaft-injected gas flow rate on the inner states and overall performance of HBF are analyzed in detail. It shows that the simultaneous hydrogen injection through hearth and shaft tuyeres can double the coke rate reduction compared with the case with only the hearth injection against the base case without hydrogen injection. A gas tracking method is used to track different gas components for hearth-generated and shaft-injected gases and quantify their respective contributions to BF performance. The results show that the CO plays a dominating role in reducing iron ore; the hearth-injected H2 is in second place, and shaft-injected H2 is the least important contributor under the conditions considered. Overall, the hearth-injected H2 is more helpful for improving iron ore reduction, while the shaft-injected H2 is better to heat burden materials. The results suggest that the CFD process BF model can be an effective tool to explore HBFs.

Particle shape effects in granular material using GPU DEM.

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(1) RCPE, (2) TU Graz, (3) Institute of Agrophysics PAS

Keywords | DEM, Particle shape, Polyhedral, GPU, Industrial Scale

Particle shape plays a crucial role in resulting macroscopic behavior of granular material yet the majority of DEM simulations using simple spheres with non-physical constructs such as rolling friction without fully understanding the implications thereof. While there have been shape studies using super quadric's and ellipsoids over the years, they still have the same single point contact resolution as spheres. The majority of granular materials are however non-curve linear and have distinct edges and flat faces that is best captured by polyhedral shapes. Apart from the mechanical behavior, thermal conduction as well as cohesion between real particles occurs over a finite-contact area which

is accurately captured by polyhedra. In this talk the role of particle shape on the shear strength and flow behavior of cohesive granular materials will be explored using a new contact law for polyhedral particles developed by the authors. In addition a number of industrially relevant cases concerning mixers, screw feeders and mills will also be presented.

How can we simulate real powder handling processes?

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(1) Department of Chemical Engineering, Osaka Metropolitan University

Keywords | numerical simulation, coarse grained method, parameter calibration

Powders have been widely used in the manufacturing processes of many industries. To achieve better manufacturing efficiency and product quality, understanding of the powder behavior is of great importance. Due to the rapid progress in computer performance and science technologies, numerical simulation have become reliable and promising tool for the analysis and rational design of powder handling processes. However, many issues still remain unsolved when it comes to the practical application. For example, simulation of large equipment scale or fine powder handling requires computation of extremely large number of powders. Also, it is still not easy to imitate the real powder properties.

This talk discusses how we can effectively simulate the real powder behavior in the powder handling processes. In DEM (Discrete Element Method), a coarse grained (CG) method, which uses artificially scaled-up larger particles, is developed and performance of the simulation is demonstrated when it applies to a dense granular shear flow. Also, a novel calibration procedure for DEM parameters in case of using fine cohesive powders is proposed based on dynamic and static angle of repose measurements. We also present DEM simulation of powder compression for real powders having elasto-plastic, adhesive and bimodal size distribution. Numerical approach of scale-up of power mixing and prediction of granule growth in wet granulation is also introduced.

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ORAL COMMUNICATIONS

Simple Modeling of the Agarose Gel Electrophoresis of Spherical and Rod-shaped Au Nanoparticles in Dependence on the Mesh Size

Barasinski, Matthäus (1); Hilbig, Julia (1); Neumann, Stefan (2); Rafaja, David (2); Garnweitner, Georg (1)
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Keywords | Separation, Nanoparticles, Synthesis, Functionalization, Agarose gel, Model, Migration

Agarose gel electrophoresis is well-known for separation of biomolecules and nanoparticles (NPs) according to their size and surface charge via different rates of migration within an externally applied electric field. The mesh size of the three- dimensional gel network thereby is a dominating parameter for the migration of NPs. Previously, we could show that depending on the mesh size, spherical SiO2 NPs migrate under two migration regimes (Barasinski et al., 2020). If the mesh size is close to the particle size the migration of the NPs is restricted and their movement is slowed down significantly due to steric and hydrodynamic NP-mesh interactions. In contrast, if the mesh size is larger than the particle size, the NPs exhibit unrestricted migration through the gel according to their surface charge. In this case the gel does not significantly disturb the migration of the NPs and just prevents their diffusion. This particle movement in the electric field can be expressed

as the electrophoretic mobility and mathematically described by Henry's equation:

$$\mu_E = \frac{2\varepsilon_{r,m} \cdot \varepsilon_0 \cdot \zeta}{3\eta} \cdot f(Ka) \left[\frac{m^2}{s \cdot V}\right]$$

$\varepsilon_{r.m}$ - relative permittivity of the medium
ε_0 - permittivity of the vacuum
η - dynamic viscosity of the fluid
$f(Ka)$ - Henry function

In this study, spherical and rod-shaped Au NPs with various particle sizes (< 70 nm) were synthesized via the seeded growth method, as shown in the transmission electron microscopy (TEM) images of Fig. 1 (left). These samples were introduced into a 1 wt.% agarose gel and a voltage of 100 V was applied. Fig. 1 (center) shows the samples after a 20-minute migration time, with the NPs visible as colorful bands within the gel. Appling Henry's equation to model the migration velocity, it is obvious that the calculated migration distances v (dashed lines) in Fig. 1 (right) do not meet the experimentally obtained values (points). The retardation imposed by the gel mesh is not sufficiently addressed by Henry's equation and therefore, a migration factor is proposed in a new model, setting the mesh size in relation to the particle size as well as the particle morphology. This new model results in a more accurate prediction of the migration velocity v* (straight line), as proven in total with four samples for varied mesh sizes.

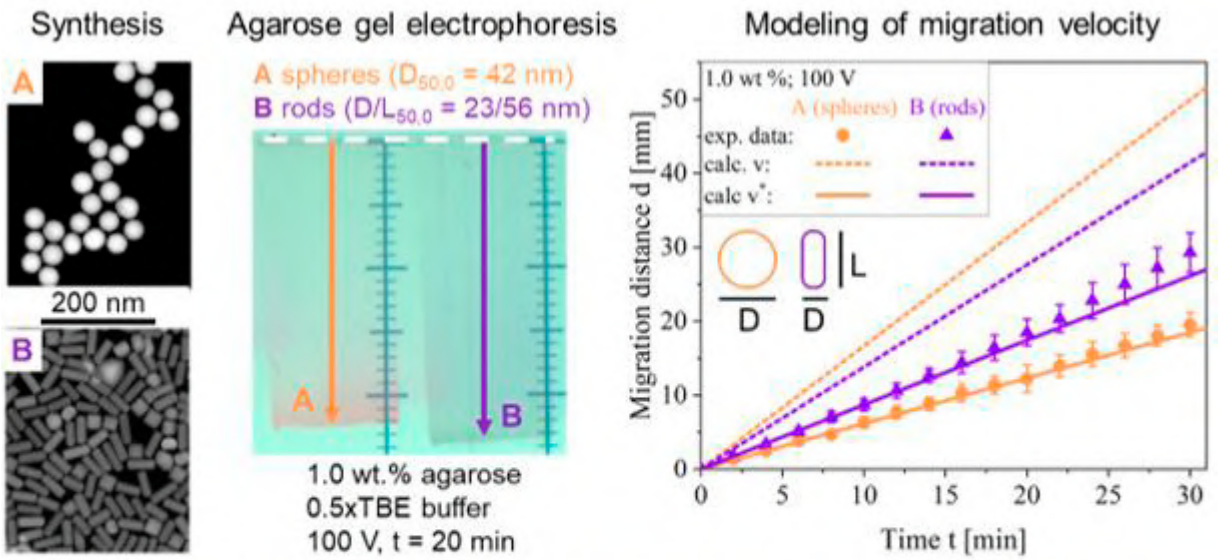


Figure 1: Exemplary TEM images of rod-shaped and spherical Au nanoparticle samples A and B after synthesis (left), samples during the agarose gel electrophoresis (white dashed line represents the starting points, arrows show the migration distance for each sample; center) and a graph showing the migration distance vs. time for the experimentally obtained data (points) and the calculated distances v according to Henry's equation and v* according to our new model.

Barasinski, M., Garnweitner, G., Restricted and unrestricted migration mechanisms of silica nanoparticles in agarose gels and their utilization for the separation of binary mixtures. *J. Phys. Chem. C* **2020**, 124, 5157–5166

Multiphase Mixture Theory for Turbulent Flows

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Keywords | Cauchy stress, Reynolds stress, frame insensitive, frame sensitive, multiphase fluids

A coupling between the velocity field and the Coriolis field of a single phase fluid can redistribute turbulent kinetic energy beyond the Kolmogoroff scale, even if the mean turbulent flow is spatially homogeneous (Koppula, 2011). The purpose of this presentation is to extend this observation to a multiphase fluid. The Coriolis Theorem predicts that the material strain rate is frame insensitive (i.e., objective); and, the Reynolds stress is frame sensitive (i.e., non-objective). Consequently, the Cauchy stress and the Reynolds "stress" are not similar for single

phase or multiphase fluids. For single phase fluids, turbulent dispersion and turbulent dissipation are positive. However, the “production” of kinetic energy may be either positive or negative (Churchill, 1996; and, Pope, 2000). For a second-order, irreducible, compressible, Newtonian fluid, the Cauchy stress and the strain rate are symmetric and objective. The phenomenological coefficients for this model are objective scalar-valued functions of the local thermodynamic state and the eigenvalues of the local strain rate. For some materials, the Cauchy stress is asymmetric and objective (Dahler, 1963). The Reynolds average of the equation-of-change for linear momentum is an exact, albeit unclosed, vector-valued equation for the mean velocity field of a single-phase fluid. Unlike the Cauchy stress, the Reynolds “stress” is a dyadic-valued, real, symmetric, non-negative, and non-objective operator for all inertial and non-inertial temporal frames-of-reference. Turbulent flows of multiphase fluids are encountered ubiquitously in industry. The mixture model presented herein will include equations-of-change for mass, linear momentum, angular momentum, energy, and entropy. The multiphase mixture theory is developed by using a phase average followed by a Reynolds average. Churchill, S.W., 1996, Critique of Predictive and Correlative Models for Turbulent Flow and Convection, Ind. & Eng. Chem. Res., 35, 3122. Dahler, J.S. and L.E. Scriven, 1963, Theory of Structured Continua. I. General Consideration of Angular Momentum and Polarization, Proceedings of the Royal Society of London. Series A. Mathematical and Physical Sciences, Oct. 29, Vol. 275, No. 1363, pp. 504-527. Koppula, K.S., et al., 2011, Turbulent Energy Redistribution in Spanwise Rotating Channel Flows, Ind. & Eng. Chem. Res., 50, 8905-8916. Pope, S.B., 2000, Turbulent Flows, Cambridge University Press.

Simulation of emulsion rheology for higher than critical Capillary numbers

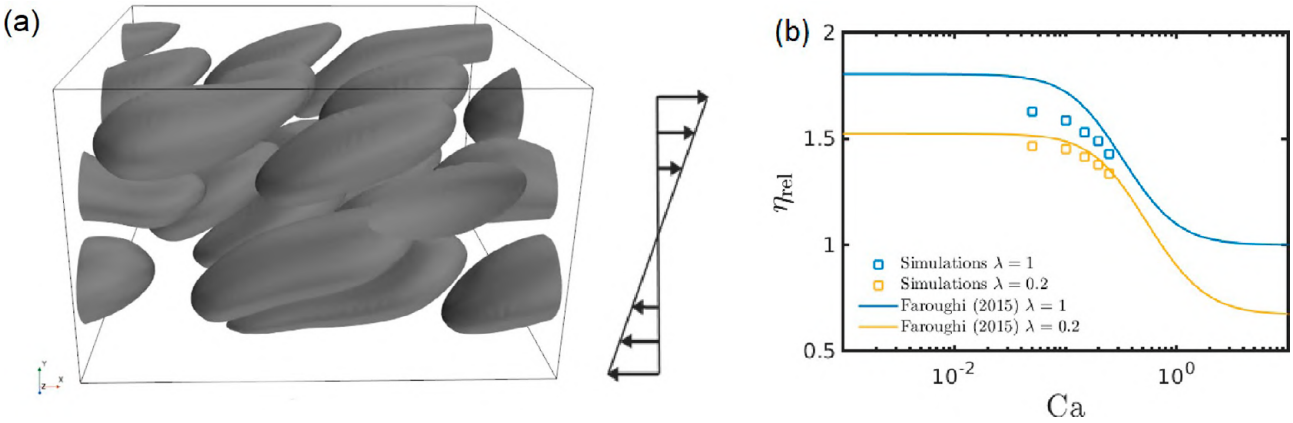
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Keywords | Emulsion, rheology, droplet break-up, volume of fluid

Rheology of fine emulsions for Capillary numbers $/left(/mathrm{Ca}/right)$ close to or higher than critical $/left(/mathrm{Ca_c}/right)$ are generally not accessible to conventional rheometers operating up to $10^3 - 10^4 //mathrm{s}^{-1}$. However, information about the rheological behaviour of fine emulsions at shear rates as high as $10^6 - 10^7 //mathrm{s}^{-1}$, i.e. around their $/mathrm{Ca_c}$, is crucial for modelling emulsification processes in high-shear devices. Semi-analytical rheology models typically assume small droplet deformation (Pal, 2003, Faroughi et al., 2015), and predictions of emulsion viscosity for high $/mathrm{Ca}$ is performed by extrapolating predictions based on this assumption. Moreover, relevant simulation studies are usually restricted to $/mathrm{Ca} < \mathrm{Ca_c}$ (e.g. Zinchenko et al., 2015, Liao et al., 2017) .

We report a numerical study of the rheology of a two-fluid emulsion in semi-dilute conditions (up to 30% v/v). We use a volume of fluid method and a repulsive force model introduced by De Vita et al. (2019) to suppress droplet coalescence. In the original model, indexing of droplets is static and, therefore, the repulsive force cannot be applied to newly formed droplets, thus not allowing simulations for $/mathrm{Ca} > \mathrm{Ca_c}$. In this work, the model is implemented in the commercial software STAR-CCM+ (Figure 1a) and extended to $/mathrm{Ca} > \mathrm{Ca_c}$ by introducing dynamic indexing. We extract shear viscosity from the simulations by evaluating the various components of bulk stress (Batchelor, 1970). A comparison of viscosity predictions with a model from literature is shown in Figure 1b.

In the dilute limit of single-droplet simulations, exceeding $/mathrm{Ca_c}$ results to increase of viscosity. This behaviour suggests that viscosity adjusts to reduction (due to break-up) of droplet size which is included in the definition of $/mathrm{Ca}$. In semi-dilute systems, where multiple droplets are simulated, not all of them break up at the same $/mathrm{Ca}$. This induces two counteracting effects, (a) droplet break-up causing increase of viscosity, and (b) elongation and alignment of unbroken droplets leading to decrease of viscosity. We discuss the effect of dispersed phase volume fraction and viscosity ratio on the balance between these two effects in view of the quasi-Newtonian plateaus which have been observed experimentally (Dubbelboer, 2016) as $/mathrm{Ca}$ approaches $/mathrm{Ca_c}$.



A Discrete Differential Geometric Formulation of Multiphase Surface Interfaces for Scalable Multiphysics Equilibrium Simulations

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Keywords | Mean curvature, Surface tension, Multiphase surface interfaces, Three-phase contact angle, Discrete differential geometry (DDG)

In many multiphase systems, such as nanoparticle films exposed to humid air, surface tension forces dominate over viscous forces. In these systems a model of the capillary interactions can be reduced to a surface interface curvature-driven mechanical problem describing the gas-liquid-solid interactions. Accurate estimates of the mean curvature of the phase interfaces are essential for simulating such systems reliably. For many complex systems, conventional numerical methods formulated in Cartesian coordinates are incapable of simulating the system over the space and timespans of interest (Endres, Ciacchi, Mädler, 2020) due to the large number of discrete elements needed. Here we demonstrate how a novel development built on recent advances from the field of discrete differential geometry that can be applied to greatly reduce the computational resources required in such simulations.

A new, generalised formulation which builds on the conventional cotan-formula (Grinspun et al. 2006) was developed that can be used to reconstruct the exact mean normal- and geodesic curvatures of convex interfaces and discretised three- phase contact angles in equilibrium. Accurately modelling these surface interfaces is essential to many scalable systems of interest, especially in nanoparticulate systems where there is little validation of physical forces arising from surface tension available. Our formulation can also provide error estimates needed for a particular mesh refinement to retain a predetermined accuracy in the simulations. In order to validate the method three test cases of surface energy minimization flow in three-phase systems were developed:

1. Capillary rise in a tube (modelled as an idealised spherical cap).
2. Particle-particle liquid bridges (modelled as ideal Catenoids).
3. Sessile microdroplet (comparisons to non-ideal physical experiments).

For all these test cases, near exact results could be demonstrated even for very sparse refinements of the mesh.

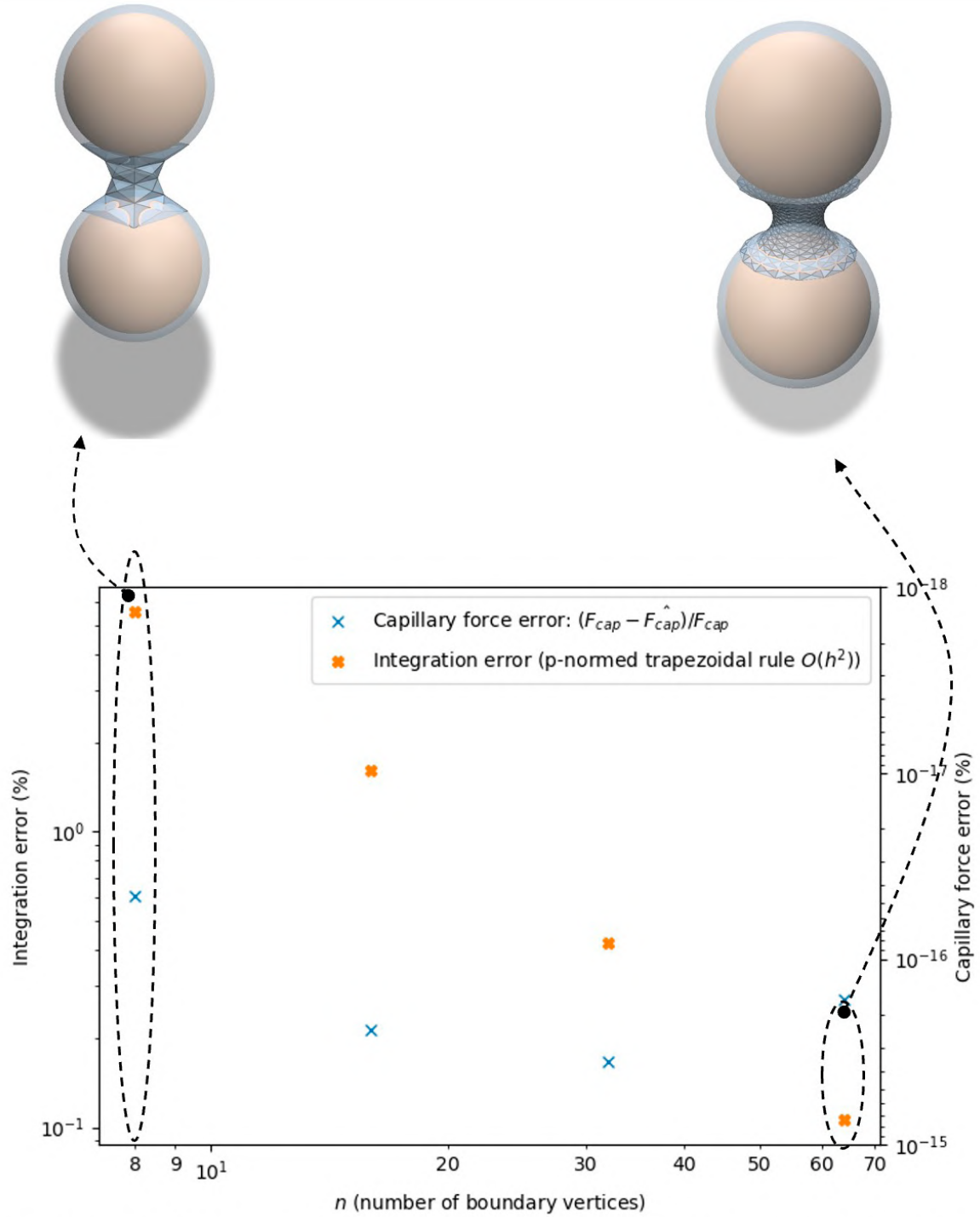


Figure 1: The Capillary force errors of the numerically calculated force when compared to the analytical result under increasingly detailed refinements of the surface. Also shown is the integration error (calculated using the p-norms over the surface).

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Approximating the van der Waals interaction potentials between agglomerates and their coagulation enhancement effect

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Keywords | Agglomerates; Coagulation; van der Waals

The van der Waals (vdW) potentials play an important role in powder handling, filtration, resuspension, restructuring, and agglomeration. While vdW potentials between spherical particles and their collision enhancement effect are well known (Ouyang *et al*., 2012), interactions between agglomerates are still poorly understood (Babick *et al*., 2011).

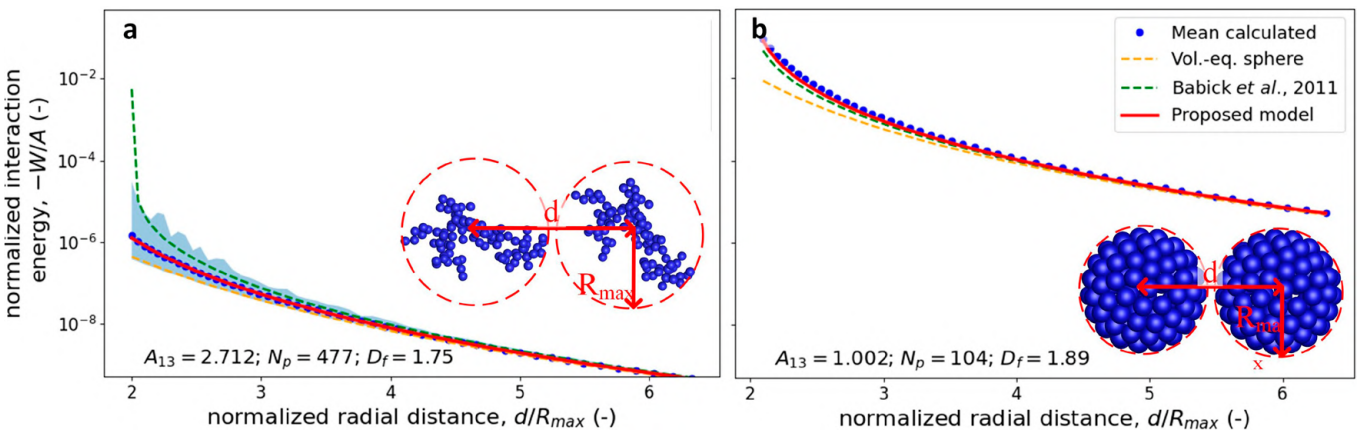


Figure 1: The vdW potentials between a pair of (a) DLCA, and (b) raspberry-like agglomerates versus their center-to-center distance (anisotropy coefficient A_13; number of monomers N_p; and fractal dimension D_f; are indicated).

The vdW interactions between a pair of hollow raspberry-like agglomerates (primary particles in a 2d spherical configuration) made with a sequential algorithm and a pair of diffusion-limited agglomerates (DLCA) generated by a discrete element code (Morán *et al*., 2020) are studied. The role of fractal-like structure, anisotropy, and finite size on the resulting vdW potentials is investigated. Predictions of the equation derived in this work are compared with Mean Calculated, Babick *et al*., 2011 and simple functions based on volume-equivalent spheres (see Fig. 1). These equations are used to predict the coagulation enhancement of agglomerated powders due to the vdW potentials and the results are validated by comparison with first- time passage translational and rotational Langevin Dynamics simulations (Gopalakrishnan and Hogan, 2011). Equations to predict the coagulation enhancement as a function of the agglomerates Hamaker's constants and morphological parameters such as the volume-equivalent diameter and fractal dimension are proposed. These equations can be directly used in population balance simulations of powders transport, pollutant formation, pharmaceutical/food granulation, and many others.

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Gopalakrishnan, R., & Hogan Jr, C. J., 2011. Determination of the transition regime collision kernel from mean first passage times. Aerosol Science and Technology, 45(12): 1499-1509.

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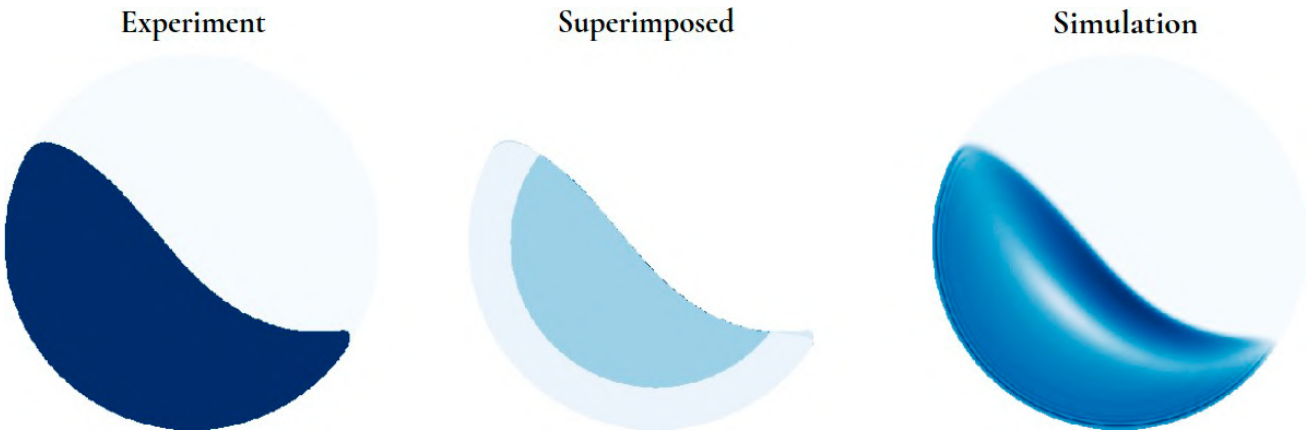
Ouyang, H., Gopalakrishnan, R., & Hogan Jr, C. J., 2012. Nanoparticle collisions in the gas phase in the presence of singular contact potentials. Journal Chemical Physics, 137(6): 064316.

ACCES: Autonomous Characterisation and Calibration using Evolutionary Simulation

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Keywords | calibration characterisation optimisation evolutionary algorithm distributed computing massively parallel

The discrete element method (DEM) is a powerful simulation technique that is capable of numerically modelling the behaviour of complex granular media, being used to better understand and optimise the internal dynamics of a large number of systems in both academic fields and industrial sectors, from fundamental research into contact mechanics to improving plant-scale reactors [Rosato and Windows-Yule, 2020]. DEM can offer exceptional accuracy through its lack of approximations over meshes and, if correctly calibrated, simulations can provide results with quantitative precision. It is this "if", however, that also represents DEM's biggest drawback: without choosing appropriate contact models and carefully calibrating multiple DEM parameters, the simulation outputs simply cannot be trusted. This calibration is a time-consuming process, typically involving the measurement of diverse particle properties including size, density, restitution and friction coefficients and, for purely "virtual" parameters such as the cohesive energy density, a great deal of experimentation [Luding, 2008].



To automate DEM calibration against experimental measurements, we have developed ACCESS – Autonomous Characterisation and Calibration using Evolutionary Simulation Software. ACCESS enables a researcher to calibrate virtually any DEM parameters against a user-defined cost function, quantifying and subsequently minimising the disparity between the simulated system and the experimental reality using state-of-the-art evolutionary strategies – in essence, autonomously 'learning' the physical properties of the particles within the system, without the need for human input. This cost function is completely general, allowing ACCESS to calibrate DEM against measurements as simple as photographed occupancy plots, or complex system properties captured through e.g. Lagrangian particle tracking. The algorithm itself is completely DEM engine-agnostic; it was implemented in an open-source Python library, providing an interface that is easy to use, but powerful enough to automatically parallelise arbitrary user scripts through code inspection and metaprogramming. It was used successfully from laptop-scale shared-memory machines to multi-node supercomputing clusters.

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Academic Press.

Analysis of Particle Impact Damage by Material Point Method

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Metal and ceramic coatings are commonly developed through methods that require high-temperature processing of materials. These high processing temperatures often lead to complications such as oxidation and adverse structural changes. Therefore, interest in solid-state coating methods like aerosol deposition and cold spraying has increased rapidly over the past two decades. In these techniques, powder particles are accelerated to high velocities by a supersonic gas jet and impinge on a substrate. When the impact velocity exceeds a material and process-dependent critical velocity, the particles deform and adhere to the substrate, building up a thin functional film. Deformation behaviour of the impacting particles plays an important role in their effective adhesion to the substrate and consequently, the quality of the deposited film. In this work, the deformation mode of the particles as a function of their mechanical properties is analysed. Material Point Method (MPM) is used to simulate the impact of particles with different ratios of Young's modulus to yield strength. Contours of strain in the

vertical direction and plots of energy versus time show that as the impact velocity increases, the particles undergo more noticeable plastic deformation. Also, qualitative observation of contours of strain suggests that as the ratio of Young's modulus to yield strength increases, the vibrating motion of the particle due to elastic waves disappears and plastic deformation becomes the dominant deformation mechanism.

DEM Simulation of Solidification of Cohesive Particles in Simple Shear Flow by using Dynamic Adhesion Force Model

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Keywords | DEM, Cohesive Particles, Modeling, Solidification, Simple Shear Flow, Dynamic Adhesion Force Model

The solidification of flowing granular materials affects many industrial powder processes, including piping blockage. The solidification of granular materials is a complex phenomenon because it is affected by many physical factors. Therefore, in this study, the solidification of adhesive particles in simple shear flow is studied by DEM simulation using the dynamic adhesion force model.

When performing DEM simulations of granular processes, the spring constant of the DEM contact model is often reduced in order to reduce computational cost. This is because the smaller the spring constant, the larger the time step required for numerical simulation. On the other hand, Kobayashi et al. [1] pointed out that reducing the spring constant promotes agglomeration in the flow behavior of cohesive particles. To overcome this problem, they proposed the dynamic adhesion force model (DAFM) for cohesive particles. This model was theoretically derived so that the collisional motion of particles with reduced spring constants is the same as that of the original particles. Therefore, the applicability of DAFM for the quasi- static behavior of contact force-dominated granular materials is still unclear.

In this study, DAFM is applied to DEM simulation of cohesive particles in simple shear flow to investigate the applicability of DAFM to the contact force-dominated or quasi-static behavior of cohesive granular materials appearing under low shear rate conditions. The results show that DAFM reproduces the original particle behavior and shear stresses well if the deformation of particles due to adhesion forces is below a critical value.

References

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DEM-BPM modelling for frozen particle fluid systems mechanics

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Keywords | Discrete element method, Bonded particle method, Creep modelling, Ice mechanics, Frozen particle fluid system

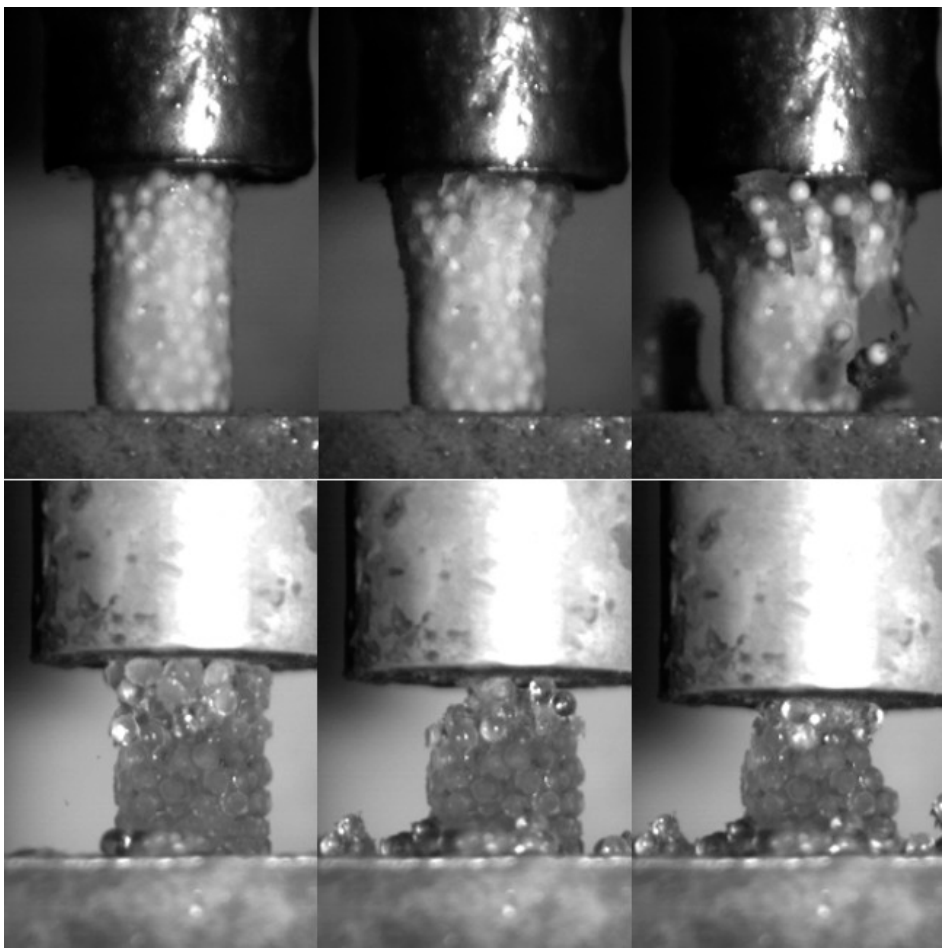
Frozen Particle Fluid Systems (PFS) play an important role in natural and technical systems. Under freezing conditions, solid bonds (ice bridges) are formed between particles, which alternate the mechanical properties of agglomerates. In this project, the behavior of the bridge area (single bond) and capillary area (multi-bonds) have been investigated. Bonded particle method (BPM), an extension of the discrete element method (DEM), has been used to describe the mechanical behavior of the frozen PFS.

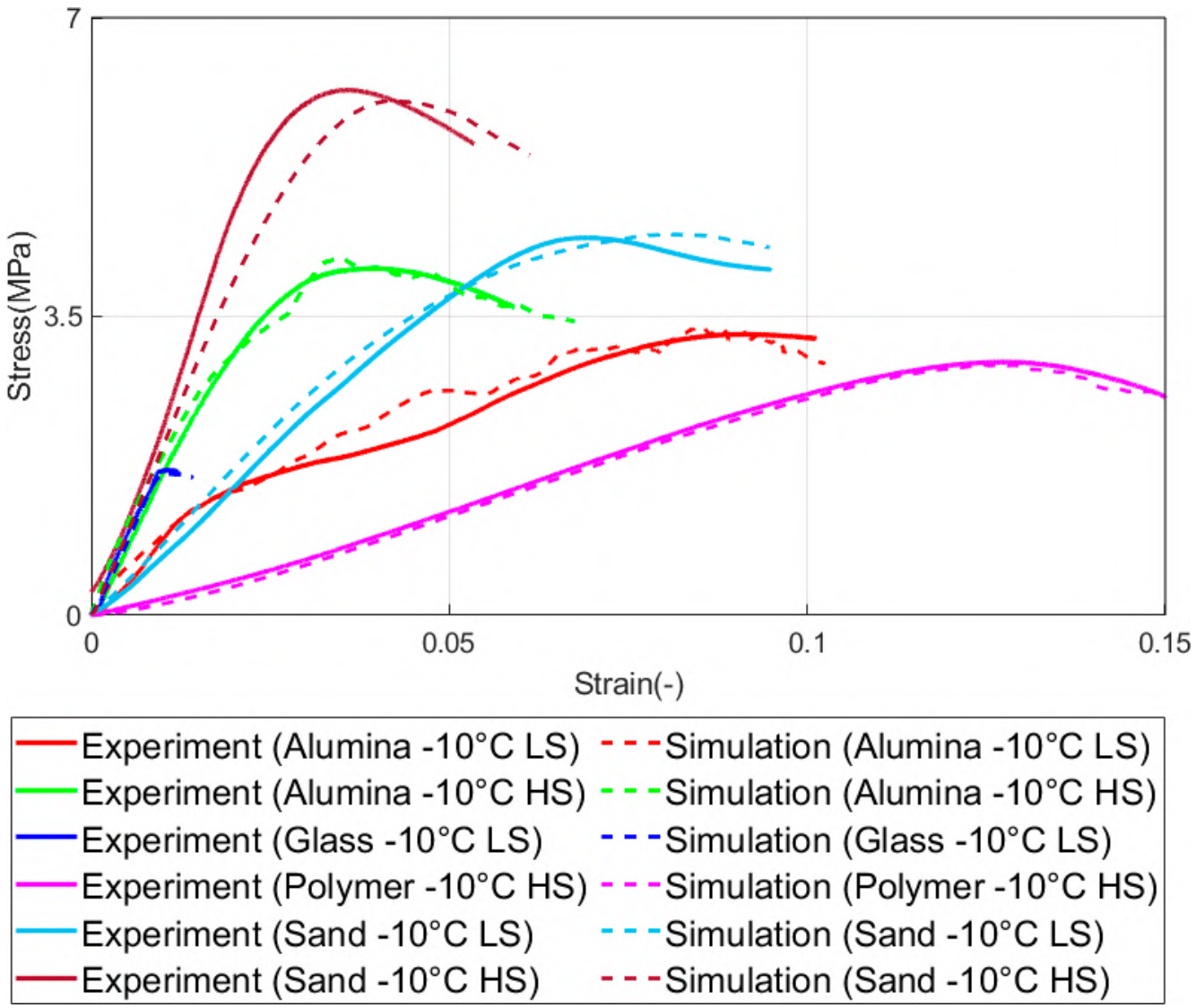
In the experimental part, a climate chamber has been created for maintaining ambient temperature and coupled with TA.XT plus Texture Analyzer (Stable Micro Systems, Great Britain). From literature, mechanical properties and mechanical behavior (Brittle, ductile, dilatant) [1] of frozen PFS are greatly influenced by temperature, saturation level, and strain rate. In experiments, spherical particles (glass, alpha-alumina, polymer) and non-spherical particles (quartz sand, I-SSO-sand) have been used. From the experiments, different mechanical behaviors were revealed.

The open-source DEM simulation framework MUSEN has been used for numerical investigation of frozen PFS behavior [2]. A new solid bond model was developed to describe the PFS rheological behavior. The new solid bond model combines strain- dependent and time-dependent

material behavior to describe the complex rheological material behavior. MUSEN calculations are parallelized for graphic processing units that have allowed to perform simulation of large samples in a relatively short period of time. Obtained simulation results, such as breakage strain or breakage stress, are in good agreement with the experimental data. Moreover, experimental and numerical samples reveal similar breakage behavior in the 100% saturation level of frozen PFS. However, more significant deviations were observed from lower saturation level frozen PFS.

With the acquired knowledge, in-situ micro-CT measurements were applied for gathering further microscopic, 4-dimensional data (3 dimensions, with time). The lack of understanding of the internal structure causes the high deviation in the porous structure simulation. The gathered 4-dimension data gives us insight into the internal structure differences between fully packed and porous structures, bond failure mode, and particle motion after breakage. The data provided allows better agglomeration generation in simulation and a more precise comparison between simulation and experiment results.



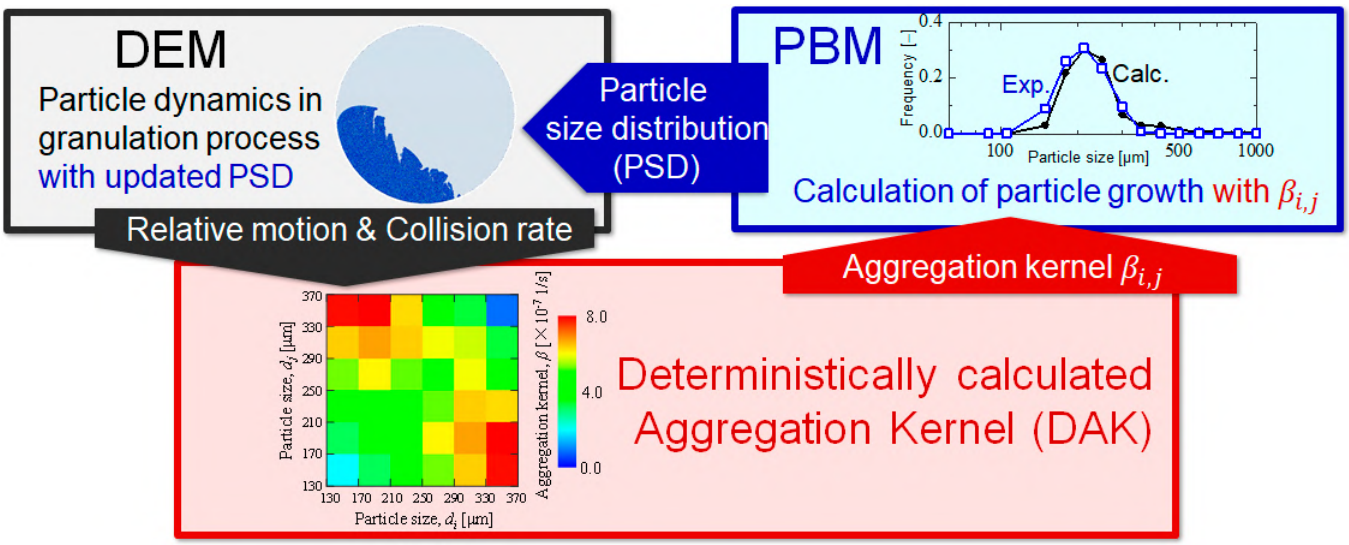


DEM–PBM coupling simulation of wet granulation without empirical parameters

Nakamura, Hideya (1); Baba, Tomoya (2); Ohsaki, Shuji (1); Watano, Satoru (1); Takehra, Kenta (3); Higuchi, Takahide (3)
(1) Osaka Metropolitan University, (2) Osaka Prefecture University, (3) JFE Steel Corporation

Keywords | granulation; numerical simulation; discrete element method; population balance model

Granulation plays an important role in various manufacturing sectors, including the chemical, pharmaceutical, and agricultural sectors. Computer simulation by coupling a discrete element method (DEM) and a population balance model (PBM) is a promising method for the rational design and operation of granulation processes. However, the aggregation kernels used in existing DEM–PBM coupling methods are semi-empirical and contain unknown parameters, hindering their predictive capability. Herein, we propose a new DEM–PBM coupling method with a deterministically calculated aggregation kernel (DAK), namely, the DEM–DAK–PBM method. In the DAK, the relative motion of the two particles is extracted from the DEM simulation. When the relative motion is nearly zero, the pair of particles can be regarded as an aggregated particle. According to this distinction, the aggregation kernel is directly computed from the DEM simulation without fitting any unknown parameters. The DEM–DAK–PBM coupling method was applied to simulate the wet granulation of calcium carbonate powder using a rotating drum, and its performance was evaluated. The change in the particle size distribution over time exhibited good agreement between the simulation and experimental results, demonstrating the effectiveness and validity of the DEM–DAK–PBM coupling method.



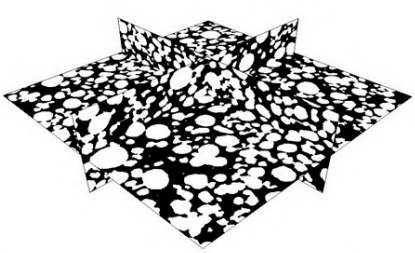
Discrete Element Method (DEM) Modelling of Lithium-ion Battery Electrode Structures under Different Calendering Conditions

Ge, Ruihuan (1); Cumming, Denis (2); Smith, Rachel (2)
(1) The University Of Sheffield, (2) University of Sheffield

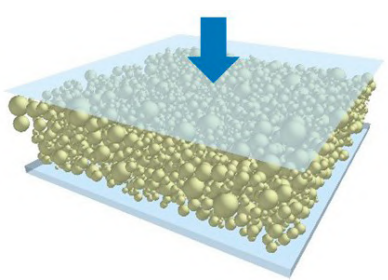
Keywords | Discrete Element Method (DEM); Lithium-ion battery; Calendering; Electrochemical analysis

Lithium-ion batteries are widely used as energy storage devices. However, the relationships between manufacturing parameters, electrode microstructure and subsequent battery performance are not thoroughly understood. Electrodes contain many different materials spanning different phases including active material (AM) particles, carbon binder domain (CBD) and the pore space. In this work, Discrete Element Method (DEM) with a bonded particle model is used to investigate the evolution of electrode microstructure under varying calendering conditions. For the first time, DEM simulations of electrode calendering are validated using X-ray tomographic data. Electrode structural properties including porosity distribution, specific surface area and tortuosity factors are studied and compared with corresponding tomography scans. A novel stochastic structure modelling approach is used to generate the nano-porous CBD phase is between AM particles.

Finally, battery performance with varying microstructures is both simulated and experimentally evaluated via electrochemical analysis. By combining DEM simulation and electrochemical analysis, this work provides a highly promising method to quantitatively predict lithium-ion battery electrode performance, and presents a potential tool for electrode micro-structural design.



(a) Active material (AM) particle of an electrode from X-ray tomography



(b) DEM simulation setup for electrode calendering

Effect of drag and contact force scaling using coarse grained particles in coupled DEM-CFD simulations

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(1) Technische Universität Berlin

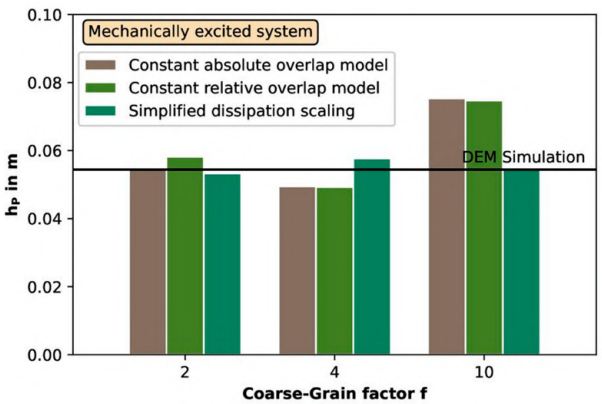
Keywords | Coarse-graining, DEM-CFD, discrete element method, fluidization, multiphase flow

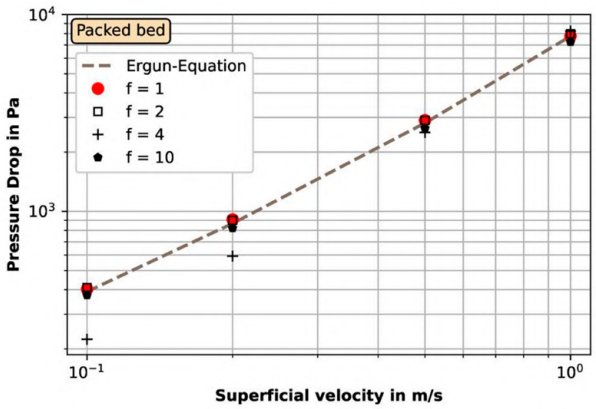
Fluidized particle systems play an important role in various engineering fields. To model these systems numerically, CFD coupled with DEM is widely used. However, the CFD-DEM approach is computationally very demanding and, as for today, it is restricted to lab scale reactors with only a limited number of particles.

Coarse grain approaches with coupled CFD-DEM have become popular over the last years to address the aforementioned issue. Particles of the original system are summarized into so-called grains and therefore the number of particles can be reduced drastically, giving the opportunity to calculate industrial scale reactors in a realistic time frame [1].

A very common approach for coarsening the particles is to assume the conservation of energy from the original to the coarse-grained particle system. Thus, the forces in the system must be scaled in an appropriate way. An important parameter for scaling in coarse grained DEM is the coarse grain factor, which is the ratio of the grain radius to the particle radius:

$$f = R_{Grain} / R_{Radius}$$





In this study, the scaling of drag and contact force is investigated in depth. In a first step, preliminary studies were conducted in a mechanically excited system, to eliminate fluid drag and in a packed bed, to eliminate contact force. Optimal scaling rules are identified based on the comparison of characteristic physical parameters,e.g. average particle height and pressure drop, of the coarse grained to the original systems as shown in the figures.

In a second step, fluidized beds comprising of monodisperse particles under different inlet velocities are modelled. Analysis if the scaling rules from the preliminary studies are equally applicable in systems governed by contact and fluid forces is conducted. To incorporate heterogeneous flow structures as they are appearing in fluidized beds in reality, drag laws are altered for large coarse grain factors.

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A revolutionary, Evolutionary Framework for Modelling Multiphase Systems

Windows-Yule, Kit (1)
(1) University of Birmingham

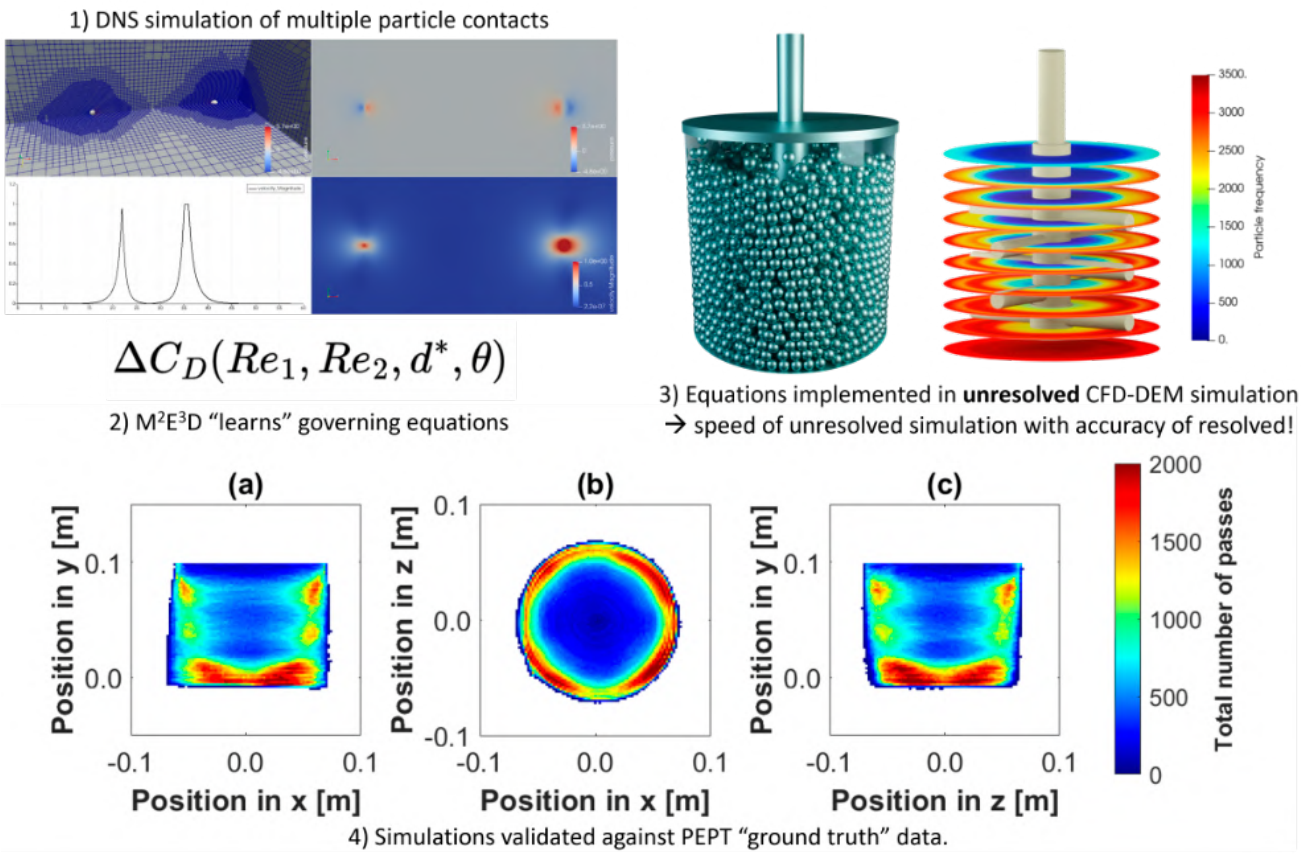
Keywords | CFD, DEM, positron emission particle tracking, multiphase, industrial

Multiphase systems are vital to the development of a myriad of products spanning multiple industrial sectors, from the formulation of novel biofuels, chemicals and pharmaceuticals in stirred tank reactors to plastic recycling in gas-fluidised beds. Numerical models of these and many other systems are used to better understand and optimise their internal dynamics and end-products. Modelling classical fluids is relatively simple: their properties may be directly measured using widelyavailable characterisation tools, and directly input to CFD models. Particulate media (powders and granulates) for which there exist no established continuum models, are significantly harder to simulate; they are typically modelled piece- wise using the Discrete Element Method (DEM), whose calibration is infamously problematic . Modelling multiphase (solid- liquid and solid-gas) systems is more complex still, requiring detailed properties of both fluid and particulate phases, and suitable drag models for the coupling thereof.

Multiphase Materials Exploration via Evolutionary Equation Discovery (M^2E^3D) is a novel evolutionary approach to determine the equations governing the interactions of solids and fluids in multiphase systems. In other words, M^2E^3D autonomously discovers the equations required to accurately reproduce a system's full, three-dimensional dynamics. The resulting combinatorial optimisation problem of discovering analytical closed-form expressions modelling previouslymeasured data points is solved using a symbolic regression engine employing an evolutionary algorithm balancing the model's error and complexity [1].

In this talk, we introduce the M^2E^3D framework, and show industry-relevant case studies pertaining to both liquid-solid and gas-solid systems., where M^2E^3D has been used to provide novel equations simulating, respectively, the influence of fluids on the inter-particle interactions within a stirred media mill, and the drag force on highly-aspherical particles.

We validate our results against detailed, three-dimensional, experimental data acquired using Positron Emission Particle Tracking [2], showing that the models produced are capable of achieving full, quantitative agreement with real-world systems.



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Spreading behaviour of particle piles under vibration: Experiments and DEM simulations

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Keywords | Discrete Element Method, Vibration, Spreading

The transport of granular media can be supported by the vibration. Due to this energy input, the particles can move and change the packing density of the bulk, which can change the flow behaviour of the granular media. To understand the influence of the frequency and amplitude on the particle movement, the change of the particle pile's height was investigated. This study focused on the in-depth analysis of the micro mechanisms of the flow behaviour during vibration using the discrete element method (DEM).

Experiments were performed to investigate the change of the angle of repose and the height under vibration over time for glass beads with a diameter of 1 mm. The particle piles were created on a circular surface (diameter of 30 mm) consisting of a layer of 3D-printed spheres with a diameter of 2 mm. The change of the pile's height and angle of repose over time depending on the vibration's frequency and amplitude was analysed with video recordings. It was showed that a static angle of repose is reached, which depends on the induced energy of the vibrating surface.

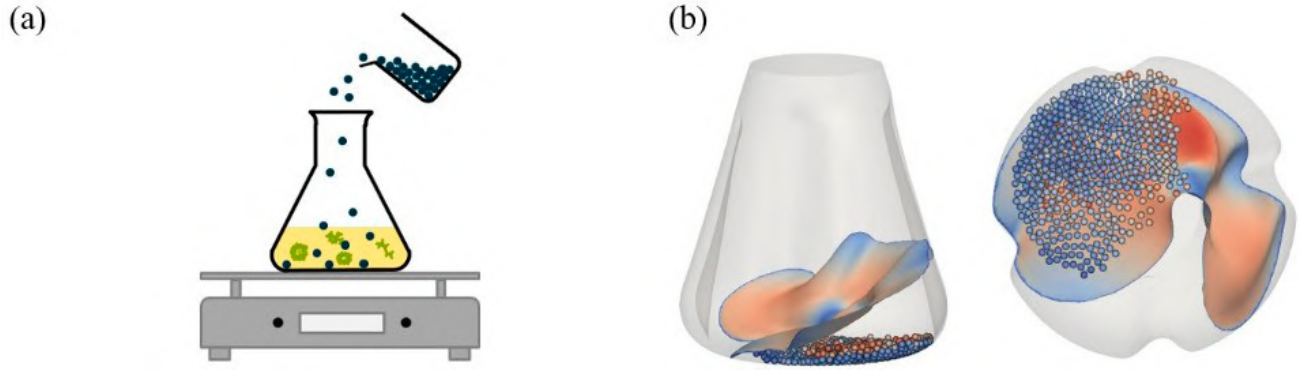
To understand the influence of the micro mechanisms during vibration, a DEM model was developed. The simulation model was calibrated by comparing the angle of repose, the pile's height-to-diameter ratio, and the resulting torque during stirring of the particles in a rotation rheometer. First, the performed spreading experiments were simulated to validate the DEM model by comparing the change of the angle of repose over time. In addition, the particle velocities inside the pile and the packing density were analysed with the simulation. With the given DEM model, the influence of different parameters, such as particle size, coefficients of friction and restitution, on the pile's height and angle of repose during vibration were investigated. Due to these simulations, the dominant mechanisms could be determined, which influenced particle movements under vibrations.

Quantification of the bead induced mechanical stress on filamentous bioagglomerates via coupled CFD-DEM simulations

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Keywords | CFD, DEM, simulation, mechanical stress, biotechnology, validation

Filamentous microorganisms are widespread in industrial biotechnology as production systems of active pharmaceutical ingredients or organic acids. Depending on the cultivation conditions, biomass growth takes place as dispersed mycelium, as clumps or in the form of dense bioagglomerates. In the case of the filamentous *Lentzea aerocolonigenes*, beads were used in shaking flask cultivations (Fig. a) to increase the formation of an antibiotic (Schrinner et al. 2021). By varying size, density and number of beads as well as shaking rate, the final product concentration is positively influenced to differing degrees. By changing these process parameters, a varying intensity and frequency of stress on the bacteria were induced. To quantify these stresses via simulations, Schrader et al. (2020) used the coupling of computational fluid dynamics (CFD) with the discrete element method (DEM). Based on this method, simulations (Fig. b) were carried out in this study for the parameter combinations used by Schrinner et al. (2021). With the help of the particle tracking velocimetry method, the discrete distribution of the bead velocities were determined and compared with simulation data. From the simulation results, the dependencies of the stress energy and stress frequency on the bead diameter, the shaking rate and the bead density were derived. By plotting the final product concentration versus a new defined characteristic stress parameter, an overarching optimal stress range was obtained for different cultivation conditions. Thus, this generated knowledge of the dependency of product formation on mechanical stress serve as a basis for choosing optimal mechanical stress conditions on filamentous systems to achieve the highest product concentration and as a scale-up criterion for cultivation processes.



Schrader, M.; Pommerehne, K.; Wolf, S.; Finke, B.; Schilde, C.; Kampen, I.; Lichtenegger, T.; Krull, R.; Kwade, A.; (2019). Design of a CFD-DEM-based method for mechanical stress calculation and its application to glass bead-enhanced cultivations of filamentous Lentzea aerocolonigenes, Biochemical Engineering Journal 148, 116-130.

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Simulating the compaction of arbitrarily shaped particles with Level-Set DEM

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(1) Cambridge, (2) Copenhagen

Keywords | powder compaction, uniaxial compression, level set, particle shape, discrete element method, YADE

Tablets are the preferred form of drug delivery within the pharmaceutical industry because of their hight patient compliance, ease of large-scale production, and ease of transport. However, the production of tablets can be challenging due to the occurrence of mechanical defects such as chipping, lamination, and capping. Defects are difficult to predict because of the many possible variations within the powder compaction process itself, and the formulation of the powder. A cumbersome trial-and-error process is typically needed to achieve a satisfactory production process for a new tablet with active pharmaceutical ingredients. Computational modelling has become an increasingly popular tool to accelerate this empirical process by performing in silico experiments that reduce the number of possibilities.

A major obstacle in the modelling of pharmaceutical powders is the description of particle shape. Continuum models (e.g. finite element analysis) ignore this aspect whereas the discrete element methods (DEM) are frequently limited to convex shapes or simply by the computational cost incurred. Recently, Kawamoto et al. (2016) described a version of DEM that makes use of level sets (LS-DEM) to describe particles of arbitrary shapes, including non-convex particles. Soon hereafter, Duriez and Galusinski (2021) implemented LS-DEM into the publicly available YADE software and showed that LS-DEM is orders of magnitude faster than DEM with polyhedral shapes.

Despite the significant advances brought about by LS-DEM, the description of interparticle interactions is still limited. Currently, interparticle interactions only consider the deepest point of particle-particle penetration or are sensitive to the discretisation of the particle. We report a new description of the particle-particle interaction in LS-DEM. This new description allows the reformulation of conventional contact laws, like the Hertz-Mindlin contact law, such that they can be used on arbitrarily shaped particles in LS-DEM.

Duriez, J. and Galusinski, C., 2021. A Level Set-Discrete Element Method in YADE for numerical, micro-scale, geomechanics with refined grain shapes, Computers and Geosciences, 157:1-12

Kawamoto, R., Andò, E., Viggiani, G., and Andrade, J., 2016. Level set discrete element method for three-dimensional computations with triaxial case study, Journal of the Mechanics and Physics of Solids, 91:1-13

Simulation of suspension drying via CFD-DEM coupling

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(1) Technische Universität Braunschweig, Institut für Partikeltechnik

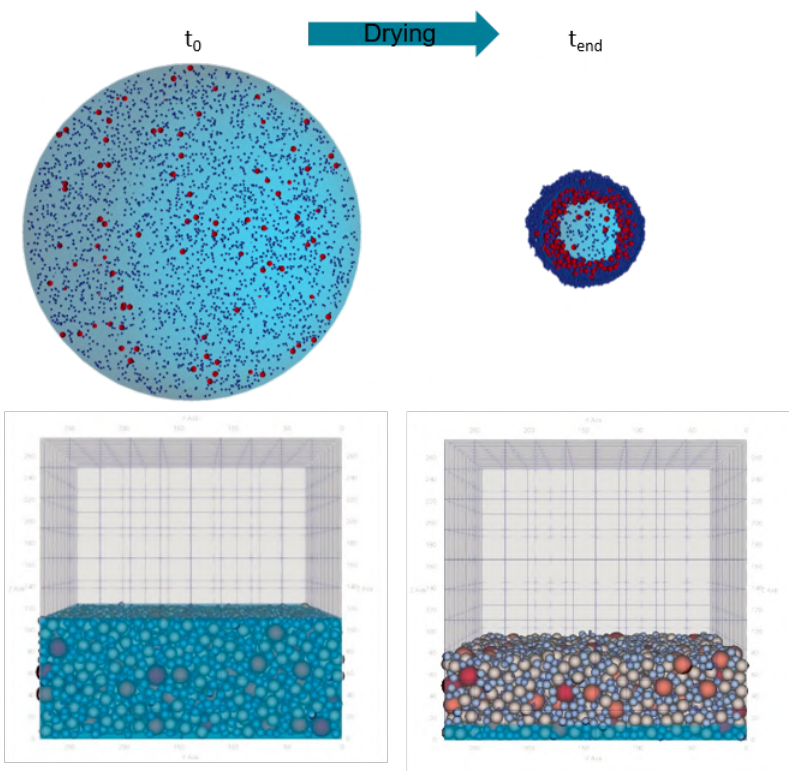
Keywords | CFD-DEM, Simulation, Suspension drying, Lithium-ion batteries, Spray drying

Particulate structures form the basis of many different products. Spray-dried aggregates are used, for example, in the pharmaceutical industry as carrier systems for poorly soluble drugs, while particulate layers serve, among other things, as electrodes for lithium-ion batteries. The microstructural properties of the particle structures have a major influence on the function of the products and can be specifically influenced by selecting suitable formulation and process parameters.

However, the influences of these parameters on the underlying structure-forming mechanisms during drying have not yet been sufficiently explored. Simulation methods can help to investigate the influence of formulation and process parameters on the microstructure and the mechanisms involved more deeply.

In this study, methods to simulate the influence of formulation and process parameters on structure formation during the drying process are presented. For simulating a two phase flow by means of CFD, the volume of fluid method is used, which was expanded by a negative source term to reflect the evaporating liquid during drying. Coupling of CFD and DEM then enables the consideration of fluid-particle interactions within the simulation. This can be done either via a computationally expensive resolved method, or with a faster unresolved method. For both approaches, models that represent the action of surface tension on the particles by means of a capillary force are used. Apart from that, structure forming particle-particle interactions are taken into account on the DEM side, such as cohesion, DLVO forces or Brownian motion.

Using those techniques, results for the structure formation of particulate aggregates and coatings are presented. More precisely, the influence of the various forces as well as formulation and process conditions on the hierarchical structuring of spray dried aggregates are discussed, as well as the effects on coatings used as electrodes for lithium-ion batteries. In the end, the ability of the presented simulation models to predict particulate structures is evaluated.



Ultra fast calculation of conductive heat transfers in a moving granular medium

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Keywords | Granular system DEM heat transfer long time extrapolation

Granular reactive flows are found in many industrial and natural processes. However, despite advances in modern computers, the simulation of such systems involving millions of particles is prohibitively expensive in terms of computational time. Therefore, the main objective of this work is to find novel algorithms to reduce it significantly.

Many researchers, such as (Bednarek, 2019) and (Lichtenneger, 2016) have looked into this issue and have developed different extrapolation methods based on the pseudo-periodic properties of many real processes. Bednarek applied the pairing algorithm to a conical screw mixer containing dry powders. His extrapolated results agreed with the full DEM results by decreasing CPU time to a factor of 10^5 . Lichtenneger used the recurrence CFD that is based on the Poincaré's recurrence method to simulated transfers in fluidized beds .This method allowed also a similar gain in computational time.

The latter approach allowed long time extrapolation for obtaining the transfers inside a granular medium . However, this method does not take into account collisions. Therefore, the newly developed method consists in an extension of the existing method developed by Bednarek to heat transfers by conduction in granular media, taking into account particle- particle collisions. Preliminary results show that the new method is promising.

CFD-DEM Modeling at High Temperatures: Effects of Gas Density Change and Radiation

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Keywords | CFD-DEM, radiation, compressibility, high-temperature, application

Under the impending climate change crisis, high-temperature processes demand efficient and precise computational tools. Here we present guidelines for selecting the simplest computational approach for a gas-particle flow problem at hand.

Specifically, we consider the effects of gas density change and thermal radiation in such gas-particle systems.

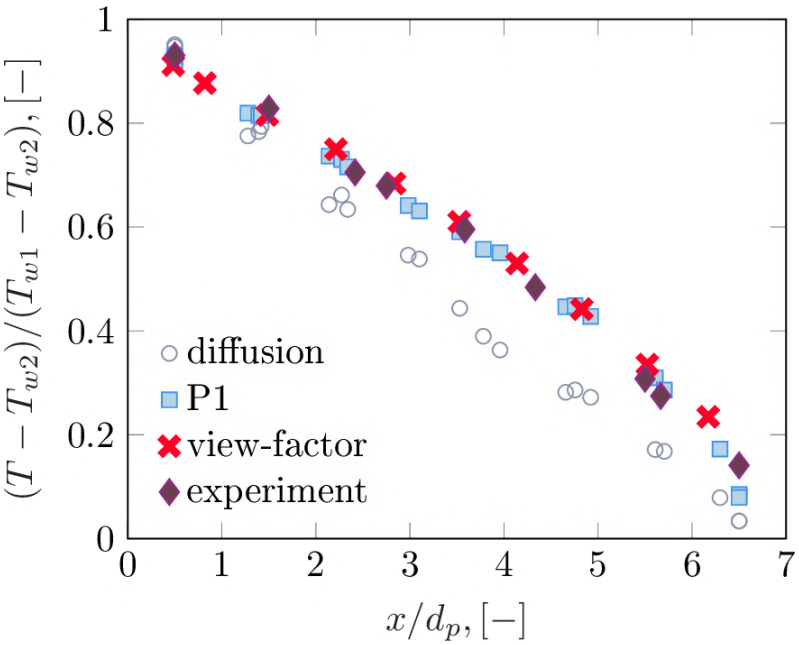
The change of gas density can alter the flow dramatically by introducing non-linear pressure drop and convective currents. From a strict theoretical standpoint, such flows necessitate a compressible flow solver. Using analytical methods, we show, for example, which isothermal flows experience significant compressibility caused by gas-particle drag forces. We find that the need for a compressible flow solver can be assessed using a relatively simple relation of the Mach number, a suitable drag closure, and the length of the domain relative to the particle diameter. Also, we present a relation to assess the need for a compressible solver for flows with heat injection.

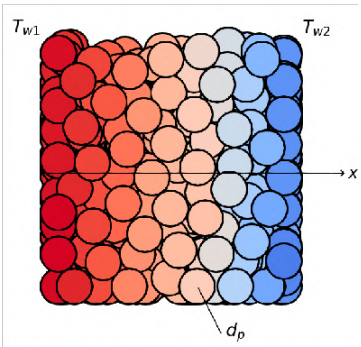
Radiative heat transfer is diffusive in opaque media. We show that this limit is reached within a length of less than 100 particle diameters for a solid fraction of 0.1. In denser systems (solid fraction of 0.5), radiation is diffusive already at a length of 6 particle diameters. Still, we show that due to temperature slip, simple diffusion approximations for radiation may fail near bounding walls. We compare numerical predictions obtained using a diffusion approximation, a P1-model, and a view- factor model (Tausendschoen & Radl, 2021), against an experimental dataset (De Beer, 2014; see Figure). Precision of the radiation models is evaluated in terms of the heat flux, the temperature distribution, and the time needed to thermally relax the system. Finally, the efficiency of the models is evaluated in terms of computational effort and applicability to parallelization and coarse-graining.

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DEM simulation strategies for the flow of dry coated powders in pharmaceutical dry powder inhaler devices

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Keywords | Dry Powder Inhalers, Discrete Element Method, pharmaceutical devices, multiphase systems

The flow of fine active pharmaceutical ingredients (API) in dry powder inhaler (DPI) devices is often achieved by handling and dispersing dry coated particles, i.e. complex particle systems made up by very fine API powder coating coarse carrier particles in order to improve the flowing properties and allow for controlled aerodispersion. In a DPI, the motion of these complex particles is induced by either a velocity and pressure field of the breathing air, such as in cyclonic devices, or by rotating capsules, which are designed to eventually lead to a controlled deaggregation during patient inhalation. The real flow field developing in DPIs is very hard to predict in detail, with the consequence that developing improved devices requires substantial research efforts. Experimental investigations are often assisted by the analysis of digital twins, i.e. simulated devices. The flow of adhesive solids with very different diameters (1-5 mm for the API and 80-200 mm for the carrier) subjected to frequent collisions is best simulated using Discrete Element Method. At the same time, however, accurately simulating mixed coarse/fine particles is very challenging as it requires special treatment of collisions, purposedly developed fluid-particle interaction models and high-performance computing resources. In the present study, the fundamental contributions for accurate representation of the flow of dry coated particles in dry powder inhalers are discussed for both cyclonic flow and capsule-based devices. The importance of selected interaction contributions, such as polydisperse drag, lift, fluid torque, and their 4-way coupling on the resulting fluid-particle flow field and deaggregation performance is demonstrated. In addition, issues in dealing with simultaneous collisions of fine and coarse particles with the walls are discussed, and testing of a novel method to overcome them is presented.

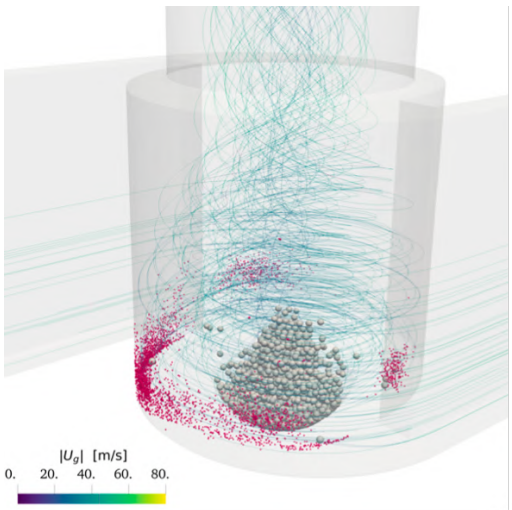


FIGURE 1. Transient cyclonic flow of air lifting carrier particles (light grey) and deaggregated API powder (purple).

Numerical Simulation of the Rheological Behaviour of Microparticulate Suspensions with Unresolved Coupled CFD-DEM- Simulations

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(1) TU Braunschweig - Institute for Particle Technology, (2) TU Braunschweig - Institute for Building Materials, Concrete Construction and Fire Safety

Keywords | Coupled CFD-DEM, Microparticulate Suspensions, Rheology, Interparticle Forces

Various forces act between the particles of microparticulate suspensions (FIG. 1). To simulate this kind of suspensions, not only the contact forces between the fluid and the particles (e.g. drag and lubrication forces) but also the normal and tangential contact forces between the particles have to be taken into account. Unresolved coupled CFD-DEM simulations are utilized in order to model the suspension's rheological behaviour. However, usually not all forces are considered in coupled simulation and herewith a realistic representation of the rheology of suspensions has not been possible. To overcome the challenge, the influence of different forces was investigated and it was found that a contact, a static friction, a rolling friction, a drag, a rotational drag and a lubrication model are needed for a correct description of the rheological properties of microparticulate suspensions. Contact and static friction models are necessary for the direct particle contacts and are already well studied. Additionally, rolling friction is essential to represent the non-sphericity of the particles (Wensrich, 2012). Drag forces accelerate and decelerate the particles while rotational drag forces ensure the rotation of the particles. These forces act from fluid to particle. Typically, the rotational drag force is only considered in resolved simulations, but should not be neglected for a correct description of viscosity (Fukui, 2018). The lubrication forces consider the displacement of fluid between particles and dissipates energy in the suspension (Kroupa, 2016). The results show good agreement with analytical models and experiments with microparticulate suspensions over a broad range of volume fractions (10-50 vol%).

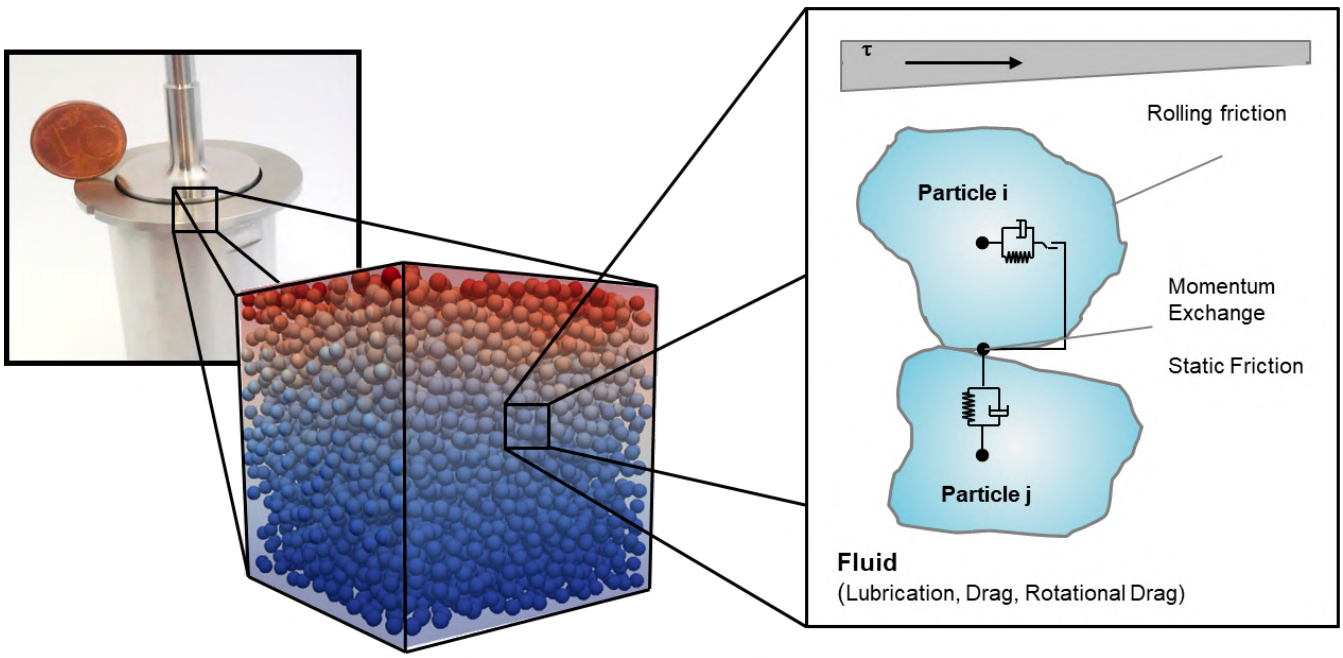


Figure 1: Forces between particles in suspensions.

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Simulation of particle erosion in DEM

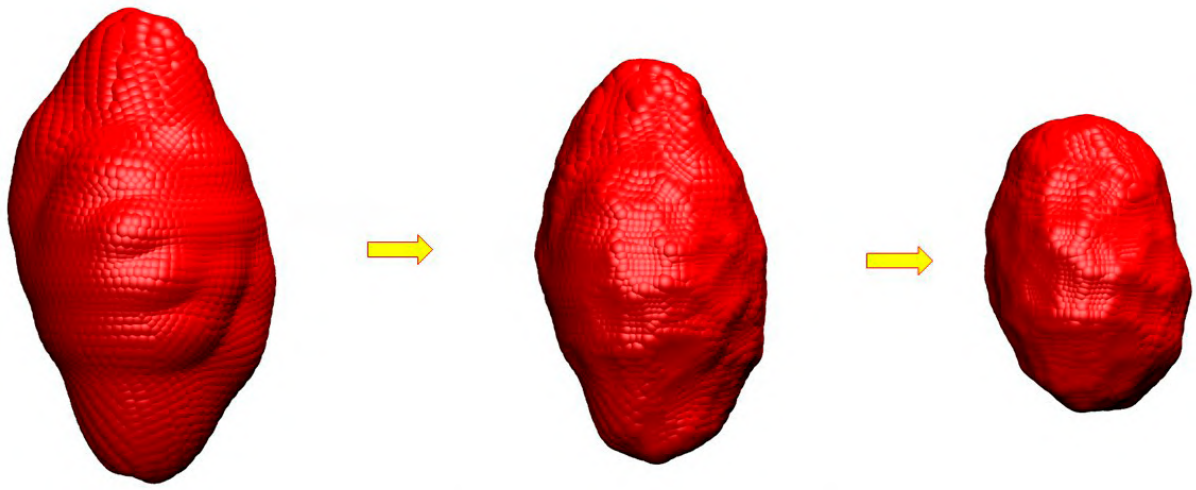
Capozza, Rosario (1); Hanley, Kevin J. (1)
(1) University of Edinburgh

Keywords | Erosion, Wear, Archard model, Finnie model

Erosion consists in wearing down a particle's surface through contact interactions with other bodies. Fines are produced during erosion which are smaller than the particle's size. Particles become smoother or rougher over time depending on the external conditions. Industrial applications are strongly affected by erosion. Fines reduce flowability, which impairs processing operations such as conveying, blending or tableting [1]. Attrition affects the bulk density, surface area, segregation behaviour and dissolution rate which has major implications for product quality [2, 3]. Therefore, understanding erosion in order to better control it is of fundamental importance.

DEM simulations have made an important contribution to understanding granular materials [4]; however, simulations of erosion are scarce and mostly limited to specific applications [5]. We present a comprehensive approach for modelling erosion in DEM, which simulates the evolution of the particle shape. The particle surface is discretized by a set of points and is eroded through the impact of non-deformable bodies. When the surface pressure overcomes a threshold, erosion is captured by displacing points along the surface normal. The model describes both the erosion due to scratching and normal/oblique impact as a function of velocity and applied load and shows a good agreement with experimental data. All sources of energy dissipation are included in the model, without additional parameters.

The implementation of this method in the open-source LAMMPS code is presented along with possible sources of performance improvement.



Evolution of particle's shape during the erosion process

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DEM based flowsheet simulation of dry granulation in roller compaction

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Dry granulation is an economical process to improve the flowability of heat and moisture sensitive powders. Roller compaction enables a continuous process to compact powder between two counterrotation rollers into ribbons.

Subsequently, these ribbons are comminuted by rotating or oscillating screen mills into any desired granule size. Flowsheet simulations offer an essential opportunity towards high Quality by Design (QbD) products. However, the modeling of the comminution part bases commonly on empirical testing, which Discrete Element Method (DEM) simulations can supersede since the DEM allows mapping of the ribbon's breakage behavior. Therefore, this study targets the complementation of comminution units and links it with the compaction subunit of flowsheet simulations. The DEM simulations were performed in the open-source GPU-accelerated simulation Framework MUSEN, and flowsheet simulations utilized a python framework.

For comminution, the ribbons implemented in MUSEN consist of bond-connected particles. The bonds underly typical mechanics and break if material strength is exceeded. This allows the replication of the breakage of particles or any complex structures into fractures.

The milling process was implemented in DEM, and the particle size distribution passing the screen was analyzed. Ribbons of different geometries and porosities were implemented, and machinery parameters like screen size and rotor velocity were variated. The results feed a database directly coupled to the flowsheet framework. Besides, different rotor geometries were analyzed.

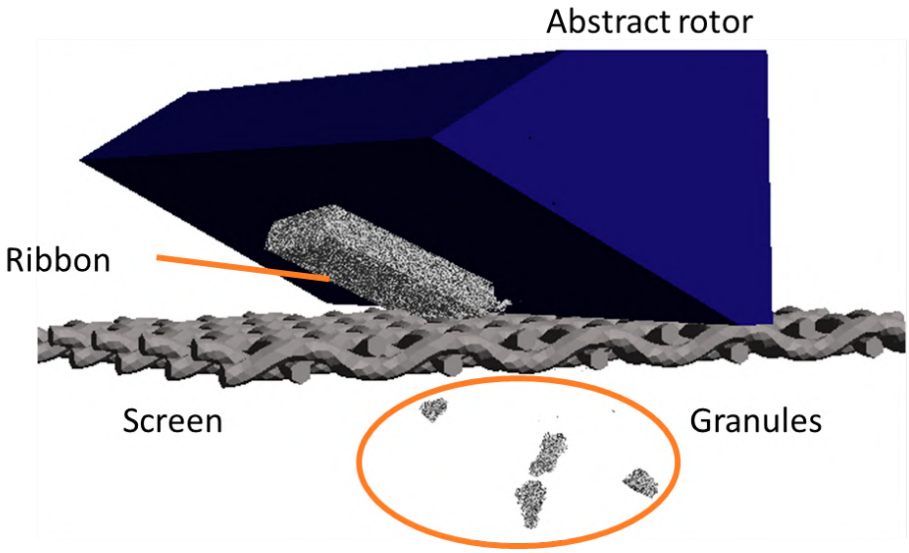


FIGURE 1: Ribbon milling in linearized screen mill. The particles are hidden to visualize the bond network.

Heat transfer and drying in a rotating drum: CFD-DEM Simulations

Rastogi, Aman (1); Vivacqua, Vincenzino (1); Hare, Colin (2); Gobby, Darren (1); Stitt, Hugh (1)
(1) Johnson Matthey, (2) Newcastle University

Rotating drums are widely used for thermal treatment of granular materials because of their ease of design and operation. Successful operation for such a process depends strongly on close control of the duration, intensity and uniformity of the heat received by the material. In this work, unresolved CFD-DEM method has been used to simulate heat transfer and drying in a rotating drum. Parameters for drying kinetics have been determined using data from multiple heating rate, non- isothermal thermogravimetric experiments. A sensitivity analysis of the reaction model in a TGA set up is performed to investigate the effect of DEM particle contact parameters, heat transfer properties, and heat generated due to reaction on simulated reaction conversion with time. The heat transfer properties and particle size are found to have a significant effect on the simulated conversion vs time. The drying model is coupled with CFD-DEM to simulate a rotating drum with gas flow along the axis and heated wall. The effect of rotation speed, wall temperature and inlet gas velocity on the drying rate of solids has been investigated. The simulations are then compared to experiments for validation.

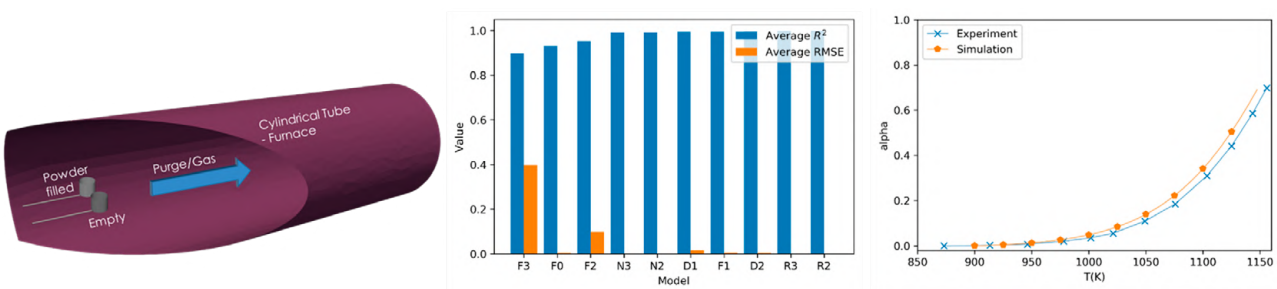


Figure (a). TGA-DSC experimental set up, (b) RMSE and R2 values for various kinetic models fitting on non-isothermal thermogravimetric data for decomposition of CaCO3 (c) Simulated vs experimental conversion of CaCO3 in a TGA crucible at temperature ramp rate of 30K/m

Modeling and DEM simulation of high stress compaction for cohesive powders with elastic-plastic contact behavior

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(1) Technical University of Kaiserslautern

Keywords | Discrete Element Method, Powder Compaction, Plastic Deformation, Simulation

Powder processing usually includes several production stages, whereby stresses can vary heavily. While storage and transport might enforce pressures of kilopascals, the final compaction step to a tablet or similar will require orders of magnitude higher stresses on the powder.

For low stresses, fine powders can be assumed to be mostly elastic, whereby contact force and cohesion modeling has already been accomplished in the past (Johnson et al. 1971). For elastic-plastic contacts, we recently developed a contact model (Weis et al. 2019) that, however, neglected the purely elastic force due to a low yield limit. We propose an extension of this model by including this pure elastic contact before the yield stress is reached. An approximation of the current cohesive forces is proposed for arbitrary elastic/elastic-plastic deformation (Fig. 1) using the Hamaker wall-wall model (Hamaker 1937). The model was then converted for the open-source software LIGGGHTS® for Discrete Element Method (DEM) simulations.

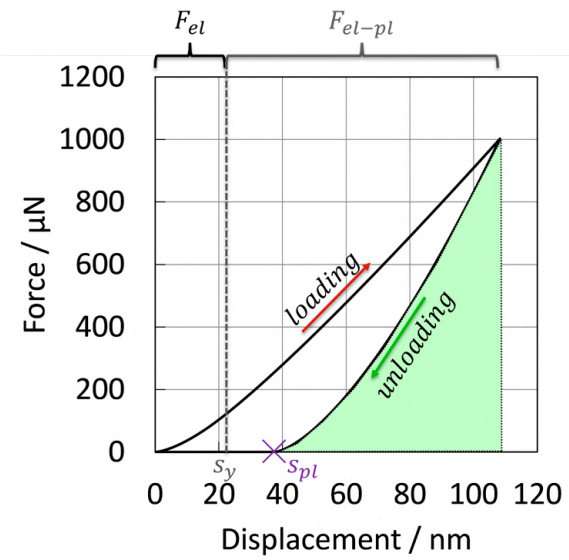


Fig. 1: Typical force-displacement curve with plastic deformation (s_pl) and elastic energy (green area).

For the validation of the model, a cohesive lactose powder (FlowLac 90, Meggle) was subjected to uniaxial compaction tests of various normal pressures. The compaction cell (Fig. 2) consisted of a sliding outer shell that is suspended by a spring, which slides downwards during compaction.

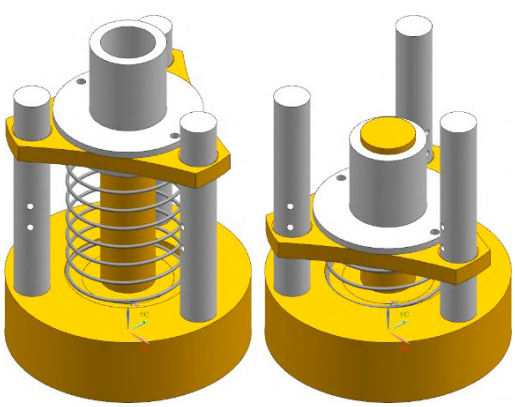


Fig. 2: Experimental compression matrix.

Fig. 3 shows a compression experiment and the corresponding simulation with repeated loading with 100 kPa. DEM model parameters were obtained experimentally using nanoindentation and other direct measurements with calibration of the Hamaker constant by flowability tests.

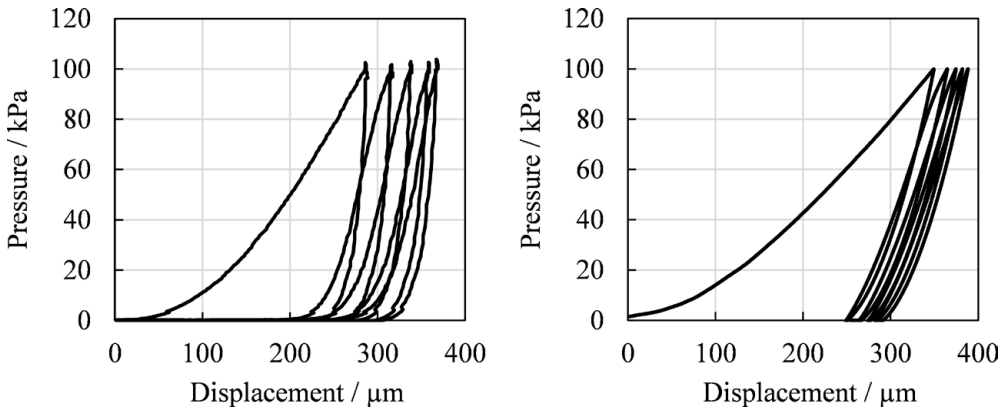


Fig. 3: Exemplary compression experiment (left) and simulation (right).

The first cycle shows high plastic deformation and high energy loss after unloading, while following cycles can further compress and consolidate the powder bed with diminishing impact of each new cycle. Overall, the simulation is in good accordance to the experimental data.

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Precision of Validated Crushed-Rock Flow Behavior in Front of a Bulldozer Blade Using Analytical, Discrete-Element Method (DEM) and Reduced-Order Model

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Keywords | Discrete element method (DEM), Bulldozer Case 750 L, Reduced-Order Model (ROM), Dummy/Primitive particles, Detail Particles, Simulation Precision Comparison

Fields tests for construction equipment are costly, time-consuming, weather-dependent, and require highly skilled/trained personnel in order to ensure consistency and to provide confidence in the results. Methods have been developed throughout the years to predict forces on construction equipment and are available on a wide range of precision. This presentation focuses on the precision of different methods compared to actual field tests (from Analytical Models to Reduced-Order models or ROMs).

The field tests were performed on standardized ground during summer 2021 using a 2016 Case 750 L Bulldozer equipped with a standard 2.6m blade pushing crushed rock. Properties of the material were measured in detail in order to compare virtual crushed rock of variable shapes and its effect on precision of the simulation (3D forces on the blade and flow behavior).

The Discrete Element Method (DEM) is a modeling tool efficient to predict the behavior of soils given its capability of accurately predicting bulk material dynamics. The results of fields tests are compared to an analytical model, a DEM simulation using dummy/primitive particles (to speed up simulation but with less precision), a DEM simulation using detailed particles, and a Reduced-Order Model (ROM). Rocky DEM software was used to simulate the family of particles (from dummy to detailed ones). The DEM simulation-scenarios results are used to train the ROM combined with real-time data. The fast- solving time of different scenarios using ROMs allows them to be used in conjunction with real-time monitoring data for a complete Digital Twin deployment and implementation.

Conclusion:

This presentation focuses on how Rocky DEM software was utilized to develop accurate simulations with dummy and detailed particles based on field tests of the 750 L Case bulldozer. It contains details of the field experiments, methodology for property measurements, simulations precision used for training the Reduced-Order Models (ROM).

DEM simulation of cohesive particles using scaled-up particle model

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Keywords | Cohesive particles; Discrete Element Method; Scaled-up particle model; Coarse graining;

Simulation of cohesive particles has gained increasing attention due to the wide range of industrial applications. Discrete Element Method (DEM) has become a popular choice of simulating particulate flows where the movement of particles is tracked in a Lagrangian manner. It solves the equations of motion of individual particles in the system to obtain the particles position, velocity and orientation. In this way, it is straightforward to consider cohesion forces between particles such as liquid bridge and van der Waals forces. One of the major challenges of DEM is the high computational cost with large number of particles. This makes it difficult or practically impossible to perform simulation of fine powders in a large-scale system.

Scaled-up particle model, which is sometimes referred to as coarse grain or discrete parcel model, has been increasingly popular to overcome the aforementioned problem: large particles are used in simulation to represent the original small particles. The scaled-up particle model in literature may be largely classified into the parameter scaling and the direct force scaling approaches. The former employs scaled-up physical properties and parameters (often based on some dimensionless parameters) to achieve similarity to the original particle system, whilst the latter first estimates the forces acting on the original particles using the original physical properties and variables, and then the resultant forces are directly scaled.

Recently, the authors proposed novel direct force scaling criteria based on the continuum approximation of powder flow. The scaling criteria for inter-particle and body forces are derived separately, and they can be universally applied regardless the forms of the cohesion force models. In addition, the overlap and inter-particle separation distance used for the force calculations are evaluated base on the geometric similarity to the original particle system. In the present study, the proposed scaled-up particle model is used to simulate various applications to investigate the validity of the model. It is found that the model can reasonably predict the original particle motion with different cohesion forces in terms of the bulk motion and velocity distribution.

DEM-Simulation of Drying and Calendering of Lithium-Ion Battery Electrodes

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Keywords | DEM, Electrode, Drying, Calendering, Battery

With the help of the discrete element method (DEM) software LIGGGHTS an approach was developed that enables the generation and characterization of particulate electrode structures with defined target properties such as porosity or coating thickness. In the first step, the drying process of the wet electrode film is simulated by mapping the active material and conductive carbon black particles. On this basis the resulting electrode structure is compacted in the calendering process, whereby the carbon black particles are transferred together with the binder into a solid bridge model.

The presentation explains how particulate electrode suspensions can be modeled via DEM and how a simulation set up for the structure generating film drying process can be built. It is also presented how it was possible to avoid coupling with a CFD simulation and still adequately represent the relevant fluid effects by using appropriate substitute models in order to depict the formation of the electrode structure from the wet film. In this way, both the required computing time and computing power can be greatly reduced. Furthermore, all relevant interparticle interactions were identified and implemented in the DEM code as contact models. Also, the influence of the preceding dry mixing and dispersing step on the carbon black structure can be taken into account. The drying model was validated using two model suspensions and different wet film thicknesses.

The electrode structure generated by the drying simulation is transferred to the calendering simulation. After a one-time calibration of the stiffness and damping behaviour of the inactive binder-carbon black network, the model is able to reproduce the experimentally achieved mechanical stresses and elastic strains of the particulate electrode for different calendering degrees and initial coating thicknesses.

The structures generated in this way can be used for further virtual analyses such as thermal or electrical conductivity. In addition, the transfer to an electrochemical simulation and the CFD-based simulation of electrolyte filling is being sought.

DEM-CFD modelling of flow rate under differential pressure

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Keywords | DEM-CFD, powder, flow rate, air pressure

We examine the flow of fine and cohesive powders through a hopper outlet under differential air pressure. A set of experimental measurements of the flow rate is presented for typical pharmaceutical powder excipients and an empirical flow equation is derived using dimensional analysis. The equation is similar to the Beverloo relationships but it also includes a pressure term. Coupled DEM-CFD analysis is carried out to match multiple experimental observations including packing density, critical orifice diameter and mass flow rate. By comparing the empirical and numerical data we derive relationships between particle properties and bulk powder behavior for the specific case of hopper discharge under differential pressure. The analysis identified density and surface energy as key particle parameters controlling the process. The results are of practical significance as it allows using the empirical flow rule with inputs from particle properties for the flow regime considered. It is shown that the flow behaviour of fine and cohesive powders is significantly affected by air pressure and pressure gradient. Air effects are critical in handling and dosing operations such as die fill on high speed rotary tablet presses used in pharmaceuticals.

Applying the Discrete Element Method (DEM) to Optimise the Design of a Lunar Mineral Tribocharger

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Keywords | Space Resource Utilisation, Tribocharging, DEM

The extraction and use of resources on the Moon, Mars and asteroids will transform space exploration. New technologies, adaptations of existing terrestrial technologies and novel approaches to mining and mineral processing flowsheets will be required to find, extract and handle these resources (Rasera et al, 2020; Hadler et al, 2020). For example, the separation of lunar soil (regolith) particles by size and particle type on the Moon presents challenges due to the high fraction of fine particles (-50 µm), the range of different components and environmental constraints (Rasera et al, 2020). On the Moon, favourable conditions exist for the triboelectric charging and electrostatic separation of the regolith, facilitating the enrichment of specific mineral components (Rasera et al, 2020).

This work describes the novel application of the discrete element method (DEM) to model and optimise the design and charge performance of a tribocharger under lunar environmental conditions. The DEM model employed has been validated previously using terrestrial experimental data (Rasera et al, 2022) and modified to replicate the lunar gravity and vacuum. The aim of these simulations is to determine the tribocharger configuration that maximises particle-wall contacts whilst minimising particle-particle interactions. Particle-wall contacts are desirable as the charge transfer is predictable based on the triboelectric series. Particle-particle contacts, however, are inherently stochastic, making it challenging to predict charge transfer for any given interaction. The optimal tribocharger is used to manipulate the electrostatic charge for a mineral mixture, prior to separation in a freefall separator under a fixed electric field. This study demonstrates the application of DEM to optimise equipment design for complex particulate systems.

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Computer Simulation of Bottom Hole Cleaning in Oil-Well Drilling operations using the Coupled DEM-CFD Method

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Keywords | Oil-well Drilling, Cleaning process,Coupled DEM-CFD

Oil-well cleaning operation is the ability of a drilling fluid to transport drilled cuttings from the bottom hole to the surface which has significant effects on drilling performance. In order to predict and prevent the cuttings accumulation at the bottom hole, it is essential to study the critical parameters affecting the cuttings transport. Computational model is one of the helpful methods to predict the hole-cleaning process as experiments are somewhat difficult and expensive to be carried out due to the harsh drilling conditions for oil and gas wells. Most of the modelling studies are limited to the methods which do not fully consider the discrete nature of cuttings in fluids. Nevertheless, very few investigations implemented fully coupled particle-fluid interactions, while there is a lack of focus and careful investigations of effect of cutting size with appropriate mesh configuration and refinement for particle-fluid interaction near the wall region. This is significantly important for the transportation, sedimentation and suspension of cuttings. This paper focuses on modelling of the hole-cleaning process using fully coupled computational fluid dynamics and discrete element method (CFD-DEM) approach with carful mesh configuration, specifically for particle-fluid interaction near the wall region. The rheology of fluid phase in the simulations is expressed by the Herschel-Bulkley non-Newtonian model, in an Eulerian framework (CFD) and the cuttings are modelled using the DEM method. The CFD-DEM coupled approach in this study, considers the particle-particle, particle-wall, particle- fluid and fluid-particle interactions. In this work, the effects of cuttings size, drill rotation, inclination angles, mud rheology and annular velocity on the cleaning efficiency are investigated. It has been found that the role of mud viscosity and annular velocity in improving the cleaning efficiency are dominant while they can be increased to their limiting values. Increasing the well deviation from vertical position leads to higher cuttings concentration particularly at the inclination angles close to horizontal. However, interestingly at low annular velocity the cuttings concentration at the inclined 45° annulus is found to be higher than the horizontal annulus due to the sliding motion of cuttings on the lower section of the annulus.

Effect of particle shape mixture on the powder spreading behaviour in Additive manufacturing: an optimisation by DEM study

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Keywords | DEM, cohesive powder, van der Waals forces, fine particles, reduced stiffness, packing, additive manufacturing

The application of discrete element modelling, DEM, to cohesive powders is challenging due to long simulation times. These come from both the time necessary to resolve the increased complexity of the contact model in these cases, and the small particle size which often leads to high particle numbers. This work shows that the accepted practice of reducing particle stiffness (Young's modulus) to accelerate the simulations fails to preserve the original behaviour for many cohesive systems. This was found to be due to the under-estimation of sliding and rolling resistances and, to a lesser extent, poor resolution of non-contact forces. A new methodology based on a modified sliding and rolling resistances is therefore proposed that allows scaling of the stiffness but preserves the particle behaviour in contact dominated systems. A new approach for calculation of non-contact van der Waals forces is also introduced that allows resolution of these non-contact forces without significant reduction of the time step.

These new approaches were evaluated using a series of simple test cases and demonstrated to give stiffness independent results when scaling the stiffness over several orders of magnitude. For example, the predictions of stop distance of a particle sliding and/or rolling over a flat surface were preserved when the stiffness was scaled down by almost four orders of magnitude, which was not possible with previous scaling approaches. A packing simulation was also performed, and the detailed packing structure was shown to be insensitive to stiffness when it was scaled over three orders of magnitude.

Finally, the approach was evaluated in via a simulation of a homogeneous shear layer. The ability to scale over a wide range of cohesiveness, normal stress and shear rate was tested, and the ability to scale the stiffness seen if the particle overlap was within bounds determined by the normal stress and the cohesivity. The method was developed to accelerate, accurate modelling of the spreading of cohesive particle layers for powder bed fusion additive manufacturing applications such as SLM/SLS (selective laser melting/sintering) & EBM (electron beam melting) but has significantly wider applications across many cohesive powder DEM applications.

Modelling Fluid-Structure Interaction and Particle Deposition on a Deformable Fibre using a Coupled CFD-DEM Framework

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Keywords | Filtration, Fluid-Structure Interaction, Discrete Element Method, Parallel Bond Model, Computational Fluid Dynamics, Immersed Boundary Method, CFD-DEM coupling, Model Calibration

Filters play an important role in many technical applications not only by assuring the functionality and efficiency of various machines but also by meeting emission regulations and environmental safety goals. To increase filter performance, i.e. increasing separation efficiency, decreasing filter pressure loss and increasing filter service life, is an ongoing challenge. While virtual or simulation-based filter media development has made great strides over the past decades, there are instances where model predictions are still poor. The present study aims to better understand the interdependency between filtration characteristics and the deformation of the fibrous filter media during loading conditions. As a first step, the interaction of the very basic unit of a fibrous filter, i.e., a deformable fibre, with particle-laden flow is investigated.

The objective is to predict the filtration and deformation characteristics of a single deformable filter fibre exposed to a particle laden crossflow with specified particle concentration and particle size distribution. Dynamics of particle deposition, particle-particle and particle-fibre interaction as well as fibre deformation due to particle loading and fluid forces are predicted based on a coupled CFD-DEM simulation approach.

Particulate matter and fibre structure are both modelled using the Discrete Element Method (DEM) with proper calibration of the force interaction model parameters. Specifically, a parallel-bonded-particle model is employed to describe the fibre material. Governing equations for the continuous fluid phase are solved using the Immersed Boundary Method CFD solver, *cfdemSolverIB,* from the OpenFoam-8 family of solvers including proper coupling with the DEM solution algorithm. Micro- scale parameters for the DEM-based model of the fibre material were calibrated to recover its macro-scale properties. DEM model parameters for the particulate matter such as friction coefficient and coefficient of restitution have been calibrated with the help of established calibration experiments.

The predicted structural deformation of the fibre is compared to classical Euler-Bernoulli beam theory for different loading conditions. Flow topology and pressure profile around a single fibre using the CFD-DEM coupled methodology is in good agreement with predictions from a single-phase CFD solution around the fibre. Effects of fibre deformation on particle deposition and on already deposited particles are discussed.

Fluidized bed dynamics with cylindrical particles: DEM simulation with the superquadric approach coupled with CFD

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Keywords | CFD-DEM, superquadric, rotor granulator, spouted bed, fluidization

In many industrial processes, fluidized bed rotor granulators are used to process a starting product such as extruded pellets into the final product, e.g., spheronized coated particles. In this work, the dynamics of cylindrical particles in a rotor granulator and spouted bed is investigated numerically and validated by experiments. Computational fluid dynamics (CFD) is coupled with the discrete element method (DEM) using the superquadric approach according to Barr (1981) to obtain additional information about the contact forces besides the general particle dynamics in the process (Atxutegi et al., 2021).

First, the applicability with regard to the deviation from reality of different drag models for the CFD-DEM simulation of cylindrical particles is analyzed based on experimental and numerical investigations on a spouted bed. The best model to describe the fluidization of the cylindrical particles in the spouted bed was found to be the correlation according to Sanjeevi et al. (2018). Validated by the average maximum bed height the deviation was less than 10%. In the second part, the particle dynamics in the rotor granulator at different operating points related to gas and rotor velocity was numerically investigated using the mentioned drag model. The gas velocity had a significant influence on turbulence, bed height and bed mixing. Too low gas velocities could not ensure sufficient particle mixing, while too high velocities reduce

the toroidal particle velocities. The rotor speed was found to have little influence on the particle dynamics, while too low rotation velocities resulted in insufficient mixing.

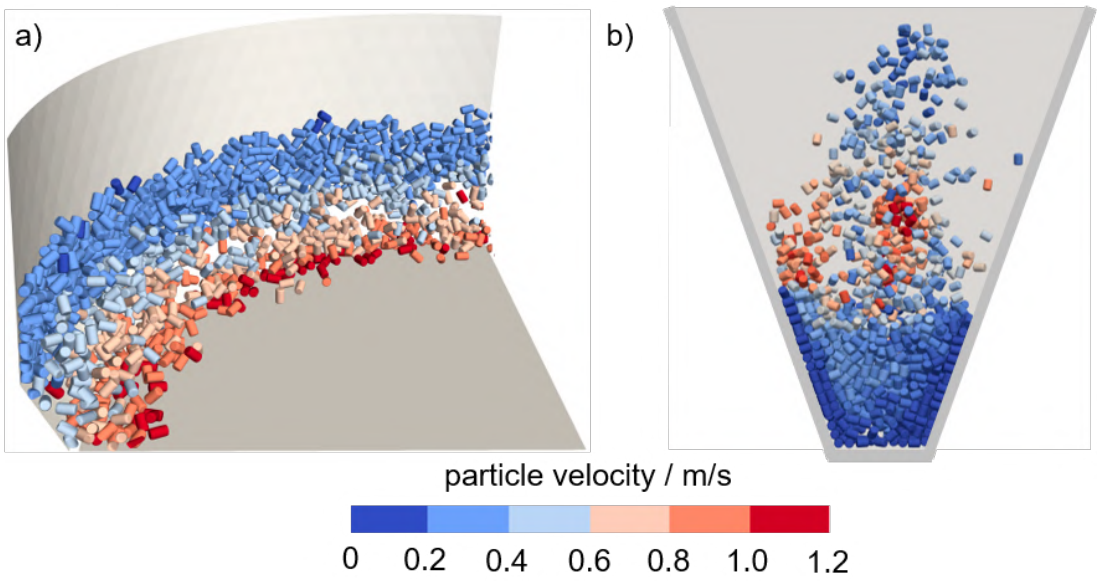


Figure: Particle velocity of the cylinders in a) rotor granulator, b) spouted bed

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An overview of DEM and CFD-DEM Modelling of Particle and Fluid-Particle applications, including Multi-Scale and Multi-Physics approaches

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Keywords | DEM, CFD-DEM, industrial applications, multi-scale, multi-physics

Discrete Element Method (DEM) and DEM coupled to Computational Fluid Dynamics (CFD-DEM) are established techniques for optimization and design of particle processes. Granular materials and flows involving fluids and granular particles are everywhere - in industry, environment and everyday life. Sugar, sand, ores, tablets, chemicals, biomass, detergents, plastics, crops, fruits need to be harvested, produced, processed, transported and stored.

Particle simulations with DEM and and particle-fluid simulations with CFD-DEM have become an essential tool for design and optimization of industrial and geotechnical processes.

Historically, DEM has been applied to geotechnical and mining applications first, where many application exist with manageable complexity and particle numbers.

As discussed in the related presentation “Simulating large scale particulate systems using a combined 3D-1D approach”, for more complex applications challenges often typically arise from a combination of large problem sizes, long process times and the fact, that phenomena that arise at micro scales influence the macroscopic behaviour of the system. Advanced models are available that allow for the depiction of complex

physical phenomena at very small scales, as well as coarser models for the mesoscopic scale and large-scale models for a whole reactor.

While in the past, DEM and CFD-DEM methods were tailored to a narrow field of applications, modern algorithms and scientific model implementations allow for real multi-scale and multi-physics application for more complex systems.

In this presentation, we focus on an overview of DEM and CFD-DEM application from industry and geotechnics. We give examples for successful application of DEM and CFD-DEM modelling in a variety of applications, such as automotive, chemical industry, pharmaceutical industry, consumer goods industry, agricultural machinery production, and plastics production.

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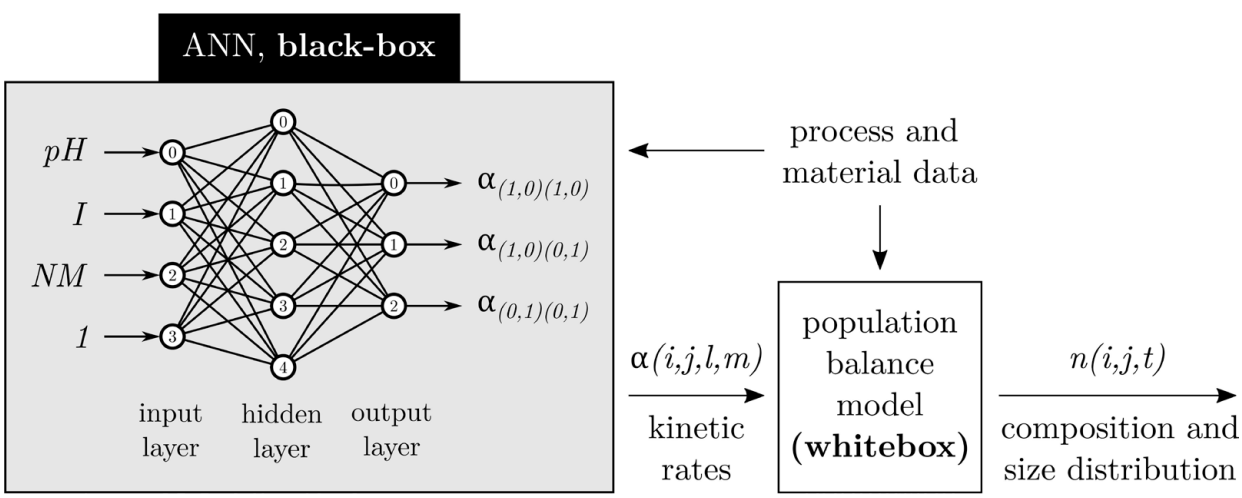
Hybrid Population Balance Modeling of Agglomerating Multicomponent Systems

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Keywords | population balance, hybrid, greybox, machine learning, data-driven

The demands on separation technology have steadily increased over recent years. Highly specific, selective separation techniques that are also suitable for fine particles in dilute suspensions, are needed more and more. In this work, magnetic seeded filtration (MSF) is presented as a separation concept that is capable of handling these challenges. Magnetic seed particles are added to a suspension and after agglomeration with the target particles, the agglomerates are removed by magnetic separation.

This presentation shows recent findings from theoretical investigations on MSF. In principle, hetero-agglomeration processes can be described using 2D or 3D population balances, depending on how many components are considered in the system. Nonlinear discretization methods allow the consideration of broad agglomerate size distributions in acceptable computational time, i.e., at least in real time. This is particularly interesting with respect to potential use in model predictive control. However, the main challenge in modeling real systems is the availability of reliable and comprehensive material data. In particular, pH-dependent surface potentials, Hamaker constants, but also fundamental correlations for the hydrophobic interaction are either unavailable or subject to errors. This totality of uncertainties complicates or prevents purely predictive modeling. Correction factors based on experimental data, are often used, resulting in complex multi- dimensional optimization problems. This work presents an alternative approach, in which the calculation of agglomeration efficiencies is performed with data-driven models from the field of machine learning (black box). Subsequently, the resulting data flows into an established mechanistic PB model (whitebox). This interconnection results in a so-called greybox or hybrid model that combines the advantages of data-driven and physics-based approaches: Model complexity is reduced, while the information gain through process modeling remains intact.



A Dual Population Balance Approach for Simulation of Particle Formation from Precursor Laden Droplets

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Keywords | Multi-phase Physics Population Balance Particle Formation GPU

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Flame spray synthesis is a promising method for large scale production of nanoparticles. In contrast to conventional flame synthesis one is not restricted to gaseous precursor chemicals. Instead the liquid precursor solution is vaporized via a pressurized spray nozzle. This gives access to a wider variety of precursor chemicals. However, achieving desired particle properties is more difficult as multi-phase interactions between particles, spray droplets and flame need to be considered.

In this work, the Dual Population Balance Monte-Carlo Method (DPBMC) is applied to investigate particle growth mechanisms inside spray droplets and flame. Hereby, the history of individual particles is tracked starting from precipitation in droplets to agglomeration in the gas phase. Furthermore, droplet micro explosions are successfully predicted by accounting for particle accumulation at droplet surface.

Initial and boundary conditions are set according to Suleiman (2021) and compared against experiment. Comparison between experiment and simulation is shown in Figure 1. Hereby, TEM and SMPS-measurements are used to find mean particle size and concentration along central axis of the flame spray reactor. The simulation correctly reproduces the 'no growth' behavior of particles. As particles already coagulate inside droplets the final particle concentration in the gas phase is too low for growth by coagulation.

As a conclusion the DPBMC method can be used to investigate the role of droplet properties on particle formation in spray synthesis reactors.

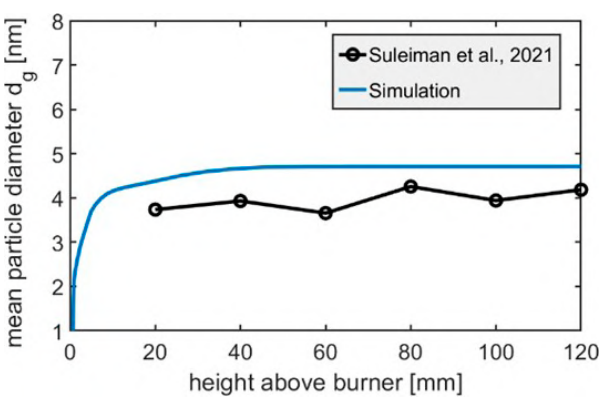


Figure 1: Simulation and experimental characterization of mean particle size evolution in flame spray reactor.

Acknowledgement

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A computational fluid dynamics - population balance approach for evaporating

cough droplets transport

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The spreading of COVID-19 pandemic makes relevant the study of the evolution of virus-laden droplets during cough or talking to break the transmission chain and control the pandemic. The evolution of expelled droplets is complex, being the system intrinsically multiphase, polydisperse, multicomponent and potentially affected by significant evaporation due to the interaction with the surrounding environment. In most of the studies the Eulerian-Lagrangian (E-L) method, based on tracking the motion of individual droplets, is used, with the drawback of computational costs that scales with the number of considered droplets. To tackle this problem, the Eulerian-Eulerian (E-E) method, treating the dispersed phase as a continuum, is used in this study. Considering the role of polydispersity and evaporation, the population balance equation (PBE) is coupled with E-E method to describe the droplets evolution. Two methods, sectional method (SM) and extended quadrature method of moments (EQMOM), are applied to the solution of the PBE, of which the performances are compared. The codes of this E-E/PBE model are developed based on the open-source software OpenFOAM and the OpenQBMM module. The codes are verified with the numerical results of freely falling evaporating droplets in literature (Redrow et al., 2011 and Wei and Li, 2015) first and then applied to the evolution of cough droplets in a room (Li et al., 2018). The results show that the predicted diameter changes during evaporation of SM and EQMOM agree well with those in literature. The developed codes can be applied to evaporating polydisperse multiphase flows. In the simulations of cough droplets in room, it is found that the larger droplets (>100μm) fall on the ground while the smaller ones (≤100μm) show a trend of suspending in the air. Evaporation would further decrease the size of smaller droplets and keep them suspend in air. Additionally, even the larger droplets travel a distance longer than the current social distance (1m) before settling on the ground, which would be beneficial for making social distancing guidelines. The novelty of this study lies in the implementation and application of E- E/PBE method in evaporating cough droplets evolution.

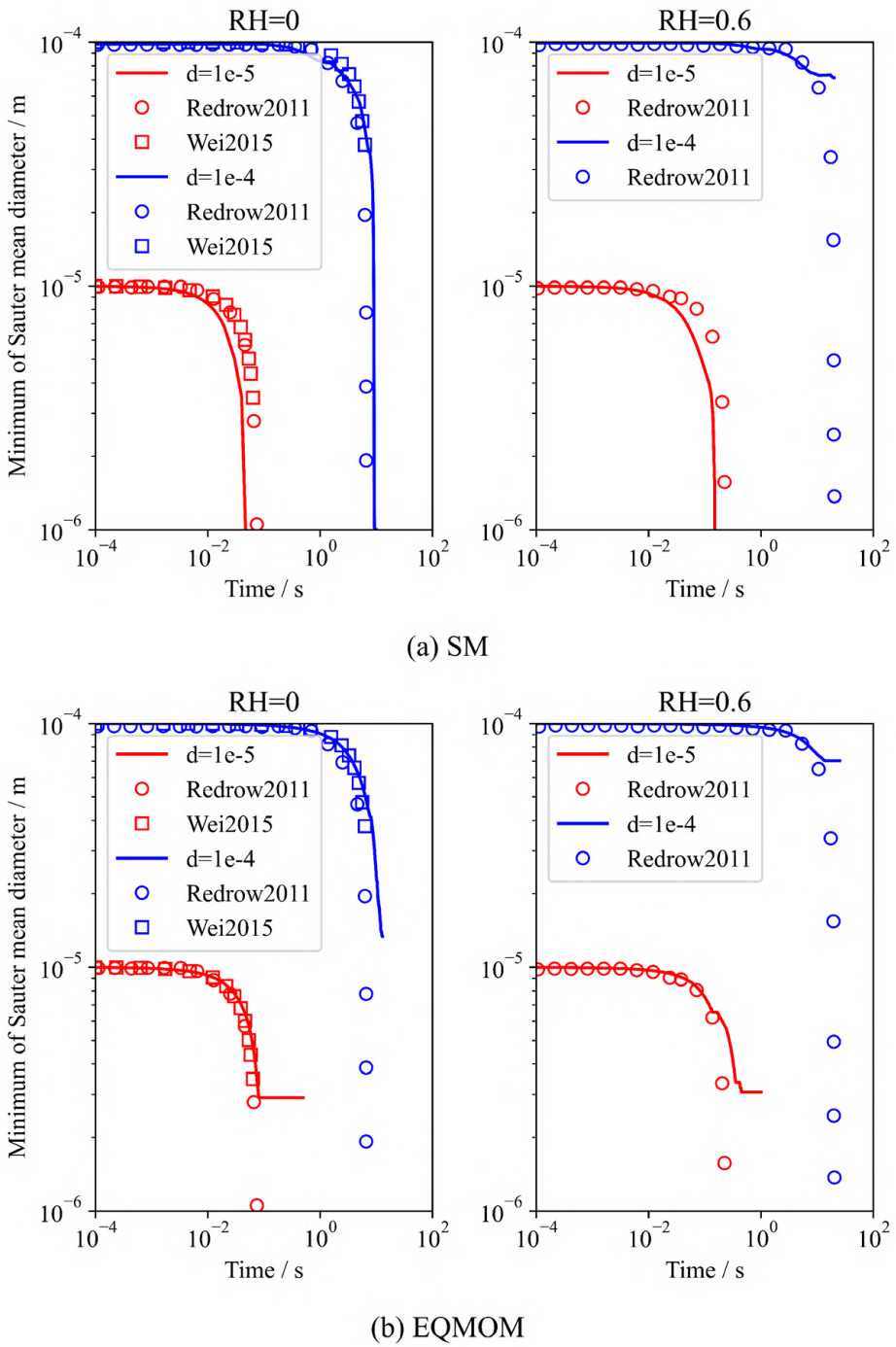


Figure 1. Evaporation verification

Formation dynamics of noble metal nanoparticles

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Keywords | Colloidal Synthesis Kinetic modelling Plasmonic Noblemetal Nanoparticles Population Balance Equation

Noble metal nanoparticles (NPs), in particular Ag and Au NPs, show interesting optical properties, due to localized surface plasmon resonance (LSPR). The LSPR depends on the metal as well as on the size and shape of the synthesized nanoparticles, which makes them interesting for

sensing applications (Lu et al. 2009; Petryayeva and Krull 2011). The synthesis routes of small noble metal NPs consist of various coupled subprocesses: First, a reduction of the noble metal salt to metal atoms, second homogeneous nucleation followed by autocatalytic growth (Sandoe et al. 2019). For defined optical properties, a narrow particle size distribution (PSD) with uniform shape is required (Wagner et al. 2008).

We investigated the kinetics of the nucleation and growth of the small NPs via the temporal evolution of the LSPR by in-situ UV-Vis spectroscopy. From the spectra the NP concentration and mean particle size can be determined as a function of time. Additionally, the final NPs size distribution was measured by analytical ultracentrifugation (AUC) and image analysis of transmission electron microscope (TEM, STEM) micrographs. In a second step, we are developing a model based on population balance equations, which is capable to predict the time evolution of the dispersed phase. We will show time- resolved UV-Vis spectra of Au seeds formation for different process conditions, which serves as an experimental input for the modelling development. We will discuss the impact of pH, ionic strength, mixing rate, stabilizer and initial concentration on the product outcome and relate the outcome to the kinetic behaviour. These seeds are subsequently for the growth of gold and silver nanoparticles of different sizes and shapes both in batch and continuous operation.

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Modelling microstructure-dependent momentum and heat exchange in dense particle-fluid flows using particle-resolved Lattice Boltzmann simulations

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Keywords | Lattice Boltzmann Method (LBM), Direct Numerical Simulations (DNS), Multiphase flows, Particle-laden flows

Particle-laden flows occur in a wide range of processes in chemical engineering such as fluidized bed reactors. Numerical simulations of these multiphase flows can be performed on different scales. Particle-resolved simulations allow the most detailed analysis of the particle-fluid interactions, because therein the flow field around the individual particles is resolved and particle-fluid forces and heat exchange rates can be directly evaluated. As a result, a variety of momentum and heat transfer correlations were obtained from such direct numerical simulations (e.g. Kravets et al., 2019), which are used as constitutive closure models for particle-unresolved simulations. In the case of momentum exchange, the most commonly applied approach for the modelling of particle-fluid forces has been the consideration of a mean drag force that is given by the average force the fluid flow excites on a rigid particle in mean flow direction of a particle-fluid system with a specified porosity and Reynolds number. However, studies (Rosemann et al., 2020; Kravets et al., 2021) have shown that these drag laws cannot accurately capture the significant scattering of forces in magnitude and direction arising from the microscopic flow structures through the gaps between the unevenly distributed particles. Here, we present a methodology for deriving microstructure-dependent momentum exchange models based on the correlation between the relative position of particles and the particle-fluid forces acting upon the particles under various flow conditions. For this purpose, particle-resolved flow simulations are carried out using the Lattice Boltzman Method. The study is extended to problems with conjugate heat transfer, since Nusselt numbers are also prone to scattering that is not considered in existing heat exchange correlations. The derivation of models is based on regression analysis aiming at correlating variables representing the relative position of particles around a reference particle with forces and Nusselt numbers (see Fig. 1). These variables are computed using a smoothing kernel that is limiting the number of surrounding particles affecting momentum or heat exchange. Additionally, we consider an approach based on the training of a neural network taking into account the position data of a specified number of the closest neighbor particles to obtain correlations.

Scaling laws for particulate precipitation processes

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Keywords | CFD, PBE, precipitation, scale-up

Motivation

Precipitation is one of the most common processes in the production of particulate products from homogeneous solutions (see Fig. 1a). Although precipitation is ubiquitous in particle synthesis, overarching relationships between process conditions and the outcome of precipitation are still lacking. Therefore, even a simple variation in the concentration of precursor phases often leads to counter-intuitive, unexpected results. In particular, the transfer between chemically different systems remains an elusive task. We present a widely applicable framework for the prediction of the size evolution of different particle systems including organic drug particles, inorganic salts, pigments, and noble metal nanoparticles. The interaction of the individual subprocess steps is shown and discussed using dimensionless numbers [2]. Using these dimensionless numbers, we uncover overarching scaling laws between mean particle size and process conditions that enable physically motivated upscaling strategies (see Fig. 2a).

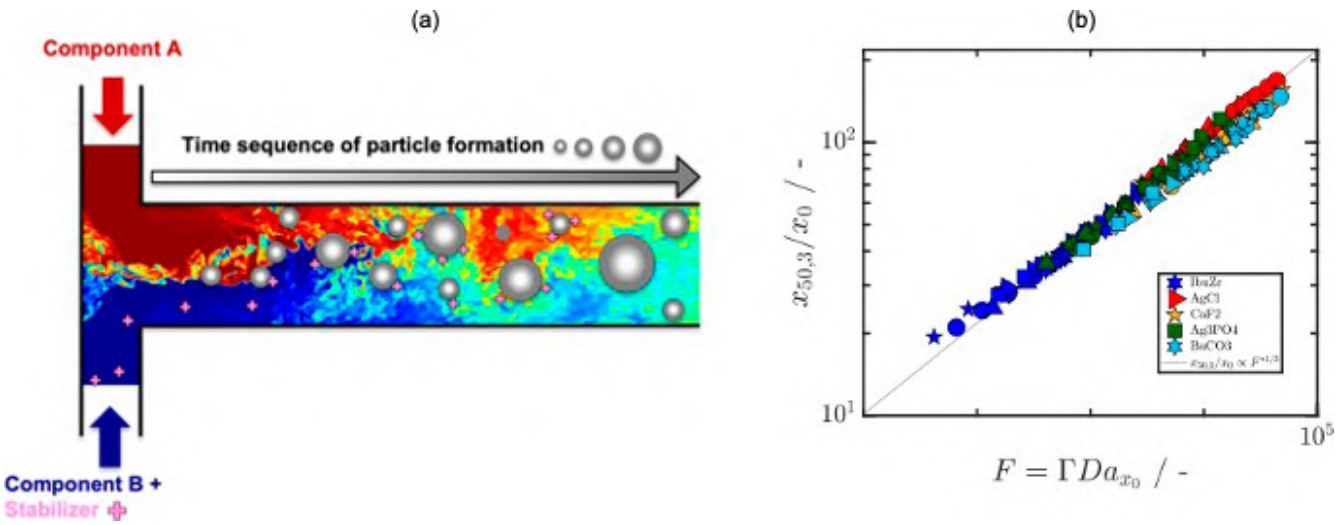


Fig. 1: (a) Illustration of the precipitation process consisting of turbulent mixing, supersaturation build-up, homogeneous nucleation, transport-limited growth, and stabilization. (b) Dimensionless mean particle size as a function of a dimensionless process function, which is a product of dimensionless interfacial energy and a characteristic Damköhler number. Different colors represent different chemical systems: Ibuprofen-Zirconium (IbuZr), Silver Chloride (AgCl), Calcium Fluorid (CaF2), Silver Phosphate (Ag3Po4) and Barium Carbonate (BaCO3).

Simulation approach

We perform direct numerical simulations (see the snapshot of mixing turbulent mixing process in the Fig.1a) of the precipitation process in a T-mixer by resolving the smallest velocity length scales. The coupling between the continuous and dispersed phase is established with a Euler-Lagrange approach, while the solid formation is described by a population balance equation (PBE). Concerning the dispersed phase, homogeneous nucleation and transport-limited growth govern the solid formation process. Along each Lagrangian trajectory, the PBE is numerically approximated with the recently developed exact method of moments along each Lagrangian trajectory. The supersaturation links mixing and solid formation, which is calculated either through the solubility product or the equilibrium solubility depending on the chemical system.

The Influence of Surface Functional Groups on the Triboelectrification of Organic Crystals

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(1) University of leeds, (2) University of Leeds, (3) AstraZeneca, (4) Malvern Panalytical, (5) Pfizer

Keywords | Triboelectric charging. DFT.

Triboelectrification is a phenomenon that is highly relevant in the formulation and manufacturing of powder systems. Uncontrolled and excessive charging of powders can lead to charge accumulation, causing unwanted agglomeration, adhesion to vessel walls and in extreme cases dangerous electrostatic discharges. Despite its ubiquity, the underlying mechanisms of triboelectric charging remain poorly understood

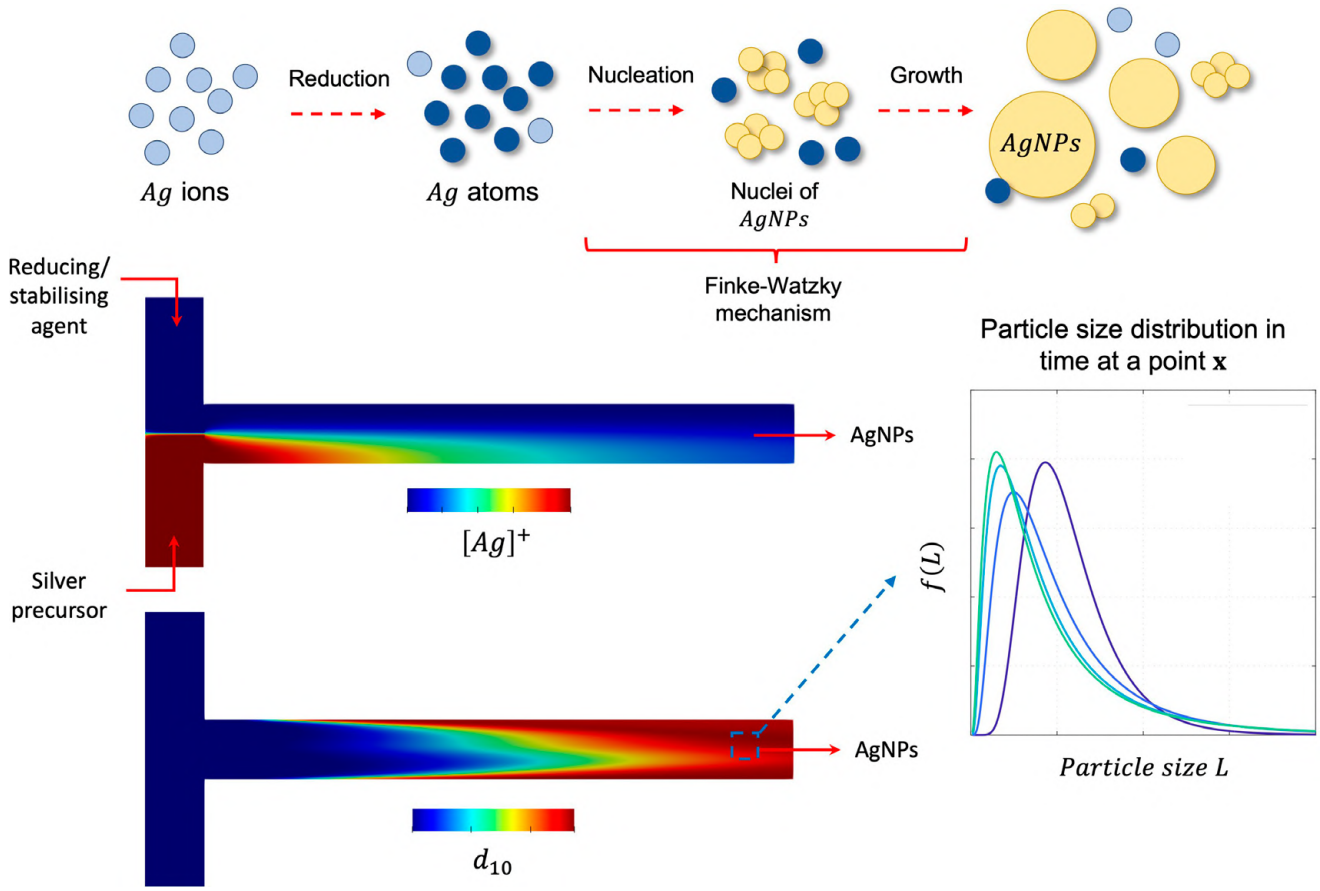
as the process is very sensitive to material properties and environmental conditions and it is very difficult to keep these factors consistent across experiments. In this work we address the influence of functional groups and adsorbed water, at the surfaces of organic crystals, on charge transfer using first principles modelling techniques. The materials of interest are organic crystals relevant to pharmaceutical applications. These surfaces are studied using the CASTEP simulation package to perform quantum mechanical Density functional theory (DFT) calculations to investigate the structure and electronic energies of surfaces at the atomic scale, which is used to investigate the driving force of charge transfer at a given contact by characterising surface work function and density of states. It is hoped that this work will further the understanding of the behaviour of the charge carriers at the surface of organic crystals which govern triboelectrification, how humidity and adsorbed surface water impact charge transfer and contribute to the development of a predicative model to describe the charge transfer between materials. It is observed that water will produce a work function shift in metal surfaces, offering an alternative perspective that surface water facilitates electron transfer rather than being involved in ions transfer.

Effect of Operating Conditions on the Chemical Synthesis of Silver Nanoparticles in Microfluidics: coupled PBM-CFD Simulations

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(1) Imperial College London, (2) University of Birmingham

Keywords | Silver nanoparticles; microfluidics; population balance model; computational fluid dynamics; reactive kinetics; Finke-Watzky

Silver nanoparticles (AgNPs) have attracted increasing attention in the past few years as a result of their unique physicochemical properties, which make them particularly suitable for a vast array of applications. These include food packaging, localised drug delivery, cancer treatments, catalysis, and inkjet printing, among others (Nathanael et al., 2022). Microfluidics has recently emerged as a viable approach for the synthesis of AgNPs with tightly-controlled properties and low size polydispersity. In comparison to traditional methods, microfluidics has been demonstrated to provide more efficient heat and mass transfer, mixing, and control over the operating conditions (Badilescu and Packirisamy, 2012). We propose a meso- and macroscale computational model of microfluidic synthesis of AgNPs in order to assess the effect of different operating conditions on the final particle size and particle size distribution (PSD). The simulations are based on a population balance model-computational fluid dynamics (PBM-CFD) formulation for diluted systems with reactive kinetics for three synthesis stages: reduction of silver ions into silver atoms, nucleation of nanoparticles (NPs), and growth. The PBM was solved by means of a quadrature-based method of moments for a length-based univariate number density function. Nucleation and size-dependent growth were implemented based on experimental measurements, in line with the Finke-Watzky two-step mechanism (Watzky and Finke, 1997). The code was implemented on the open-source tool of PBM solvers OpenQBMM and validated against experimental data sampled through dynamic light scattering and silver electrode measurements. The results show that the competition between mass diffusivity and reduction reaction kinetics has a major effect on the concentration of silver atoms and therefore the driving forces for NPs nucleation and growth. We also show that some PBM-related parameters, such as critical nucleus size, aggregation efficiency, and growth rate, critically influence the reconstructed PSD in terms of average size, standard deviation, and polydispersity index.



Small scale crystallisation kinetics: Leveraging microscopy imaging and immersion cell growth studies to model and control particle morphology

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Keywords | Population balance modelling, growth kinetics, particle size distribution, morphology

Crystallisation is a critical step in the isolation of active ingredients (AI) for agrochemical production and has major impacts on downstream processing and ultimately product quality. Key performance indicators of the crystallisation process include particle size and morphology and a detailed understanding of the system kinetics is necessary if desirable particle characteristics are to be obtained. Crystallisation phenomena are made of several competing processes including particle growth, nucleation, and agglomeration. Population balance models (PBM) can be used to capture the contributions from each of these mechanisms, although obtaining accurate parameters is a major challenge in the development of a high- fidelity model.

Our work focuses on the development of new systematic workflows for constructing mechanistic population balance models using targeted methods and precise measurements to ensure model accuracy. A variety of experimental measurements are incorporated to understand crystal growth behaviour at varying supersaturations and temperatures including isothermal desupersaturation experiments, particle size distribution measurements, and immersion cell growth studies. We find that the use of microscopy imaging provides the most reliable measurements of particle size and shape, while immersion cell growth studies can elucidate relative kinetics along different crystal facets. These are combined in a 2-dimensional morphological crystallizer model in gPROMS FormulatedProducts (gFP) to simulate changes in particle size and shape under different crystallisation conditions. Custom modifications are also made to the standard morphological crystallizer model in gFP to provide closer links with experimental measurements and understand the effects of process conditions on particle aspect ratio in more detail. Finally, the model is used to assess how the morphology of the particles are influenced by seeding conditions with the aim to target desirable process operating conditions and produce low aspect ratio particles.

CFD Simulation of The Entire Circulating Fluidized Bed (CFB) Carbon Capture Loop Using Solid Supported Amine Sorbents

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(1) IIT, (2) Energy Research Corporation

Keywords | CFD, Carbon Capture, Circulating fluidized bed

Carbon capture using solid sorbents in considered to be a very promising alternative for aqueous solutions of amine-based absorbents to reduce the cost of regeneration during carbon capture and regeneration processes. The computational fluid dynamics (CFD) approach which is an advanced tool for design and scale up fluid-particle processes was used in this study to simulate two- dimensional gas and solid flow in a circulating fluidized bed (CFB) that was designed by the National Energy Technology Laboratory (NETL) for carbon capture using solid supported amine sorbents. To obtain better understanding of the complex flow patterns of particles and fluid through entire CFB loop, a two-dimensional non-reactive CFD simulations of the entire loop including the carbonator, regenerator, the solid circulation rate and pressure drop at different locations of CFB were compared with the experimental data.

In addition, two-dimensional reactive simulation of the CFB carbon capture unit was also performed to study the extent of CO2 adsorption and regeneration using amine-based solid sorbents in the temperature swing carbonation regeneration combined system.

A Multiphase Operator Splitting Approach for the Multiscale Euler-Euler Simulation of Reactive Industrial Fluidized Units

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Keywords | Euler-Euler, Fluidized bed reactor, Multiphase operator splitting, Multiscale modeling

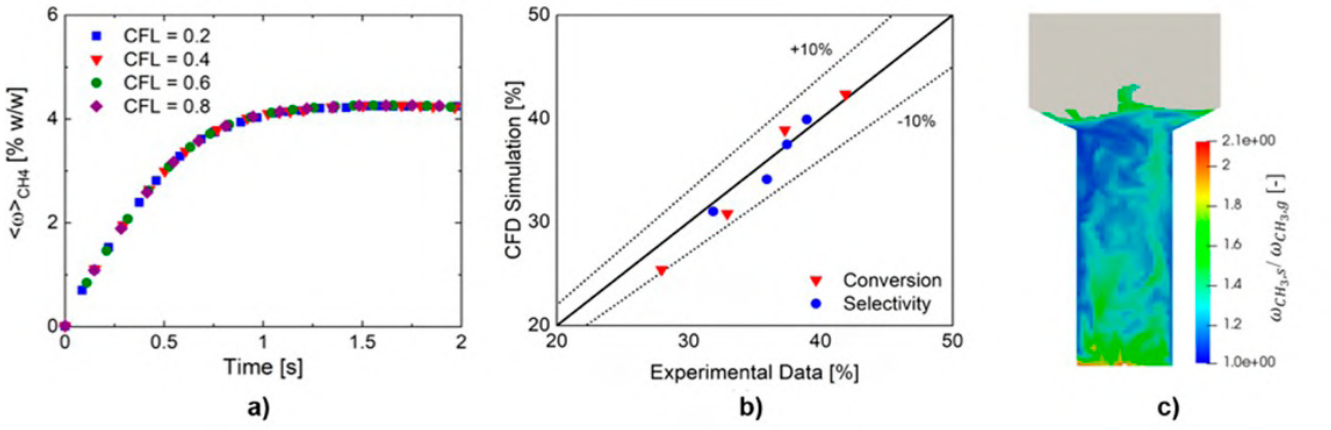
The design and scale-up of fluidized units is challenging and motivates the development of computational approaches aimed at assisting the experimental investigation. Contextually, the computational multiscale model (Maestri & Cuoci, 2013) is acknowledged as a promising strategy since it describes in detail all the phenomena occurring in the reactive environment (Bruix et al., 2019).

The multiscale modeling has been successfully exploited for lab-scale units (Uglietti et al., 2018). However, the numerical stiffness of the detailed chemistry hampers its applicability to industrial systems, because its dynamics cannot be managed with the small simulation time steps needed (Jašo et al., 2011). Hence, in this work, we develop the Multiphase Operator Splitting (MOS) approach which allows for the multiscale investigation of industrial fluidized systems. Accordingly, the energy and species balances are solved by adopting a two-step sequential procedure. First, the advection terms are considered and solved. Then, the homogeneous and the heterogeneous chemistries and the gas-solid transport as well are solved with an ODE solver. In doing so, the MOS allows for the coupled evolution of the gas and solid phases, avoiding the decoupling of phenomena strongly intertwined.

The MOS is applied in a lab-scale configuration whose reactivity is described by a microkinetic mechanism of the Oxidative Coupling of Methane process (Micale et al., 2021). Its predictions are only slightly dependent on the time step (Figure 1a), in contrast to the literature ones (Micale et al., 2021), and they are in good agreement with experimental data (Jašo et al., 2011) (Figure 1b). Then, an industrial scale configuration has been simulated with the same detailed mechanism. The MOS enabled the management of the detailed chemical kinetics along the whole dynamic of the industrial systems (about 50s) with an average computational cost of 6 h per simulated second providing insights of the process which cannot be caught with simplified macroscopic kinetics (e.g., the methyl radical gas-solid mass transfer limitations shown in Figure 1c).

As a whole, the work paves the way for the combination of computational modeling and experimental campaigns for the design and scale-up of fluidized systems.

EU's Horizon 2020 (G.A. 814416 ReaxPro) is acknowledged.



Comparison of stochastic turbulent dispersion models in predicting particle collection efficiency in Euler-Lagrange cyclone numerical simulations

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Keywords | cyclone, Lagrange, stochastic turbulent dispersion, DRW, CFD, efficiency, Fluent, OpenFOAM

Given the low volumetric fractions of solids in cyclones, CFD numerical simulations for predicting cyclone collection efficiency are usually of the Euler-Lagrange type. However, it is noted in the literature that only LES simulations can correctly predict the separation efficiency of particles smaller than 2 μm. Some works point to the importance of using stochastic models of turbulent dispersion, such as the DRW (Discrete Random Walking), in conjunction with RANS simulations. This work thus aimed to evaluate the use of the DRW model with RANS Euler-Lagrange cyclone numerical simulations, using a one-way coupling Lagrangian method and two different CFD software, ANSYS Fluent 19-R2 and OpenFOAM 7. The *icoUncoupledKinematicParcelFoam* solver in OpenFOAM has been used, where the implemented DRW model considers isotropy of the velocity fluctuating components. In Fluent, DRW considers their anisotropy. Two integration schemes were evaluated: the Euler-Implicit (I) and the Analytical-Simplified (A). A Stairmand high-efficiency cyclone with a diameter of 29.0 cm was simulated in a transient regime for 2.0 s and Δt=0.0001 s. Inlet feed consisted of a continuous air flux, and a dispersed phase of solid spherical particles with diameters between 0.5 μm and 7.0 μm. The structured computational mesh had 393000 elements. The Reynolds stress turbulence model of Gibson and Launder was used. The experimental data from Zhao (2005) was used for validating the simulations, whose inlet particle size distribution was adjusted to a sigmoid with parameters β=3.3 and =1.56 μm. Figure 1 shows that only the anisotropic DRW with the first- order Euler-Implicit (I) scheme agrees with experiments. The Analytical-Simplified (A) scheme in Fluent resulted in an overestimation of the particle collection efficiency. In OpenFOAM, neither of the two schemes accurately predicted the particle size efficiency, highlighting the importance of considering the anisotropy of fluctuations in the DRW model for accurate prediction. If the DRW model is not used, small particles become trapped in OpenFOAM simulations, and Fluent overestimates their separation efficiency significantly, corroborating the need for DRW in RANS Euler-Lagrange cyclone numerical simulations.

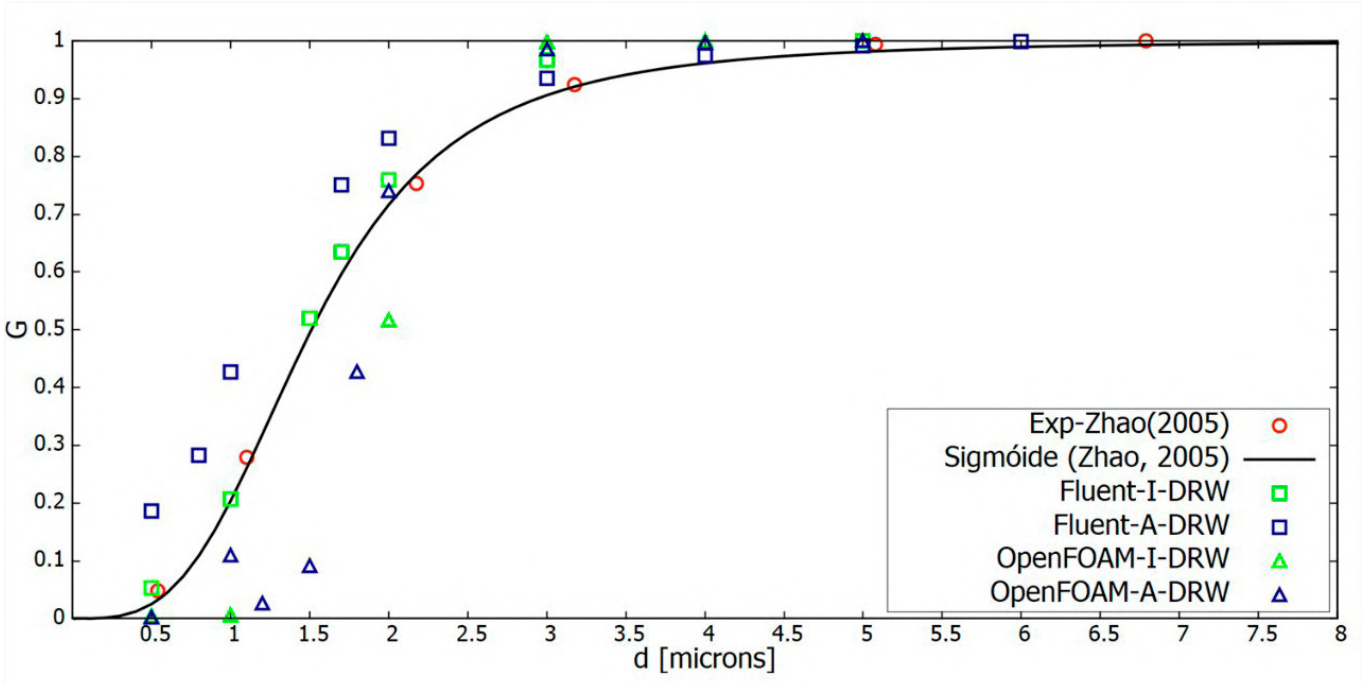


Figure 1. Grade efficiency (G) as a function of particle size (d).

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Assessment of the operability range of dynamically structured gas-solid fluidized bed reactors

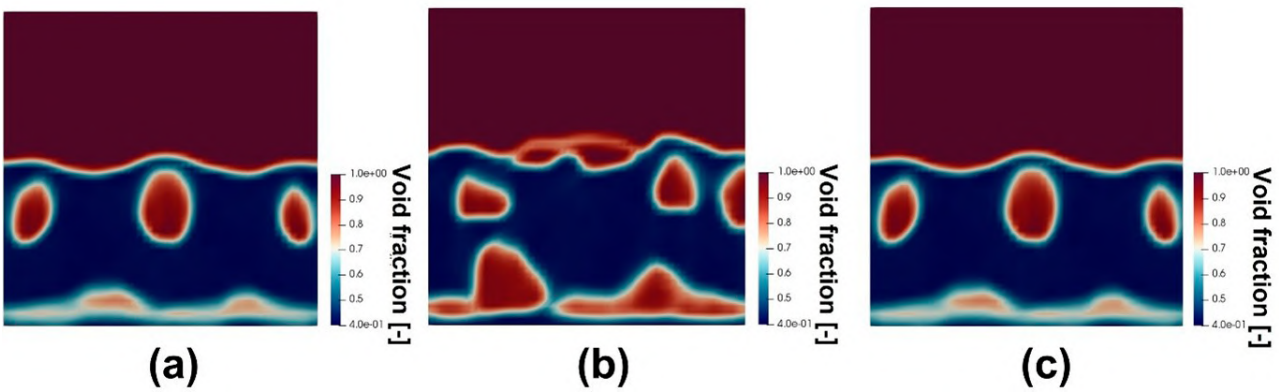
Cafaro, Davide (1); Micale, Daniele (1); Uglietti, Riccardo (1); Wu, Kaiqiao (2); Bracconi, Mauro (1); Coppens, Marc- Olivier (2); Maestri, Matteo (1)
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Keywords | Dynamically structured fluidzation, Euler-Lagrange, Fluidized bed reactors, Pulsed Fluidized Beds

Fluidized bed reactors show a complex fluid dynamic behavior, which complicates their scale-up. A possible solution to simplify their design is to achieve an ordered bubble pattern by supplying a periodic inlet gas flow. Experimental investigations have demonstrated that the bubble structuring strongly depends on the solid and gas properties [Francia et al., 2021, 2022]. Up to now, only room temperature conditions with a gas viscosity of 1.8x10-5Pa*s (Figure 1a) have been analyzed [Francia et al., 2022]. However, similar analysis in operating conditions commonly used in catalytic processes (e.g., high temperature) is still missing. Thus, in this work, we numerically analyze pulsed reactors to investigate the effect of the transport properties and pulsing parameters on the stability of the structured fluidization by considering a gas viscosity in the range of 3-5x10-5Pa*s. We observed that by using the same pulsed flow characteristics adopted in the literature [Francia et al., 2021, 2022] and a gas viscosity of 3x10-5Pa*s an irregular bubble pattern is obtained (Figure 1b), instead of the regular pattern at room temperature. By reducing the fluidization ratio, i.e., the average gas velocity normalized by the minimum fluidization velocity (A), it is possible to re-establish the structured fluidization (Figure 1c), showing that the lower the value of A the higher the bubble structure quality; this trend is even more pronounced by increasing the gas viscosity up to 5x10-5Pa*s.

In conclusion, this study highlights how the gas viscosity strongly affect the bubble structuring, and thus the pulsation parameters must be properly tuned to achieve an ordered bubble pattern in any operating conditions. Therefore, this analysis paves the way towards the investigation of dynamically structured fluidized units for heterogeneous catalysis.

Funding from European Union's Horizon 2020 under G.A. 14416 (ReaxPro) is acknowledged.



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Estimation of DEM contact parameters based on block-like motion characterization for cohesive powders

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Keywords | cohesive powders, block-like motion, characterization, rotating drum, DEM

For strongly cohesive powders, the influence of the consolidation state gets prominent and therefore makes a reliable characterization, especially in the low consolidation state regime, challenging. Examples of such powders are the materials used in powder metallurgy, which often consist of extremely small and irregular grains. Besides the consolidation state, the flow behavior depends on various factors like aging and environmental conditions. A reliable computer simulation is essential to understand how these powders behave in different flow situations in their current state. A powerful tool to model the flow of powders is the Discrete Element Method (DEM) (Cundall & Strack, 1979). However, it relies on material parameters which can be tricky to estimate for these cohesive powders.

In this talk, we first report on a technique that characterizes the powder's dynamics in a rotating drum setup. Second, we describe how we employed the results to fit DEM material parameters. The block analysis method (Kronlachner et al., 2022) uses a neural network-based optical flow estimation (Bar-Haim & Wolf, 2020) to determine a two-dimensional velocity field of the cross-section of the drum. This is used to track block-like structures breaking from the rigid-looking powder bed rotating with the drum, which are described in terms of size, velocity, and direction.

Further, this technique can also be employed directly on DEM simulations, enabling a good comparison between experimental and simulation results. Based on this comparison, accurate material parameters for the current powder state can be obtained with the help of optimization. We used the DEM solver LIGGGHTS (Kloss et al., 2012; Nasato et al., 2015) and tried to get contact-mechanical parameters of the widely used Hertz model, including cohesion and rolling friction representing our measurements. The results lead us to conclude that simple DEM models of this type cannot fully describe the complicated behavior of such cohesive powders. However, the results are significantly improved by using a modified rolling friction model which resembles stick-slip friction. This model enables reasonable matching of material behavior and the simulation of industrially relevant processes.

Contact Parameter Selection - an Approach based on Multi- objective Optimization and Metamodeling

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Keywords | DEM; Calibration; Optimization; Machine Learning; Multi-objective;

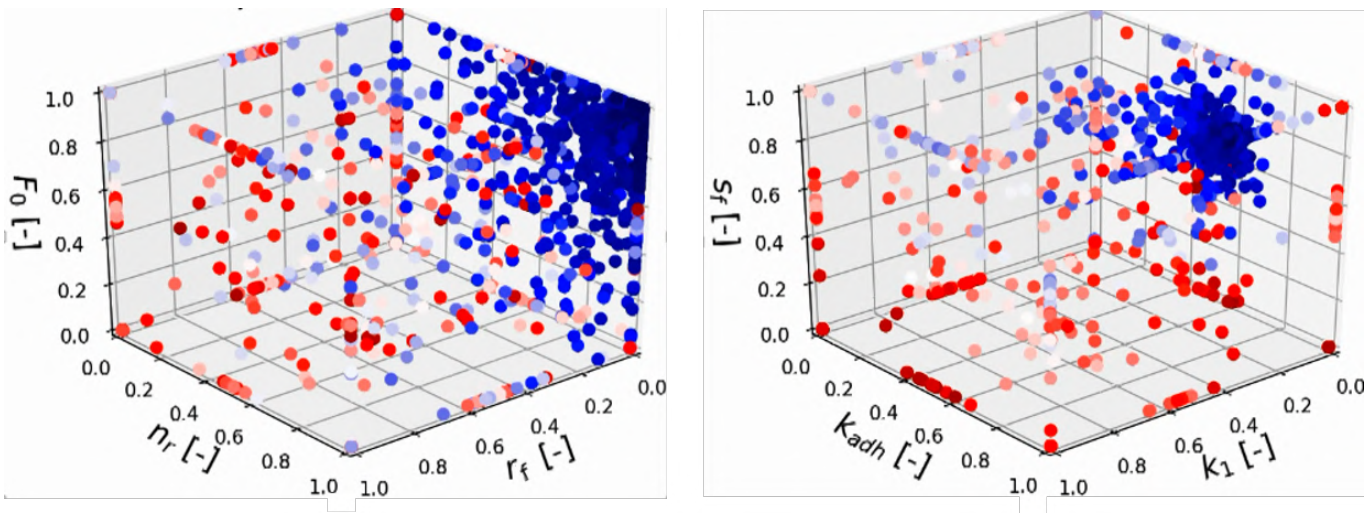
The specification of contact parameters in a discrete element method (DEM) based approach remains one of the key challenges in order to describe a powder´s flow. Ideally, in such a calibration various macroscopic responses should be targeted to identify a suitable set of contact parameters. Due to the complexity of DEM contact models, the input space presents itself with at least 10 degrees of freedom. In addition, each parameter of the input space will influence the target macroscopic behaviour with different magnitude and direction.

The macroscopic target space is of high-dimensionality itself since several bulk powder tests are common throughout industries. The delicate task arises to develop a framework that remains flexible and computationally feasible.

Besides reviews on the DEM parameter calibration [1], a handful of multi-objective optimization techniques related to DEM parameter calibration have been reported ([2], [3]). Yet, they do not remain truly efficient for the above mentioned problem.

In the current contribution we present a novel approach by combining (i) initial DEM parametric studies, (ii) regression models with data augmentation, (iii) training of surrogate models and (iv) a multi-objective optimization algorithm.

We apply this workflow to multiple bulk tests while outlining the DEM parameter importance. As a result, contact parameters for model powders are identified in a fast and effective manner. Figure 1 depicts the outcome of the optimization of six contact parameters while matching the yield locus, compressibility and relaxation powder bed height, static and dynamic AoR. The points are colored according to the distance from the target.



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Development and application of a Quick Image Analysis Calibration Procedure for In-situ Crystal Size Measurement

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Keywords | Crystallization, Image Analysis, Calibration

Crystallization is a purity and particle control unit operation commonly used in industries such as pharmaceuticals, agrochemicals, and energetics. Often, the crystal mean size, morphology, and distribution can impact the critical quality attributes of the final product. Traditionally,

offline measurements via image analysis or laser diffraction methods are used to analyze the crystal product. It is often beneficial to extract crystal samples intermittently during the crystallization process to better understand the complex dynamics in batch and continuous crystallization. However, frequent invasive product sampling can lead to undesired disturbances in the process dynamics. Non-invasive solutions to address data scarcity in particle size is to obtain estimated crystal sizes with in-situ measurement tools such as chord length measurements or in-situ imaging (Acevedo et al., 2021 and Larsen et al., 2006). These particle images of various solid density, opaqueness, size, and morphology are often analyzed with generic algorithms and/or complex mathematical models which can have discrepancies with offline measurement data or lead to long computational time.

To obtain a fast and accurate in-situ image analysis measurement of particle size for a high aspect ratio nature crystallization system, a systematic off-line image analysis calibration methodology was performed to model offline Malvern Morphologi image analysis results. Grey-scale image analysis algorithms via ImageJ were calibrated to extract size distribution data from in-situ images of varying size and solid density. Different sets of algorithms with varying parameters in the image analysis methods were tested and optimized by minimizing the size distribution error between the model and offline image analysis measurement. Trends observed in the calibrated parameters were then fitted to continuous functions varying in solid density to be able to adapt to changes in solid loading. The algorithms were then validated with a different particle data set with known solid loading for algorithm predictiveness. Lastly, the usability of the algorithms was tested on a dynamic data set to gauge the processing time and predict the size distribution with varying solid loading throughout the crystallization process due to dissolution, nucleation, and growth.

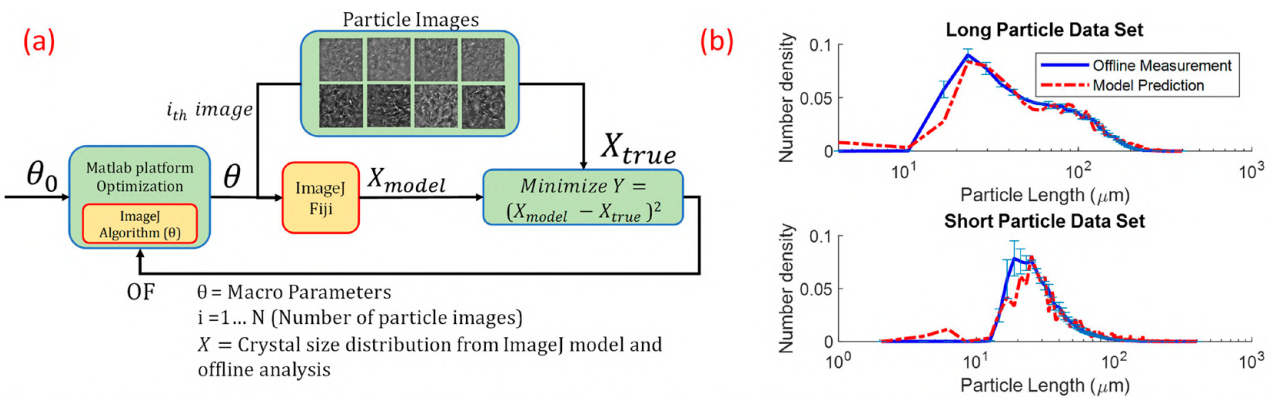


Figure 1. (a) ImageJ algorithm optimization illustrated as training loop to minimize error between model predictions and offline measurement, **(b)** Nominal distribution (red dashed) fits between two different data sets at 0.1% solid density (blue solid) compared to offline measured distribution (blue solid).

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DEM parameter calibration based on flow data from a split- bottom shear cell

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Keywords | Discrete element method, split-bottom shear cell, calibration, deformable particles

We have studied the rate-independent flow of submersed and drained hydrogel particles in a split-bottom-shear cell geometry using the discrete element method (DEM). The main goal was to calibrate DEM input parameters based on experimental data from such a flow geometry.

Although the DEM is widely used to study particulate systems, selecting the DEM input parameters is still challenging. Directly measured particle properties often do not yield the actual bulk and mechanical behaviour (Coetzee 2017) because it is either not simple to measure

certain microscopic level properties, or the simplifications made in the interaction models and shape of the particles are too unrealistic. Therefore, conventional calibration methods based on the bulk behaviour of particles - such as static and dynamic angle of repose, ring shear tests, etc. - have been compared with imaging experimental data in our present study. Specifically, our aim was to examine how the employment of MRI (Magnetic Resonance Imaging) and PIV (particle image velocimetry) data can reduce the ambiguity in the calibrated parameters, in contrast to conventional calibration methods. Also, we have considered the deformability of soft particles in a high-stress variant of the split-bottom shear cell by using an advanced contact force closure recently developed by us (Ghods et al. 2022).

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Wet milling of mefenamic acid for seed generation: model- driven size reduction for maximizing yield

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Keywords | wet milling, breakage, growth, design space exploration

There is an increasing interest in the application of continuous processing technologies using continuous crystallization in pharmaceutical manufacturing to control crystal properties and deliver consistent particulate products (Bosetti & Mazzotti, 2020; Brown et al., 2018). Whilst widely used in the production of large volume commodity chemicals, there is increasing number of industrial examples and support from regulatory authorities including the FDA in demonstrating the potential value of continuous crystallization in the supply of medicines and other high value chemicals. Recent integration of wet media milling technologies, particularly rotor-stator devices, have been shown to avoid common issues encountered with traditional dry milling units. These include introduction of crystal lattice disorder, undesired polymorphic and amorphous transformations, reduced exposure to high potency APIs, broad bimodal particle size distributions, loss of yield and process control as well as increased energy cost (Agimelen et al., 2018; Ahmed et al., 2019; Szilagyi & Nagy, 2019).

The aim of this work is to explore the region of attainable particle sizes based on the performance of a wet milling model. In detail, this work focuses on the development of a mechanistic model for wet milling, with a view to parametrize the breakage kinetics, optimize the milling performance and explore the effects on downstream processes such as crystallization. Lab-scale experiments with varying rotor frequency were conducted with a rotor-stator wet mill to collect data that would allow for the estimation of breakage kinetics. Results from model validation showed that with the dataset used for modelling, the model was able to effectively estimate the crystallization and breakage performance, as shown below:

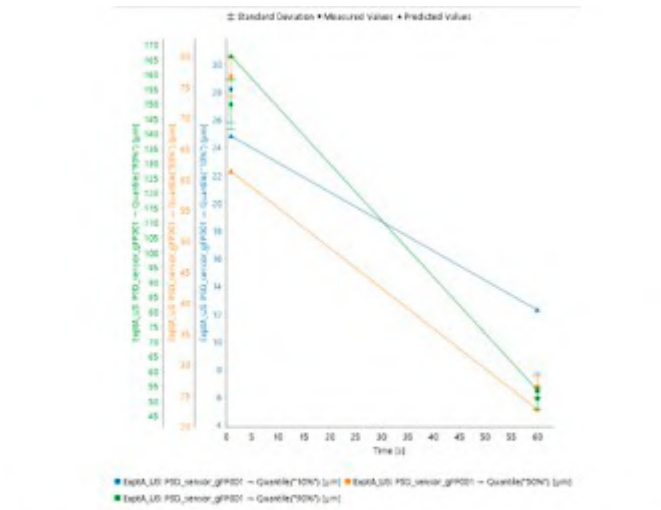


Figure 1: Predicted vs Experimental fit for a high-shear wet milling process

A controlled and robust wet mill model used for seed generation would aid in continuous crystallization processes by allowing for better control in final crystal quality attributes like particle size distribution. The effect of seed sizes on a continuous crystallization process can therefore allow for a wider study of the design space and a better understanding of the optimal process conditions.

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Improving the accuracy of coarse-graining bidisperse particle flows by tuning friction parameters

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Keywords | Discrete element method, Coarse graining, chute flow, rolling friction, sliding friction, segregation.

The discrete element method (DEM) is a powerful tool for simulating granular flows. The computational cost of this method is one of the major challenges when a large number of particles is involved. In this study, the coarse-graining (CG) model, as a computationally cost-effective

solution, is employed to simulate particles in an inclined chute (Queteschiner, Lichtenegger et al., 2018). The particles form a bidisperse mixture of glass spheres with diameters of 1 mm and 5 mm as well as mass fractions of 0.33 and 0.67, respectively. By coarse-graining the smaller particles by the factor α, the mixture changes to α mm and 5 mm particles with the same mass fractions. However, such a strategy breaks the similarity constraint usually implied by CG models and thus, the CG model alone is not accurate enough especially with respect to segregation. The accuracy of the CG model is assessed by calculating the mass flow rate, velocity profile, flow height and volume fraction. As a procedure, by comparing these values with the reference fine-grain model and adjusting parameters like sliding and rolling friction the optimized parameters can be determined. Mass flow rate can be matched for fine and CG models by changing the sliding friction coefficient (Figure 1). However, to fit the velocity profile (maintaining the mass flow rate) the flow height and velocity should agree simultaneously. This can be achieved by changing the rolling friction coefficient (Figure 2). The volume fraction of CG model particles, especially in near-wall regions, does not represent the same values as the fine-grain model. Furthermore, by introducing the overlap factor β in the contact force model (Kanjilal and Schneiderbauer, 2021), corrected segregation patterns can be retained (Figure 3). Finally, by repeating this procedure, a new mixture with a much lower number of coarse-grained particles will be defined. This mixture can represent the behavior of the fine-grain model with a considerably lower computational cost and reasonable accuracy.

Pressure-dependent tuning of a semi-empirical breakage kernel for modeling the micronization process of pharmaceutical powders

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(1) Scuola Universitaria Professionale della Svizzera Italiana, (2) Munit SA, (3) Jetpharma SA, (4) Politecnico di Torino

Keywords | Euler-Euler; Spiral Jet Milling; CFD; 1D model.

The micronization of pharmaceutical powders via spiral jet milling is an industrial process that uses high-energy gas to realize collisions between particles, which eventually break down into smaller size components.

The technology still lacks of a solid theoretical ground able to properly describe the physics behind the process and the determination of substance-specific operating conditions is usually based on expensive experimental campaigns driven by the technicians' experience. This, together with the impossibility of experimentally characterize the system to avoid the multiphase flow field corruption and the difficulties in running fully coupled CFD simulations, motivated the construction of a compartimentalized quasi-3D model based upon the finite volumes formalism to study the process in a simplified manner at an affordable computational cost. A simulation strategy based upon the dilute flow assumption that uses the detailed 3D gas velocity profiles computed on real-scale jet mills is developed, and algebraic expressions valid for pneumatic transport are used to calculate the particles velocity. A semi-empirical breakage kernel tuned over micronization experiments is exploited to model the brittle and semi-brittle breakage of pharmaceutical powders as function of reactor hold-up and grinding pressure.

In this work we focus on the definition of a pressure-dependent procedure for fitting the breakage model parameters over experiments carried out at different grinding pressures and to improve the model prediction capabilities for brittle substances with large inlet particle sizes.

Three different pressure ranges have been studied to characterize the particles fragmentation at medium and high specific energies and simple interpolating laws have been implemented into the code.

Simulation results are validated comparing the model predictions obtained for two substances with D10, D50 and D90 size values coming from Design of Experiments iso-surfaces.

It is shown that simulation predictions are remarkably improved if a specific-pressure parameters set is used, especially when treating fragile materials with wide inlet distributions. Model results are better if high-specific energy processes are considered, *i.e.*, high pressure and low solid feed-rate (dilute flow conditions) and small particles are processed (low Stokes number).

Soluble gas absorption by slurry droplets as a way to intensify the granulation process during spray drying

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Keywords | Slurry droplet, gas absorption, heat transfer, mass transfer, spray drying

In the present study, we developed a transient model for drying a moving single slurry droplet in multicomponent gaseous mixture containing a soluble gas. The comprehensive model accounts for the effects of the soluble gas absorption/desorption, filtration, and compressibility of the gas-vapor mixture inside the porous crust on the intensity of the drying of the slurry droplet. The model is described by a system of transient conjugate nonlinear energy and mass conservation equations using an anelastic approximation. Darcy's law is used to describe the filtration of the vapor-gas mixture inside the porous shell. It is shown that the presence of the active gas increases the evaporation rate during the entire drying process (see David Pour et al. 2020, 2022) and Fig. 1. As shown by numerical calculations, in a gas mixture containing air and ammonia with an ammonia mass fraction of 0.2, at a temperature of 293 K and humidity of 50%, the drying time of silica-aqueous slurry droplets with a radius of 250 m is approximately 35% shorter than that in a gas mixture not containing an active gas. We also found that at the second stage of slurry droplet drying, the desorption of the dissolved gas from the wet core decreases the temperature of the porous shell and reduces the mechanical stresses inside it that prevents the destruction of porous granules. The computational results obtained using the developed model are validated based on a good agreement with the available experimental data. Accordingly, the suggested model can be considered as a basis for alternative drying technologies.

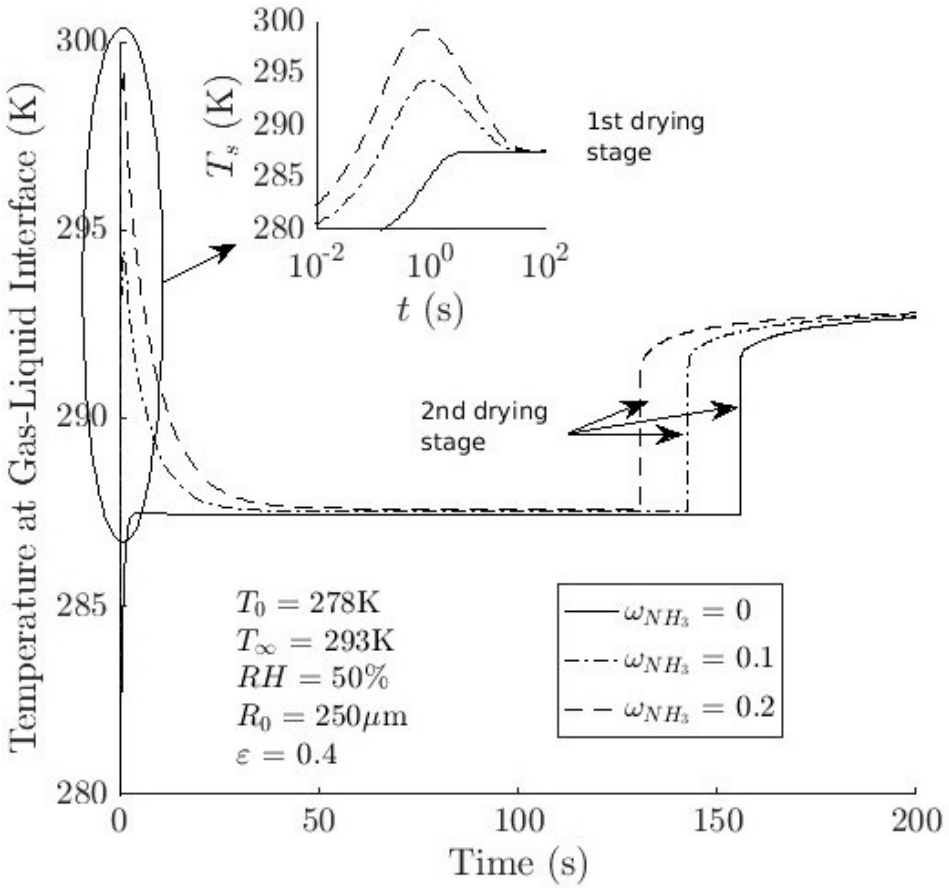


Fig. 1. Temporal evolution of the temperature of the gas-liquid interface during first and second drying stages for three different mass fractions of ammonia in the surrounding gas.

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Coarse grain DEM-CFD simulations for the analysis of tribocharging in fluidized bed

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Keywords | DEM - CFD; Coarse Grain; Tribocharging; Fluidized bed; Riser; hydrodynamics;

The fluidization of electrically insulating particles often leads to charge accumulation on the particles over time, whose level may cause severe issues in their flow and costly plant shutdowns. The speed at which the effect becomes relevant depends on the size of the particles, with the finer ones being affected sooner. Yet, agglomeration problems have been reported also with millimeter-size particles, e.g. polymers [1,2]. The formation of agglomerated structures in localized portion of the fluid bed is still not fully understood, as it results from the complex interplay between the hydrodynamics and the charged particle interactions in those regions. Simulations based on the DEM-CFD approach can help understand the origin and characteristics of the issues affecting fluidized bed operation. To overcome limitations on the feasible size of the system, coarse graining methods have been proposed for contact and drag forces acting on the particles. In this contribution, coarse- grain DEM-CFD simulations are considered in the framework of triboelectric charging phenomena. In particular, scaling relationships are examined in relation to a condenser-like model of the charge exchange process. Representative grain (or parcel) flow properties are set to mimic those of the original particles. After the presentation of the parameter scaling formulas, discussed tests include single collisions tests, shaken systems and riser flow (see Figure 1). The results show that scaling of the charge exchange process accounts only for part of the phenomena and electrostatic interactions require particular attention as well. Possible solutions are proposed and discussed.

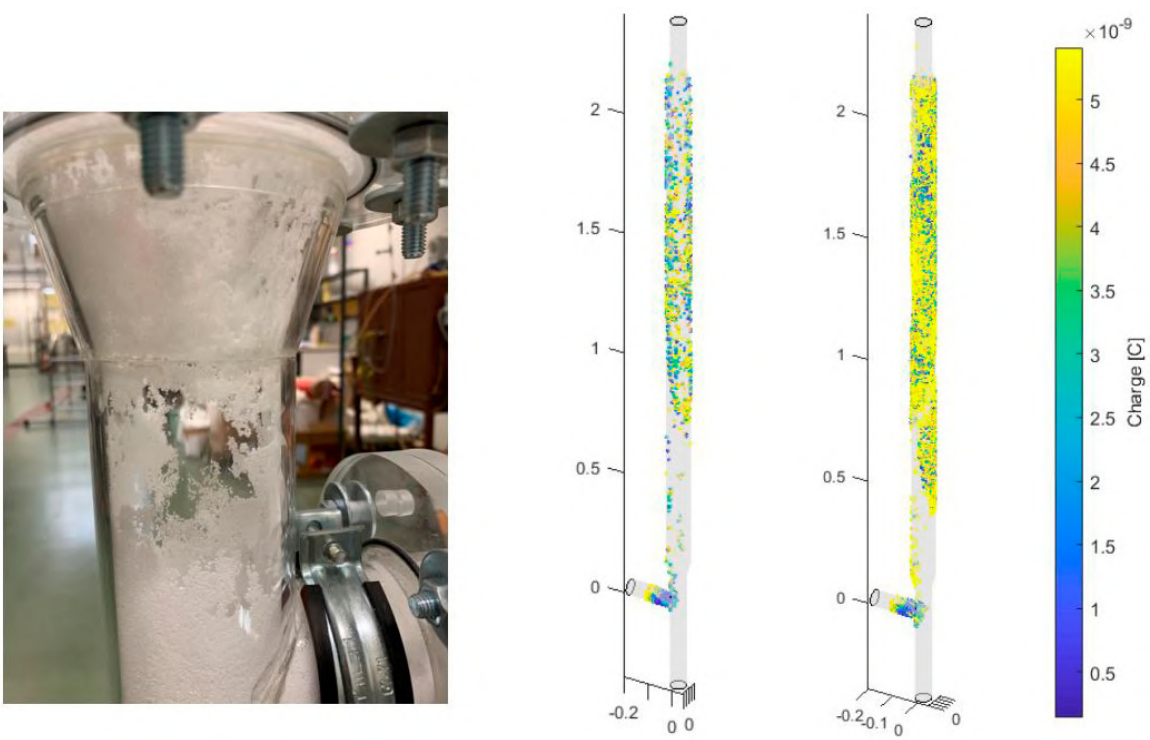


Figure 1. Experimental evidence of charged particles at the base of a riser (a). Simulated position (coordinates in meters) and charge level of fluidized particles in the riser at 1% (b) and 5% (c) inlet solid loading.

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Optimization of a Jaw Crusher Using DEM and MBD Coupling

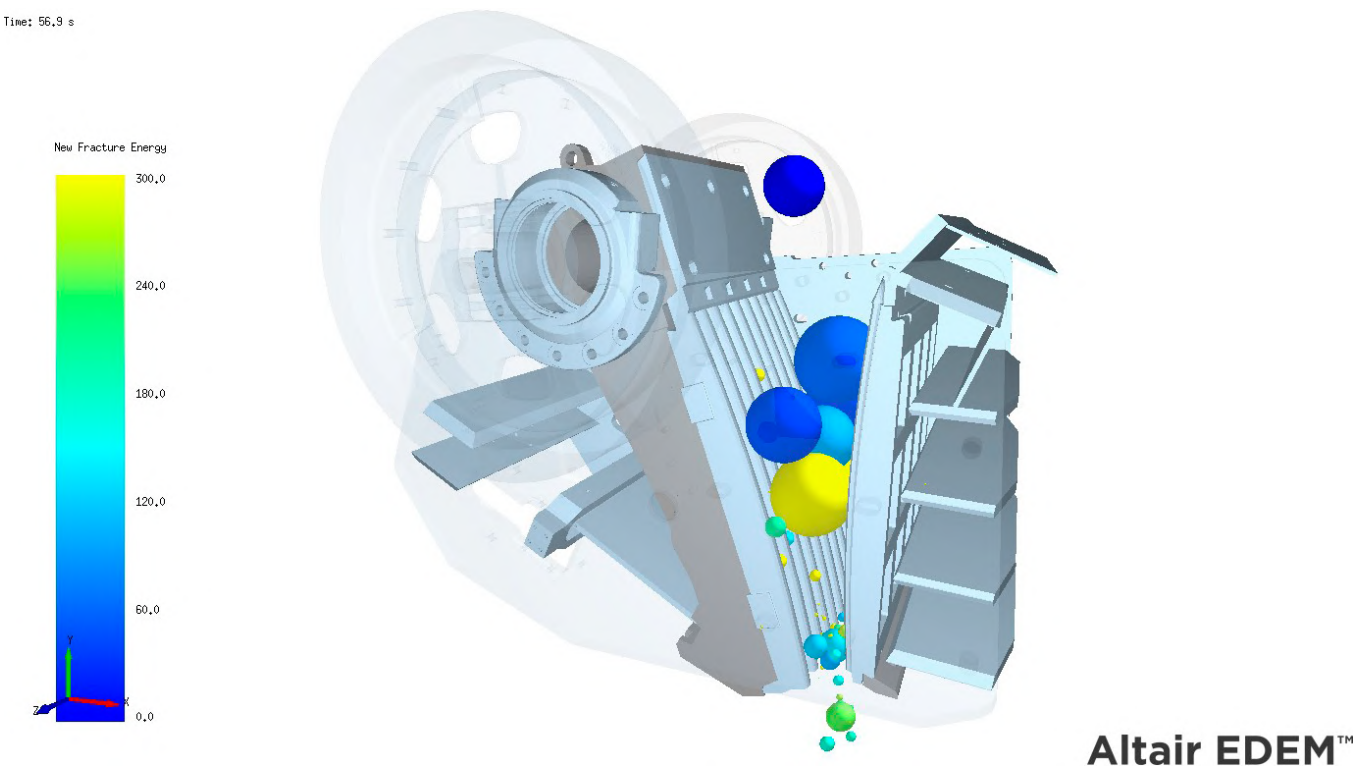
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(1) ASTEC Indstustries, (2) Altair

Keywords | DEM, breakage, simulation, optimization, multi-body dynamics, coupking

Reducing aggregate sizes by crushing is an important step of producing raw materials used in many construction methods. Furthermore, minimizing energy requirements and machine wear while improving crushing performance are key objectives in the design of jaw crushers. The latter use a four-bar mechanism driven to drive the motion of the moving jaw plate that compresses aggregate particles against a stationary jaw surface. The kinematics of the moving jaw are dependent on the position and dimensions of the four-bar mechanism and the shaft speed.

This study focuses on the understanding of the complex kinematics and its impact on the material behaviour as well as the optimization of the simulation workflow. The kinematics of the equipment was parameterized by using a set of DEM simulations and a design of experiment conducted using multi-body dynamics (MBD) to determine the relationship between geometric inputs and the moving jaw kinematics. An optimization routine was used to automate the simulation for a range of kinematics. The material behaviour was modeled using the Tavares Breakage Model (Tavares. L. M. et al., 2002, 2013). This method uses an advanced, stochastic replacement method to reproduce the resulting fragments from particle collisions. The r model captures well the complex material behaviour including both catastrophic particle breakage as well as their weakening due to low stressing events.

Finally, the results of the uncoupled MBD and DEM studies were verified using coupled simulations where geometry kinematics, used in the DEM simulations, were provided by the MBD simulation. Forces from the particles were fed back to the MBD model and were used as inputs to stress calculations performed using finite element analysis (FEA). Coupled DEM and MBD simulations provide engineers with greater confidence in the mechanical design and performance of the crusher permitting improved efficiency and longevity.



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Dynamic flowsheet simulation of an industrial zeolite production process

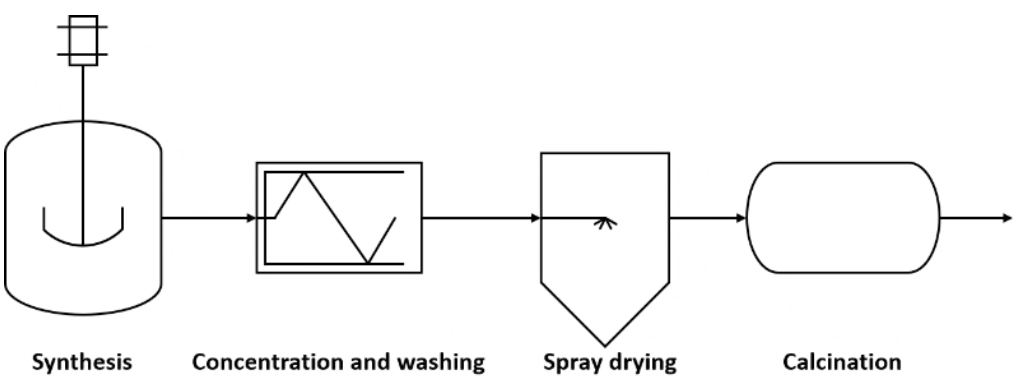
Skorych, Vasyl (1); Buchholz, Moritz (2); Dosta, Maksym (2); Wettich, Helene (3); Gleiss, Marco (3); Haus, Johannes (4); Weis, Dominik (4); Hammerich, Simon (4); Kiedorf, Gregor (4); Asprion, Norbert (4); Nirschl, Hermann (3); Kleine Jäger, Frank (4); Heinrich, Stefan (2)

(1) Hamburg University of Technology, Institute of Solids Process Engineering and Particle Technology, (2) Hamburg University of Technology, Institute of Solid Process Engineering and Particle Technology, (3) Karlsruhe Institute of Technology, Institute of Mechanical Process Engineering and Mechanics, (4) BASF SE

Keywords | Flowsheet simulation; Zeolite production; Data-driven modeling; Synthesis; Solid-liquid separation; Spray drying; Calcination; Multiscale modeling

The production of catalysts like zeolites is a complex multi-stage process. Various material properties and operating parameters significantly affect the outcome of each process substep, and hence determine the properties of the final product. Designing and optimizing such processes is a complex task, which can be greatly facilitated with the help of numerical simulation techniques.

We propose a system, based on the Dyssol modeling framework (Skorych et al., 2020), for dynamic flowsheet simulation of a zeolite production chain consisting of 4 stages: precipitation, concentration and washing, formation of droplets and drying, burning of organic residues. For synthesis in the reactor, a multi-stage strategy was used, comprising microscale DEM simulations, generation of the data-driven surrogate models of the agglomeration kernel, and formulation of the macroscale population balance model (PBM). This allows to correctly take into account various interdependent operating parameters of the precipitation process. The concentration stage consists of several multicompartment decanter centrifuges (Gleiss et al., 2017) alternating with water mixers. They can predict the sedimentation of solids from the slurry and compaction of the resulting cake. Drying is described by a co-current spray dryer model (Buchholz et al.) developed as a 2-dimensional PBM, refined with CFD simulations of droplet motion. It takes into account the kinetics and solidification of droplets, considering mass and heat transfer between particles, gas, and walls. Calcination is done in rotary kilns developed as a multi- compartment model. It describes the gas-solid reaction in countercurrent solids and gas flows, taking into account heat and mass transfer between particles, gas, and kiln walls. The information on the kiln particle dynamics was extracted from coupled CFD-DEM simulations.



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Computational Modeling of Drying in a Fluidized Bed Dryer using Coupled CFD-DEM approach

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(1) University of Connecticut, (2) Takeda, (3) Northeastern University

Keywords | Computational Fluid Dynamics, Discrete Element Method, Multiphase-Multicomponent Flow, Fluidized Bed Dryer, CFDEM

Introduction:

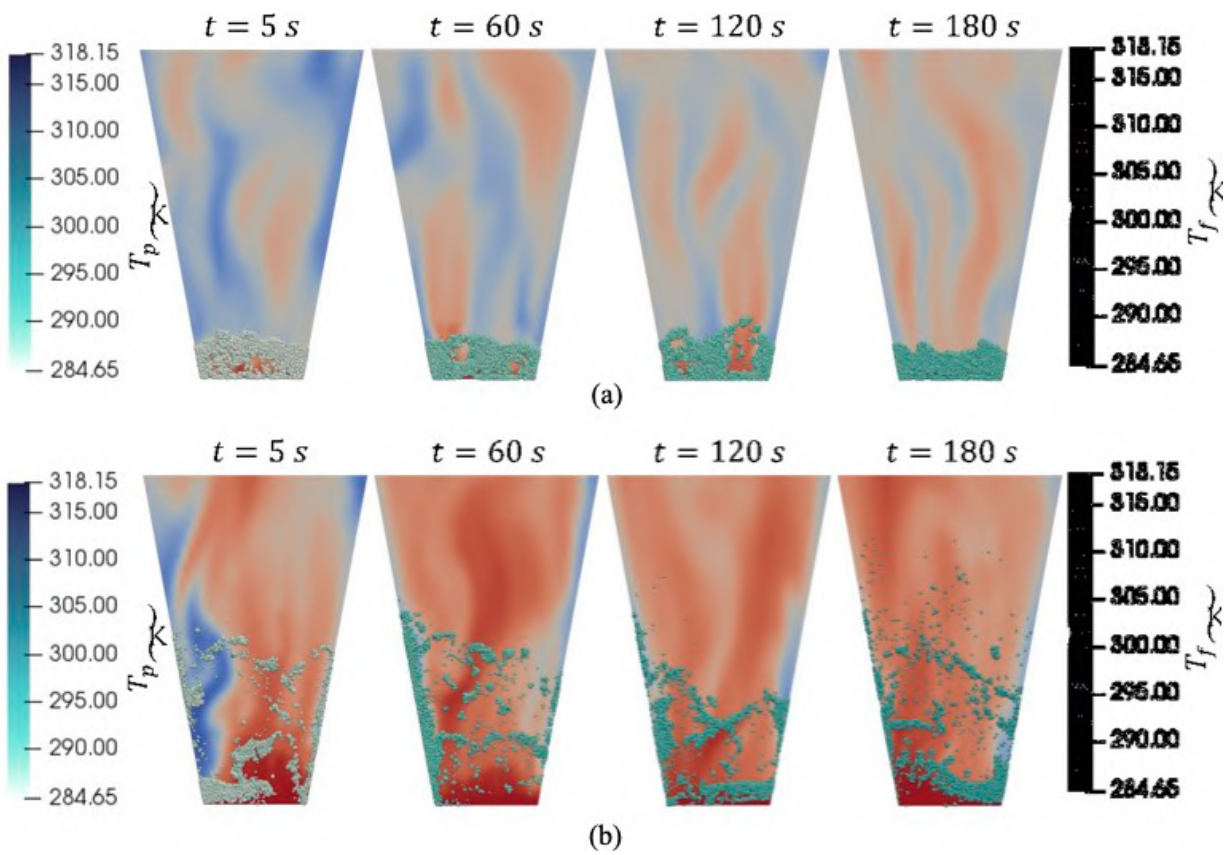
Fluidized bed drying is a topic of interest for scientists and engineers because of its complicated physics and its presence in a wide range of industrial processes e.g. pharmaceutical product manufacturing, food processing, wood processing, flue gas cleaning, the roasting of sulfide ores, drying of coal, catalyst industry and many more. Here, we propose a model based on coupled Computational Fluid Dynamics (CFD) and Discrete Element Method (DEM) approach to simulate the drying of granules in fluidized bed dryer associated to the drug manufacturing process. The drying process of wet granular particles in a fluidized bed dryer involves momentum, heat and mass transfer between the particles and the drying medium which is inherently a multiphase-multicomponent flow problem.

Methods:

The model was implemented using an opensource CFDEM® coupling where the flow of fluid and solid phase were solved by OpenFOAM® and LIGGGHTS® codes respectively. The information between the fluid phase and particles were exchanged at certain time intervals. A reaction engineering based approach was used to modify the CFDEM code to model the evaporation of water from the wet granules. The cohesion between the wet granules due to the formation of liquid bridge between the wet granules was considered in the model.

Results:

Our model was capable of capturing the heat, momentum and mass transfer between the lactose monohydrate particles and the drying medium. Numerical simulations were performed to predict the change of particle moisture content and temperature with time during the fluidization process. The variation of average particle temperature and average moisture content with time agreed well with the experimental results obtained from the literature. Agglomeration of wet particles was observed during the drying process due to the cohesion force generated by the liquid bridge forces. The agglomeration of particles was not present after the particles became dry as expected. It was also observed that the temperature of the dry particles increased at a much higher than the wet particles since the latent heat of evaporation for the water in the wet particles was supplied by the particle itself. This was also observed in the experiments.



How we teach Discrete Element Method in Engineering

Schott, Dingena (1); Mohajeri, Javad (1); Fransen, Marc (1); Yan, Yunpeng (1); Roeplal, Raïsa (1); Hadi, Ahmed (1); Chen, Qianyi (1)
(1) Delft

Keywords | DEM; modelling; education; constructive alignment;

We educate our students to contribute to sustainable solutions for existing and future complex issues in the transport domain all over the world, either in industrial positions or as academics. Given the increasing popularity of Discrete Element Method (DEM) in science and industry, in this paper, we outline how we teach DEM in the Multi-Machine Engineering track in the two-year Master programme Mechanical Engineering.

The main learning objectives are to choose and implement a simplified but appropriate representation of a granular system, and subsequently test and evaluate the influence of different simulation parameters on the performance of the system (Schott, 2021). To reach these objectives, the students practice their research skills by defining research questions, find relevant papers in the literature, select the appropriate contact model and its parameters, define performance criteria, use Design of Experiments techniques and perform stability and reliability assessments of their model.

We use constructive alignment in the design and assessment of this course. The principle of constructive alignment (Biggs & Tang, 2011) states that the assessment and learning activities in education must be focused on the learning objectives. We adopt a flipped classroom approach in which on the one hand we deliver a theoretical basis of DEM, on the other hand students execute assignments supported by the theory taught during lectures. These assignments are defined on topics closely related to ongoing PhD research in our GranCHaM lab (Granular Characterisation, Handling, and Modelling group).

This provides the opportunity for PhD candidates to gain experience in teaching, engaging students in their specific research, and to interest them in working on a MSc thesis or research assignment in their Master programme. The students receive formative feedback on their learning tasks by presenting their approach and discussing those with their mentor. The summative feedback reflects the degree learning objectives have been achieved by using grading rubrics to assess their midterm and final reports and presentations.

We will present the course outline enriched with examples and results of assignments students executed over the past years that show our approach to teaching DEM in an engineering context.

Numerical modelling of leading-edge erosion of wind turbine blades due to particles impact

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(1) Institute for Materials and Processes School of Engineering, University of Edinburgh

Keywords | peri-dynamics, discrete element method, erosion, granular contact model, wind turbine blades

The expanding advancements in wind turbine technology, combined with an uncertain operating conditions, present significant challenges relating to erosion issues on the leading edge of the blades. This paper provides a novel methodology for particle impact modelling that combines peridynamics (PD) with the discrete element method (DEM). The impactor particle is described using DEM, whereas the wind turbine blade is represented by bond-based PD particles. The Hertzian contact law governs the force and displacement relations in the normal and tangential directions. By executing normal and oblique hits on the target material without allowing failure, the present contact algorithm is validated for contact parameters such as maximum penetration, rebound velocity, contact time, and force of response. Once the proposed technique for the contact parameters during the impact event is verified, failure analysis is performed by simulating the motion of a rigid sand particle and its impact on the surface to predict the damage patterns along with subsequent material removal. After the analysis of the contact damage results and literature findings, it is observed that the present results correlates well with corresponding FEM and experimental results.

Validation of MP-PIC simulation of propane combustion in a fluidised bed reactor using Positron Emission Particle Tracking and fast X-ray Radiography

Adio, Niklas (1); Windows-Yule, Kit (1); Thorpe, Rex (2); Seville, Jonathan (1)

(1) School of Chemical Engineering - University of Birmingham, (2) Department of Chemical and Process Engineering - University of Surrey

The plastic waste crisis is a great challenge faced by many industries. One of the main solutions is chemical recycling, as it results in plastic waste being reverted to feedstock. A commercially viable option for this are fluidized beds. It is crucial to ensure that their chemical kinetics and hydrodynamics are understood, as otherwise major losses of efficiency occur.

In this work, the combustion of propane in a fluidized bed reactor was simulated using barracuda, as a way of taking steps toward simulating a full chemical plastic recycling system. Barracuda is a commercial MP-PIC software, which enables CFD calculations to incorporate particle interactions. The hydrodynamics of the model were validated via the comparison between key properties including circulation rates, velocity distributions, flow patterns and bubble size distributions acquired using Positron Emission Particle Tracking , as well as empirical data such as predicted UMF (Windows-Yule et al., 2022)(Windows-Yule et al., 2020).To then validate the system with included chemical kinetics, the average volume of bubbles and concentration of uncreated propane, as factors of height above the distributor were compared to experimental data in (Dennis, Hayhurst and Mackley, 1982). Furthermore, the model was compared to data from an Aspen+ simulation to evaluate which of the techniques is more viable and whether the increase of complexity and computational cost is justifiable (Mahooda, Thorpe and Bhattacharyab, 2022). This study represents the potential barracuda has for simulating more complex larger systems, as will be done in latter studies.

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CFD modeling for Industrial FCC riser feed injection with extension to other vapor/liquid/solid applications

Blaser, Peter (1)
(1) CPFD Software

Keywords | fluid catalytic cracking, riser, ebullated bed, simulation, computational fluid dynamics

Computational fluid dynamics (CFD) modeling has been used to simulate FCC risers since the 2000s using various commercial CFD tools including Barracuda Virtual Reactor.

Historically, for CFD modeling with reactions, the hydrocarbon feed to the riser is assumed to be instantly vaporized, or very simple approximations are made to close the mass balance between phases. However, the liquid injection, liquid penetration, spray angle, droplet size distribution, liquid-solid contact dynamics, vaporization and gas-phase expansion are some of the important parameters that can only be captured if the feed is injected as liquid. Therefore, liquid feed injection, as opposed to injecting the feed in gas phase, is central to typical phenomena of interest for risers.

In this work, we have used Virtual Reactor to simulate an industrial scale FCC riser for a US Gulf Coast refinery. The feed is injected as discrete liquid droplets. Part of the liquid feed directly vaporizes into the gas phase while some of the liquid forms a film on the catalyst particles as a result of contacts between droplets and particles. This film transfers to other particles and vaporizes into the gas-phase using the heat from both the gas and the particles.

For this application, the liquid feed injection is characterized by features such as droplet size distribution, spray angle, composition and momentum flux distribution. Liquid penetration, liquid film transfer from droplets to catalyst particles and vaporization are captured. Overall vaporization times obtained from the model agree with commonly known commercial riser design criteria. We also demonstrate that liquid feeds, consisting of multiple components with different boiling points, show vaporization profiles agreeing with expectations for multi-component hydrocarbon feed vaporization.

Extension to more general vapor/liquid/solids applications, including ebullated beds is discussed. In cases where the carrier fluid is a liquid, the model is used to capture gas bubble and solid particle dynamics, multi-component vaporization, gas absorption, and multiphase reaction chemistry. Results from a full scale ebullated bed hydrocracker are shown, with comparisons made to available experimental data.

CFD Modelling of Closed-Couple Gas Atomisation Process

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(1) University of Leeds

Keywords | Gas Atomisation, Discrete-Phase Model (DPM), User-Defined Function (UDF), Aspiration pressure, Pulsation Mechanism.

The Gas Atomisation process stands in the forefront of producing metal powders with a high degree of sphericity and superior material property (Dunkley, 2019). However, the process is chaotic, and extremely rapid, making it really hard to understand the background physics of the process even when studied using high-speed camera. Due to this chaotic nature, the particle size and the spread in the particle size distribution increases to a greater extent, making the process highly inefficient. Consequently, the yield is less than 35% and the rest of the powder is sent to re-melt (Chang and Zhao, 2013). Hence a huge amount of energy and capital is lost in the process, and in addition to this, since the process involves compressed inert gas, a much bigger share of energy is lost in its recompression.

In this research, CFD analysis is carried out to understand the instabilities present between the deep sub-ambient pressure formed in front of the melt nozzle known as aspiration pressure and the melt flow rate. Rapid variations in melt flow rate caused due to the change in aspiration pressure affects the particle size and particle size distribution. A two-phase flow is modelled using coupled Euler-Lagrange framework. The primary phase is Argon, modelled as a compressible gas, and the secondary phase is modelled as inert, constant diameter particles depicting the melt. The particles' mass flow rate is coupled to the aspiration pressure that is obtained in front of the melt nozzle tip using User-Defined Functions (UDFs). This research confirms the presence of a pulsating flow field as postulated by Ting (2003),exhibiting repeated formation and disruption of a Mach disk causing fluctuation in aspiration pressure, which explains the rapid variations in melt flow rate and the chaotic nature of the process.

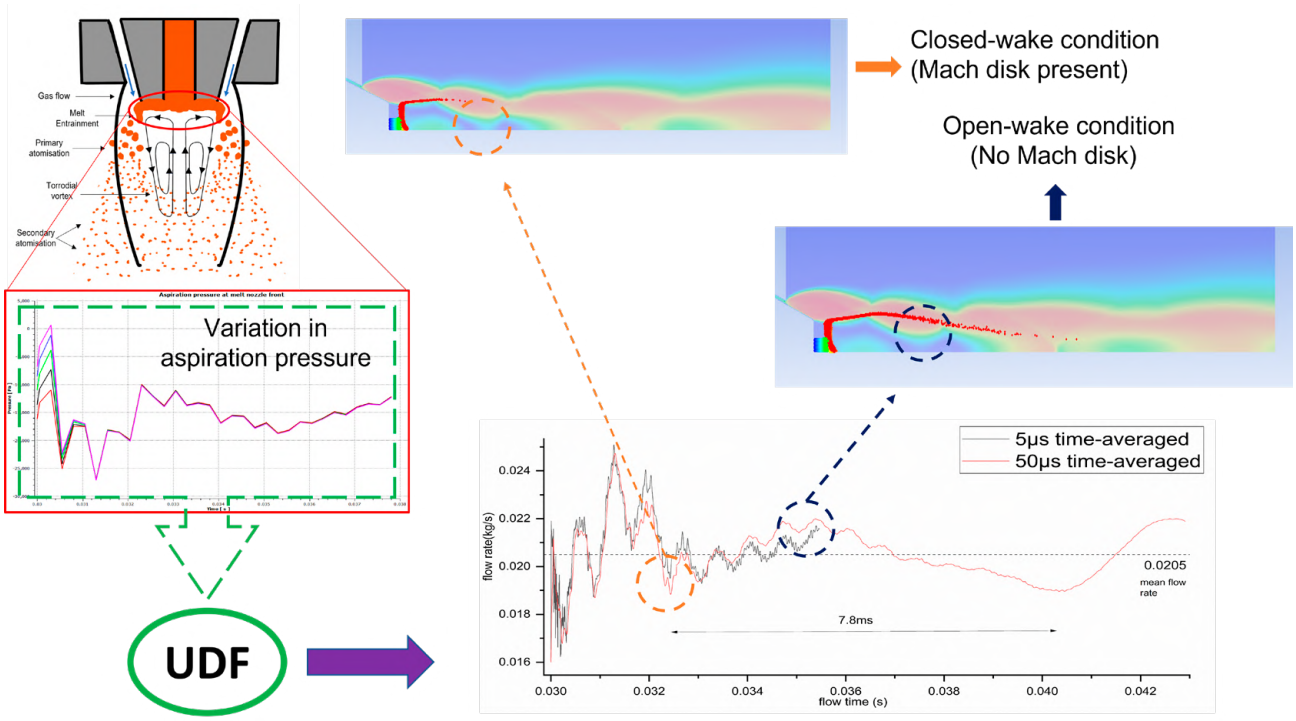
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Graphical Abstract



The effect of particle adhesion on tablet uniformity during the die filling in a rotary tablet press

Alizadeh Behjani, Mohammadreza (1); Zheng, Chao (1); De Souter, Lisa (2); Nitert, Bernardus Joseph (3); De Beer, Thomas (2); Wu, Chuan-Yu (1)

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Keywords | Die filling, DEM, adhesion, pharmaceutical powder, tableting

Die filling is an important stage during the tableting process in the pharmaceutical industry, where most tablets are produced using rotary tablet presses. The die filling performance and powder flow within the rotary tablet presses are intertwined. Powder flow behaviour in a feed frame of a rotary tablet press depends on many parameters, including powder attributes, operating conditions, and feeder type. Assessing the impact of these parameters on the uniformity of the tablets has been a matter of interest for many years; nevertheless, there is still a lack of a sound understanding of the relation between the aforementioned parameters and the quality of tablets.

This study investigates the effect of particle adhesion on die filling uniformity in a linear die filling system fitted with a two- chamber forced feeder (feed frame) using the Discrete Element Method (DEM). Microcrystalline (MCC) VIVAPUR® Spheres 350 powder and its measured particle size distribution (PSD) are utilised as the model powder. To obtain reliable DEM input parameters, a calibration study is performed using simple experimental and numerical tests of a hopper discharge process. For simulating the die filling process, Rocky 4.5 DEM software is used, where the Hertz-Mindlin and JKR contact models are applied to describe the particles' collisions and adhesion during the process, respectively. A validation study is also performed using the actual rotary tablet press and the results show a good agreement between the DEM and experimental tests.

DEM simulations suggest that for free-flowing powder, size-induced segregation occurs within the feed frame due to the wide size distribution of particles. The segregation is also reflected in the deviation of the size distribution of the modelled particles in the die from the original PSD. However, it is observed that particles adhesion diminishes the particles' tendency to segregate within the feed frame and in the dies. Nevertheless, increasing the adhesion leads to a lower die filling ratio and higher variability of the tablet weight, which are both undesirable. DEM modelling can have a meaningful contribution to the optimisation of the operating conditions to reduce the die weight variability and achieve the highest die filling ratio.

Experimental study of red volcanic sand particles in a directly irradiated fluidized bed for CSP applications

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Keywords | CSP applications, thermal radiation, fluidized bed, thermal energy storage

The high mixing rates and elevated heat transfer coefficients in bubbling fluidized beds, makes this particle technology the proper one to distribute a concentrated solar energy on the top of the bed in the whole mass of particles (Almendros-Ibáñez et al, 2019). The use of solid particles, instead of typical molten salts in CSP applications, permits to increase the maximum operating temperature, which nowadays is limited at 565 ºC due to corrosion and degradation processes. Thus, there is a potential interest in studding different materials suitable for this application (Diaz-Heras et al., 2021).

The present work presents the characterization and the experimental results of a novel material: red volcanic sand. This material has not been previously tested for CSP applications. First, a complete morphological analysis of the particles was carried out, including the particle size distribution and circularity. Second, the particle was tested in a lab-scale fluidized bed with direct irradiation (4 kWe). The particles were fluidized and heated during 1.5 h, which was tested enough for reaching steady state conditions, and cooled with atmospheric air during 1.0 h. This cycle was repeated 10 times, and for three excess gas velocities: 1.5-, 2.0- and 2.5-times minimum fluidization velocity.

The objective of the cycles was to test possible changes in the morphology of the particles, which were also characterized after the cycles, and in the thermal energy storage capacity of the material. No relevant particle deterioration was observed and neither reduction in the thermal energy storage capacity. The results were compared with previous results of the authors with other commercial materials (Diaz-Heras et al., 2021).

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DEM study of a vibrational powder transport system

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Keywords | Pharmaceutical Manufacturing, Solids Flow, Handling and Processing, Equipment, Vibration

Vibrational transport systems are used in the pharmaceutical industry to transport the powder from one unit operation to the next downstream one. The important parameters used to control the vibrational powder transport are the amplitude and the frequency. Challenges may arise when transporting powders that have poor flowability, high cohesivity and adhesion levels.

For the vibration system used in this work the powder is introduced from above in the hopper. The vibration source is located below the transport tube, which has a diameter reduction in one place, and the whole system oscillates horizontally. The difficulties in this system include particle agglomeration, system clogging and flow blockage in the hopper and transport tube, because the transported material has poor flowability and is highly cohesive.

During experimental trials, it was determined that the operating frequency affects the flowability of the powder. However, to resolve the clogging and flow blocking problems using experiments is challenging.

Therefore, the Discrete Element Method (DEM) was used to model the process. With this approach it is possible to investigate various process conditions and equipment designs quickly. The process was replicated in the DEM model, with the same operating conditions and equipment design. The powder was calibrated prior to the investigation, in order to closely represent the behavior of material used in the experiments. The particle size was scaled in comparison to the original, keeping the distribution width as close to reality as possible.

The DEM results identified an interesting mechanism, i.e., the segregation of the smallest particles in the fill hopper, along the side walls

of the system and just before the tube reduction. The investigation of the particle stress distribution in the system showed a peak of the normal and shear stress at the position of the segregated particles. The fact that these positions overlap led to the conclusion that the system clogging is caused by the small particles agglomerating when exposed to high stress. The investigation of the operating frequency showed that reducing the oscillation frequency disrupts the small particle ensembles, and works in favor of the powder flowability. This was also confirmed by experiments.

Optimization of Fluidized Bed Granulation Processes using Hybrid Modeling Strategy

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(1) Boehringer Ingelheim Pharma GmbH & Co. KG

Keywords | Simulation; Fluidized Bed Granulation; Hybrid Models

Due to the complex phenomena occurring on different time and length scales, the development of the fluidized bed (FB) granulation processes for the new pharmaceutical products is a challenging and time-consuming task. A relatively large number of experiments is often needed to gain comprehensive process understanding, predict the influence of critical process parameters (CPPs) on the critical material attributes (CMAs), and find optimal process conditions.

To improve the development of FB granulation processes and to implement quality-by-design (QbD) principles the hybrid modeling framework has been developed. The central role here plays a tight combination of phenomenological (mechanistic) and data-driven submodels, as well as an efficient integration of the data obtained from the in-line and off-line PAT tools.

The decomposition of the model into the data-driven and mechanistic parts, as well as the identification of appropriate submodels, strongly depends on the type of solved problem. For example, to describe the fluidized bed coating process, where the bed material remains relatively dry during the whole process, the combination of the simplified population balance model with the linear regression model can be used as an effective strategy. For other problems like FB agglomeration more advanced approaches should be applied. Here the thermodynamic model for the description of heat and mass transfer is used to describe loss on drying (LOD), and the data-driven artificial neural network (ANN) is used to predict aggregation kinetics. Finally, their combination enables the prediction of the process behavior over the entire process time.

Overall, the proposed digital framework and developed mechanistic and data-driven submodels allow to derive better process understanding, find optimal process conditions and support development of new processes at different stages of the development lifecycle. Furthermore, the application of the hybrid modeling strategy has a high potential to be used to reduce the number of required experiments and therefore speed-up process development.

FLASH COMMUNICATIONS

Numerical analysis of tensile strength of annular agglomerate during ring compression test

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Keywords | DEM, Diametrical compression,Surface energy, Tensile strength, Agglomerate

Understanding the effect of material properties, interfacial properties and structure of the agglomerate on the tensile strength is critical in many processes. For instance, for manufacturing of pharmaceutical tablets, nuclear graphite, and pellets with dry granulation, the tensile strength of agglomerates is critical in controlling the granulation behaviors. In addition, the tensile strength is determined by the material properties of feed powders, interfacial properties between particles and the process condition, which determine the structure of the specimen (Loreti et al.,2018). This study aims to study the dependence of the tensile strength of pharmaceutical annular tablet on surface energy and inner diameter, for which discrete element modelling (DEM) with a cohesive particle model based on the JKR theory was performed.

Diametrical compression tests with specimens of different inner diameters and surface energies were modelled by DEM, as shown in Figure 1(a). It was found that there is a strong correlation between the tensile strength with the surface energy and inner diameter of specimens. In Figure 1(b), the maximum loads on the compression wall increase with the surface energy and hence lead to the tensile strength grow with the increasing surface energy. In addition, for specimens with the same outer diameter and thickness, the annular tablet with larger inner diameter has a smaller tensile strength than these with smaller inner diameter, as shown in Figure 1(c).

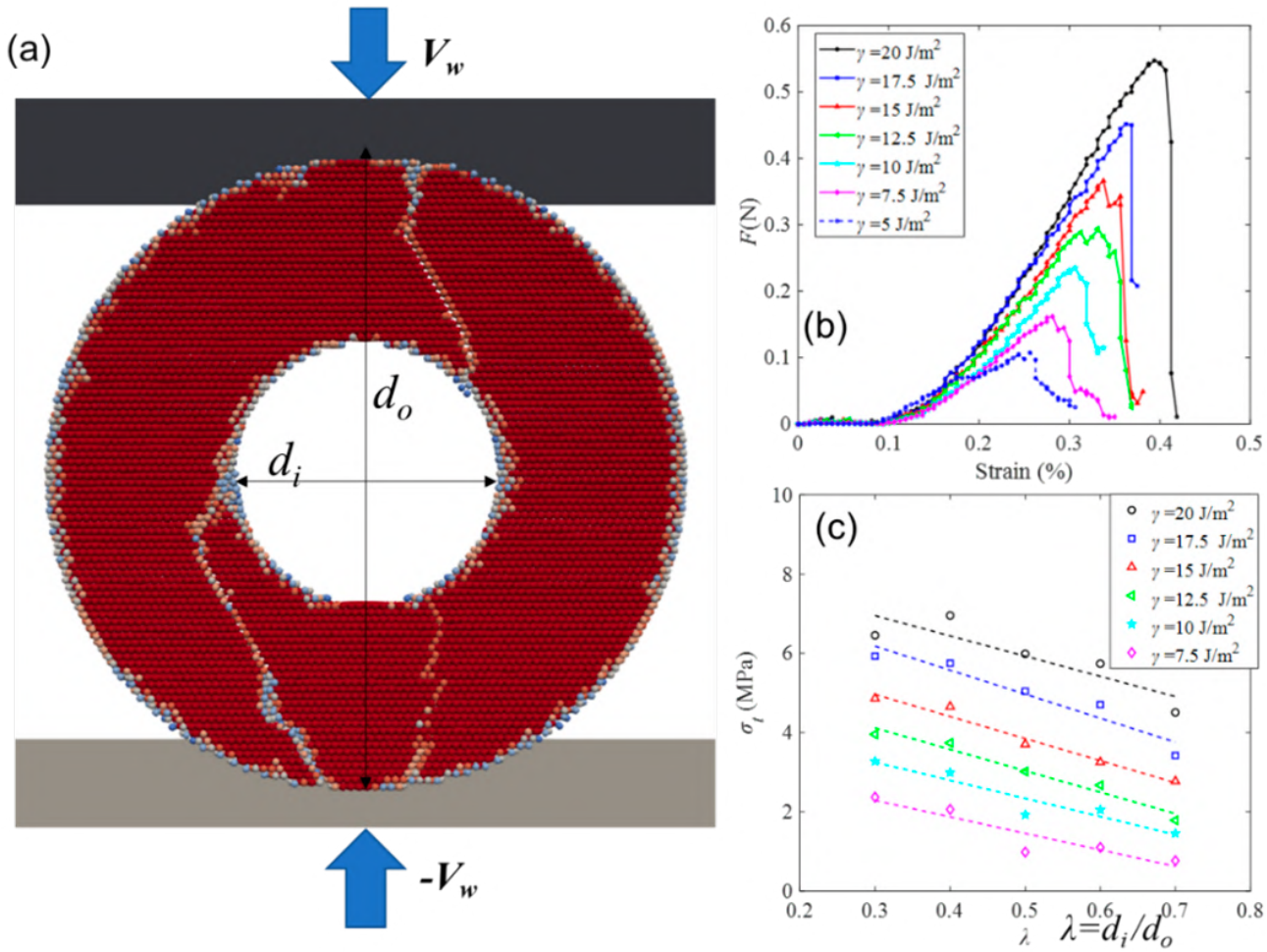


Figure 1. (a) Typical breakage pattern for annular tablet during diametrical compression; (b) Load-strain curve during diametrical compression; (c)The variation of tensile strength with surface energy at different inner diameter.

S. Loreti, C. Wu, Three-dimensional discrete element modelling of three point bending tests : The effect of surface energy on the tensile strength, Powder Technol. 337 (2018) 119–126.

Dry powder modelling through AI as basis for numerical simulation

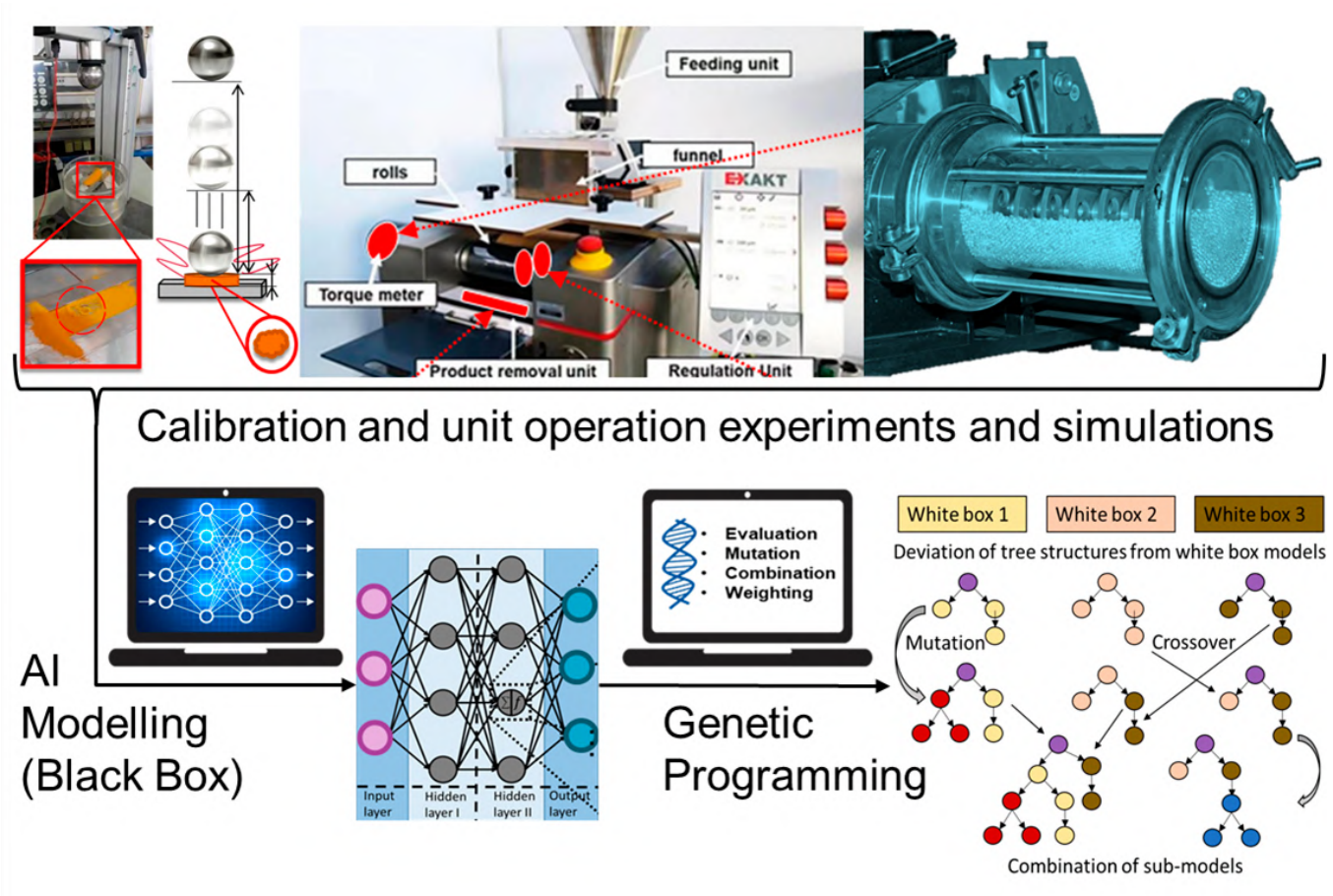
Thon, Christoph (1); Somayeh Hosseinhashemi, Somayeh (1); Böttcher, Ann-Christin (1); Kwade, Arno (1); Schilde, Carsten (1)
(1) Technical University Braunschweig, Institute for Particle Technology

Keywords | Artificial Intelligence, Predictive Modelling, Neural Nets, Genetic Algorithms, Genetic Programming

Dry powder modelling through AI as basis for numerical simulation
Christoph Thon, Somayeh Hosseinhashemi, Ann-Christin-Böttcher, Arno Kwade, Carsten Schilde Institute for Particle Technology (iPAT), TU Braunschweig, Germany

The processing and characterization of dry powders, be it primary particles, aggregates or mixtures, is a laborious and time consuming

endeavour. It encompasses a wide variety of possible experiments and simulations. In addition to unit operations like milling in ball mills or stirred media mills preliminary calibration operations are often required e.g. for the coefficient of restitution, frictions, stressed powder, cohesion, powder breakage function, etc. Artificial Intelligence has the advantage to be able to train generalised models on a training data set, so it can make predictions for novel parameter sets not executed before or used in the training process, see the figure. [1]



With such predictive models of dry powder systems like calibration, unit operations or for powder characterization, systematic accurate predictions can be obtained. The parameter space can occupy a predictive domain, otherwise unobtainable through experimental or numerical means due to computing power, required manual labour, time or cost.

These models have the disadvantage of usually being intransparent black box systems. On the basis of the predicted generalised data sets, genetic programming can be performed as a means of breaking down the neural net models to transparent equation systems. In this presentation, in addition to an overview of the used AI techniques, practical use cases from dry powder technology will be shown such as a predictive model for a milling process and modelling of a breakage function through AI methods.

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DEM simulation of Elasto-Plastic Powder compression Process

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(1) Osaka Prefecture University, (2) Osaka Metropolitan University

Keywords | Powder Compression, All-Solid-State Batteries, Discrete Element Method, Elastoplastic compression behavior, Cohesive powder

All-solid-state batteries are expected as next-generation batteries for electric vehicles because of their high safety and high battery capacity. To improve the performance of all-solid-state batteries, it is essential to increase the density by compression. However, the densification process for battery materials is complicated due to their unique powder properties such as cohesiveness and plastic deformability. In this

study, the elastoplastic compression behavior of cohesive powders was analyzed by using the discrete element method (DEM). EDEM2019.1 was used for the DEM calculations. The particle properties were referred to as the positive electrode NCM (LiNi0.33Co0.33Mn0.33O2), and Young's modulus was halved to reduce the calculation costs. First, the compressive behavior of the cohesive particles by considering only their elastic deformation was investigated. The Hertz-Mindlin with JKR model was used as the contact model. The effects of particle size distribution and cohesiveness were investigated. Bimodal particles were used to study the effect of particle size distribution. The particle size ratio and mixing ratio of large and small particles were varied. The structure of compacts was evaluated with the void fraction and the contact number between particles. DEM simulations indicated that a large cohesive force had a small effect on the void fraction and a large effect on the contact inside the powder bed. Also, the effects of the plastic deformability of the particles were investigated. The Edinburgh Elasto-Plastic Adhesion model, which is an adhesive plastic deformation model, was applied as a contact model. The plastic deformability λp is perfectly elastic at 0 and perfectly plastic at 1. In the case of the elastoplastic mixed powder, increasing the number of plastic particles had a significant effect on the contact state inside the compacts. We hope that our results will be useful for the design of electrode structures.

DEM modelling of triboelectrification and electrostatic dispersion of particles

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Keywords | electrostatic dispersion, electrostatics, frictional electrification, granular temperature

Frictional electrification between particles and surfaces is prevalent in many powder handling processes. In the previous particle contact electrification model, it was assumed that the charge transfer from one surface to another only takes place once two contacted surfaces separate (Pei et al.,2013). If the contact persists, e.g. a particle slides or rolls along a wall, the charge transfer is assumed not occurring and is not considered. These models do not consider frictional charging that does occur in many powders handling processes. Aiming to explore the friction-induced electrostatic charging behaviour, a discrete element method (DEM) is developed for the first time in the current paper, in which a frictional charging model and electrostatic interaction models are implemented.

The charge accumulation on both the particles and the surface in a rotational container is then analysed numerically and experimentally to evaluate the developed DEM, as shown in Fig 1. It is clear that the numerical results are in good agreement with the experimental data, where the average net charge of a single particle gradually increases and achieves an equilibrium state (Hu et al.,2021). It is also shown that both the net charge on the particles and the degree of the particle dispersion are a function of the charging time. Moreover, it is revealed that the friction-induced particle charge enhances particle dispersion, and increases the granular temperature [2] due to the electrostatic interactions.

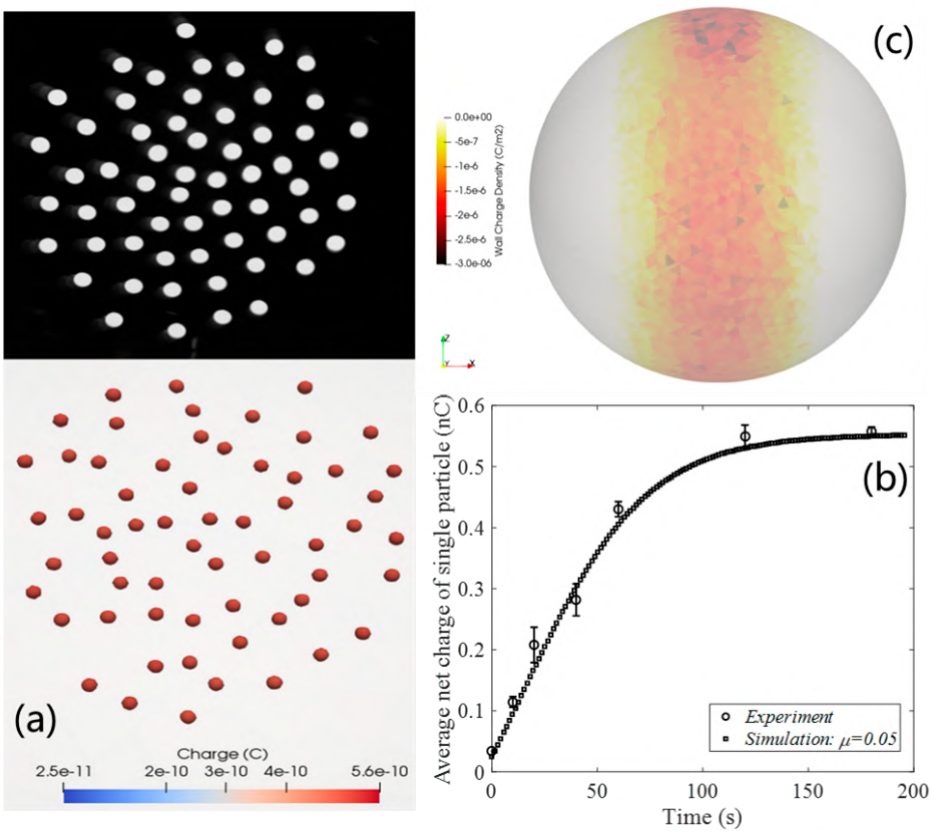


Fig 1. (a)Dispersion of charged particles from experiment and DEM simulations;(b) Evolution of net charges of the particle from experiments and DEM simulation; (c) The charge density distribution on the container wall.

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A discrete element method based digital twin for evaluating tableting process performance

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Tableting is a fundamental unit operation in the pharmaceutical solid oral dose manufacturing process and has a significant influence on tablet quality attributes such as weight, dosage, structural integrity, and dissolution rate. Reliably meeting the associated quality standards requires optimal operation for a given formulation but the effects of operational parameters on the mechanics of the tableting process are still not fully understood and process optimisation is primarily conducted empirically. Consequently, achieving high degree of reliability in tableting operations is challenging and time consuming for new formulations, leading to economic losses. Employing simulation based digital twins to gain insight into the mechanics of the tableting process beyond the one obtainable through experiments alone has significant utility in this context.

In this work we employ a discrete element method based digital twin of the tableting process to evaluate the effect of operational parameters on process performance. Both the die filling stage shown in Figure 1 and subsequent compaction stage are modelled using an experimentally calibrated model for micro-crystalline cellulose. We evaluate the effect of blade speed, die diameter and punch stroke on the material flow rate and distribution in the die, the degree of particle size segregation and propensity for attrition as well as the stress state during

compaction and the resulting density distribution of the compact.

The modelling reveals a complex behaviour characterised by a high degree of non-linearity and significant parameter interaction. This is particularly true for the die filling stage where the observed evolution of the mass flow rate is non-linear both with respect to the operational parameters and the number of blade passes. Additionally, a complex stress-state emerges during the compaction stage where a significant spatial variation of consolidating stress magnitudes and a corresponding variation of intra-tablet density can be observed. Beyond the mechanistic insight that was generated, the work demonstrates a practical methodology for the generation of a discrete element model based digital twin of the tableting process.

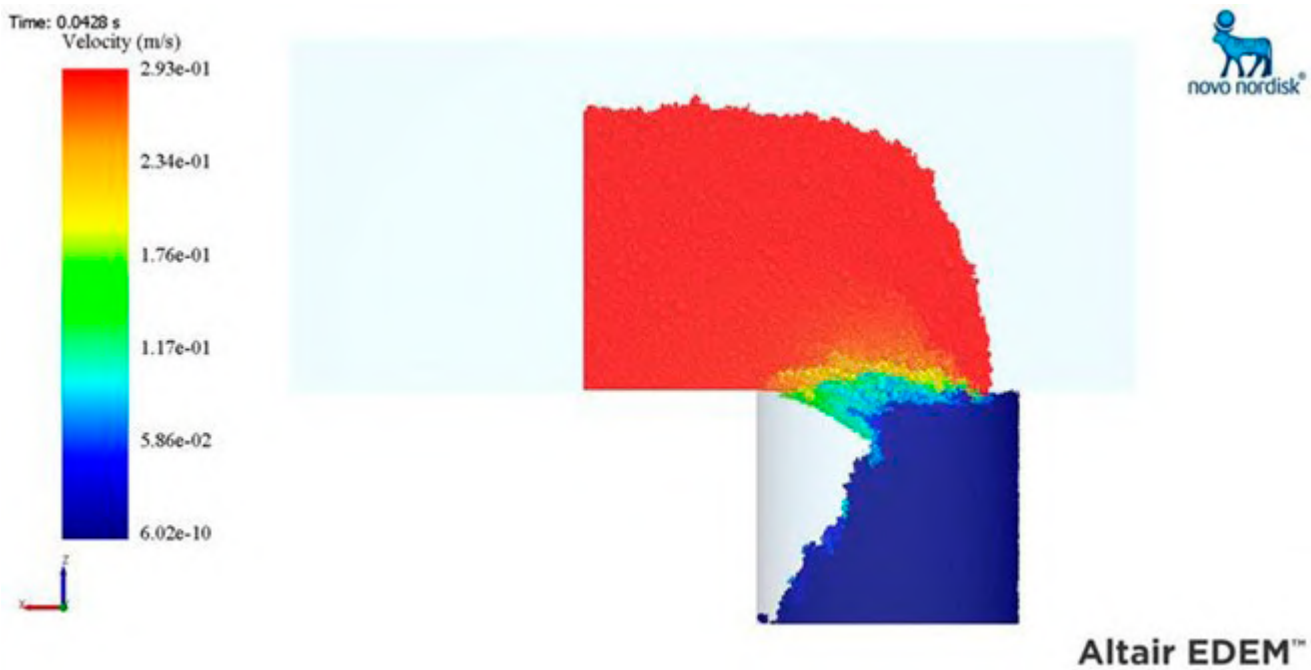


FIGURE 1 Altair EDEM digital twin of the tableting process – particle velocities during the die filling stage

Turbulence induced aggregation of solid particles in perikinetic and orthokinetic regimes

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Keywords | population balance, aggregation, orthokinetic, perikinetic

Aggregation of solid particles occurs in many industrial processes such as precipitation, crystallization and polymerization. It affects the particle size distribution (PSD) and morphology, hence determines the quality of many products. The evolution of the PSD can be modelled using population balance equations (PBEs) by applying kernels suitable for specific processes and conditions (Hulburt and Katz, 1964). In this work we consider aggregation in turbulent flow.

A basic aggregation kernel is determined by solving the convection – diffusion equation for the pair probability function of the solid particles present in the DLVO potential field for sub-Kolmogorov scale (Melis et al. 1999). Although this method is very precise there are difficulties to apply it in simulations with the PBEs. For such applications one needs to use the simplified aggregation kernels to decrease computational efforts.

In this paper an ensemble averaged convection-diffusion equation is used to derive a simplified collision kernel of small solid particles subject to turbulent flow. The concept of turbulent diffusion is applied to close the turbulent flux term and derive a collision kernel in a closed form. The results obtained using proposed closure scheme are compared to the solution of full convection diffusion equation and predictions of a collision kernel from Bałdyga and Orciuch (2001). The collision kernel introduced in this work can be applied for both perikinetic and

orthokinetic regimes including transitional region, providing a computationally inexpensive technique without significant loss in precision compared to the full model. In addition, developed kernel predicts effect of the system scale relevant in case of turbulent flow.

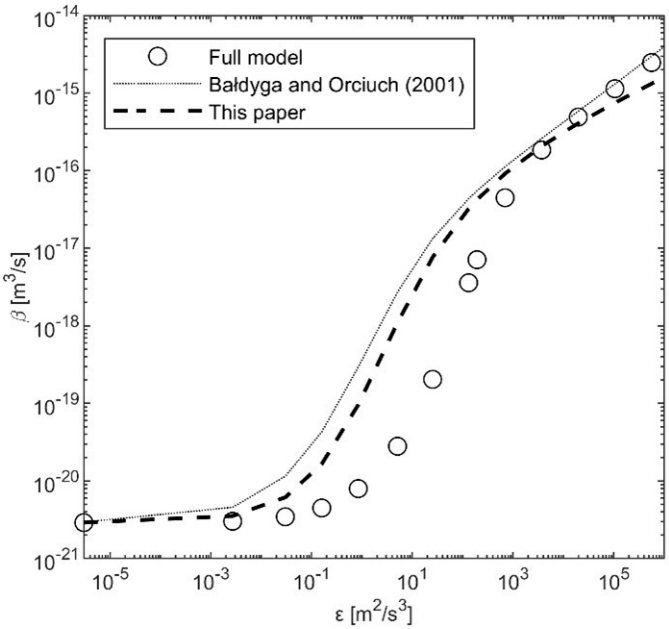


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CFD model validation of a fine particle cold plasma coating bed using Eulerian approach

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Keywords | Multiphase simulation, Eulerian simulations, KTGF, Spouted bed, Cold plasma coating

Surface functionalization of particles is an effective approach to introduce new characteristics such as improved flow properties for otherwise difficult to handle powders. The application of cold plasma coating technology for surface functionalization requires fluidization of the particles to improve the transfer of the coating media to the precursor. However, active pharmaceutical ingredients are mostly fine and cohesive particles, and hence Geldart A or B which are challenging to fluidize. Spouted beds solve this difficulty by applying a high velocity point-source gas jet which can break particle clusters. However, spouted beds must operate within a narrow operating conditions window, which makes both their design and operation a complex task. Models can aid in optimization and design tasks and, in this regard, the objective of this work is the computational fluid dynamic (CFD) model setup and validation.

A laboratory spouted bed cold plasma coater is modeled using the two fluid model (TFM), which has lower computational costs compared to discrete elements models (DEM). DEM models describe the system at a more fundamental level, but large scale DEM simulations are still prohibitively expensive. The TFM challenge is that it requires several submodels and coefficients that must be chosen to accurately predict the behavior of the particle phase, especially for small sized or cohesive powders.

Simulations are implemented in OpenFOAM CFD software for different combinations of models and coefficients, and the results are both quantitatively and qualitatively compared to experimental measurements. 3D simulations are averaged in time and in the depth direction to obtain a 2D map of steady state solid fractions. The 2D map is compared using the structural similarity index measure (SSIM) against

the experimental 2D map obtained from photographs of the laboratory spouted bed. The experimental 2D map is the per pixel average of tens of photographs using a stationary camera. The results show that the parameters with the largest impact on the SSIM for the studied Geldart A particles are the minimum solid fraction to start adding friction stresses, the empirical coefficients of the Johnson frictional pressure, and the choice of drag model.

Effect of the calibration method on the flow of a pharmaceutical powder in a screw feeder: A Discrete Element Method study

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Keywords | Discrete Element Model, calibration method, screw feeder

Powders are used in a wide range of pharmaceutical, chemical, and other industrial applications. Nowadays, modeling tools, such as the Discrete Element Method (DEM), have proven to play a key role in the development and implementation of manufacturing processes in such powder handling industries. The accuracy of these models is directly linked to the selected contact models and the defined input parameters (e.g., static and rolling friction, surface energy). Thus, to gain confidence in the modeling results, it is necessary to link the material properties to the real bulk behavior through calibration procedures.

One relevant and often overlooked aspect in the calibration process is the identification of a suitable experimental test that can capture the critical mechanisms that occur in the real application. As the selection of the calibration test directly influences the predictions of the DEM simulation, this contribution aims to compare the results of two commonly used calibration methods for dynamic flow regimes in the prediction of powder dynamics for a screw feeder, the FT4 powder rheometer and the rotating drum.

The methodology used in this contribution first considers the calibration process, where the DEM input parameters (e.g., static friction, rolling friction, surface energy) are iteratively modified so that the DEM simulation prediction matches the experimental bulk response (e.g., dynamic angle of repose, total flow energy) for SuperTab® 11 SD as a model powder of pharmaceutical relevance. Subsequently, the selected set of calibrated parameters for each calibration method is used to predict the flow dynamics in the screw feeder. The contact model considered is Hertz-Mindlin with JKR.

The results of the calibration allowed the identification of the most influential parameters for each calibration test (i.e., static friction, surface energy). The changes in DEM simulation predictions in terms of screw filling percentage, mass flow rate, and average particle velocities in the screws, highlighted the importance of the adequate selection of a calibration test to prevent the prediction of misleading results. This contribution serves as a preliminary basis on the determination of a suitable calibration test for screw feeding applications and is an important step towards accurate DEM predictions.

Development and numerical simulation using CPFD of a novel configuration of Dual fluidized bed gasifier for increased residence time and tar reduction of biomass

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(1) Wachemo University, (2) Retired

Keywords | dual fluidized bed gasifier, cpfd,biomass,simulation

Abstract

The Dual fluidized bed gasifier has been identified as a promising future for its nitrogen free and medium heating value gas production. residece time of char, steam to char and gas reaction , decrease of tar production and optimization of bed temperature are issues that should be addressed for the gasifier best performance. To address this issues a novel modification of configuration in conventional dfbg has been proposed. The modified configuration consists of a riser a loopseal, a cyclone and chute with a gasifier which has two comparments to

accomplish an internally solid circulating motion to increase the char residence time, temperature optimization, reaction of steam to char and gas reaction in the bed as well as in the freeboard to increase. For investigation of this configuration the commercial code CPFD Barracuda virtual software has been utilized.A normal cpu processer has been used for simulation and it has taken 28days for 90sec simulation. Due to speed of the computer only very important reaction equation has been used for input in the software.validation of the model based on the gas composition is used from the literature.

Numerical study of the effect of an external magnetic field on soft abrasive flow machining by using smoothed particle hydrodynamics

Mohseni-Mofidi, Shoya (1); Bierwisch, Claas (1)
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Soft abrasive flow machining (SAFM) is employed to achieve high quality surfaces for tiny internal channels and passages of complex geometries. Abrasive suspensions used in SAFM are typically two-phase mixtures of a low viscous fluid and abrasive grains. Over the past decade, several researchers have modified SAFM to attain a higher performance. For example, Tan et al. (2016) proposed a double-inlet SAFM and showed that a higher material removal rate is obtained due to a higher flow turbulence that leads to a stronger random movement of abrasive grains. Li et al. (2019) improved the performance of SAFM by applying ultrasonic excitation to help vorticity generations near walls by increasing the internal energy of the flow.

In this study, borrowing the concept of magnetorheological abrasive flow machining proposed by Jha and Jain (2004), we aim to numerically investigate the viability of magnetic-assisted SAFM by studying the change of surface roughness during a machining under the influence of external magnetic fields of different strengths. To this end we employed smoothed particle hydrodynamics (SPH) to solve governing equations for all different phases (Figure 1), namely carrier fluid, abrasive grains, and surface, as well as to resolve interactions among them and to calculate magnetic forces. Hence, a unified SPH scheme was developed that is able to straightforwardly handle the multi-physics of the problem in addition to moving and deforming boundaries. Moreover, although abrasive grains are discretized by SPH particles they are modeled as rigid bodies. Therefore, Constituent SPH particles of an abrasive grain are rigidly rotated and translated together. The results (Figure 2) showed that the application of an external magnetic field can lead to a better performance for SAFM if the strength of the magnetic field is defined carefully.

Prediction of powder flow of pharmaceutical materials from physical particle properties using machine learning.

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Keywords | Particle properties, powder flow, machine learning, Industry 4.0

Understanding powder flow and how it affects pharmaceutical manufacturing process performance remains a significant challenge for the pharmaceutical industry. Materials with small particle sizes tend to be poorly flowing, which can lead to manufacturing issues, and thus, they can add cost and time to the development of a robust process. On the other hand, spherical particles tend to be free-flowing. However, in a real-world application, it is very challenging to achieve spherical particles (Capece, Silva, Sunkara, Strong, & Gao, 2016; Ghadiri et al., 2020).

Machine learning (ML) models are used for this work to predict the powder flow of pharmaceutical materials from physical particle properties. Particle and bulk properties are analysed for 118 pharmaceutical materials, which are classified into cohesive, easy flowing, and free-flowing, based on Jenike's classification for powder flow.

Results show that classification and regression algorithms are useful to predict powder flow from physical properties, achieving a performance of 0.758 for regression and 0.835 for classification. An additional experimental dataset is used to validate the models. This work suggests that powder behaviour can be predicted from particle properties using ML methods.

These ML models will be implemented as a screening tool to decide the manufacturing route, helping to achieve the key goal of digital design within Industry 4.0 of being able to better predict properties whilst minimizing the amount of material required and time to inform

process selection during early-stage development (Arden et al., 2021).

		Predicted			
		Cohesive	Easy flowing	Free flowing	Σ
Actual	Cohesive	23	3	3	29
	Easy flowing	8	17	9	34
	Free flowing	0	13	42	55
	Σ	31	33	54	118

Figure 1: classification model confusion matrix.

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Simulating large scale particulate systems using a combined 3D-1D approach

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Keywords | CFD-DEM; Coupled Simulations; 3D-1D; Simulation

Particle and particle-fluid simulations have become an essential tool for the design and optimization of industrial processes. Advanced models are available that allow for the depiction of complex physical phenomena at very small scales. Challenges typically arise from a combination of large problem sizes, long process times and the fact, that phenomena that arise at micro scales influence the macroscopic behaviour of the system. We present a combined 3D-1D approach for the simulation of industrial scale units that consist of repeating elements and show two application examples.

The first application is the simulation of a plant-scale catalytic reactor that is used for methane dehydroaromatization (MDA). At micro scale we resolve the structure of the 3D printed catalyst using Computational Fluid Dynamics (CFD) and discrete element method (DEM). An immersed boundary method (IB) is used to resolve the particles in the flow field and to calculate the heat transfer between the solid and the fluid. Furthermore, the MDA reaction is calculated at the fluid-catalyst interface, the chemical composition of the fluid is traced. Several representative volume elements (RVEs) are defined along the reactor and simulated this way. A 1D low-order model is used to bridge the solution between the RVEs and thus obtain a solution for the entire reactor.

The second application of the proposed model is the simulation of the heating of a conductive porous medium with pore sizes in the order of micrometers. Both electrical current and external heat sources lead to an increase of the temperature. The internal structure of the medium requires a fine-grained resolution to properly resolve the complex contact network. However, due to the difference in scale between a whole unit and the internal pore structure it is unfeasible to use a direct simulation approach. RVEs are used to compute the heat transfer at local level. A specifically developed 1D model is used to extrapolate the boundary conditions between the RVEs and thus allow for the simulation of the entire process.

Use of the Discrete Elements Method for describing abrasive wear in bulk material handling technology

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Keywords | Discrete Element Methode, DEM, Wear, Abrasive Wear, Sliding Wear, Impact wear, Ship loader

In the field of bulk material handling technology, one of the main reasons of system failures and downtimes is the high level of wear that occurs when highly abrasive mineral bulk materials and powders are conveyed. Due to the potential complex geometries of the conveyor systems and the often-unknown loading conditions of the individual system elements, conventional analytical and experimental methods for determining the type and location of the wear are only suitable to a limited extent. The use of the Discrete Element Method (DEM) makes it possible to get an insight into the system of the plant to analyse both, the flow behaviour of the bulk material and the stress on individual system elements. In this way, a qualitative statement on the extent of wear can be made using suitable models.

In order to be able to predict the service lifetime based on quantitative volume losses due to the wear process, including wear characteristics and location of occurrence, a suitable calibration process is necessary, considering the specific wear liner materials and bulk materials. In this article, a general procedure for wear prediction using DEM simulations is presented on the basis of an industrial project of a cascade chute of a ship loader.

Modelling hopper discharge of elongated particles with different shape representation methods

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Keywords | DEM, spherocylinder, hopper flow

The design of bulk material handling equipment in many industrial and process engineering applications is directly linked to the understanding of the material's behaviour. Hoppers are a crucial part of the chain since their smooth usage dictates the succesfulness of the downstream process of the final product. Therefore, investigating the flow and the parameters that affect the discharge rate of the material play a significant role in the design process.

Many researchers have looked at the material flow in hoppers by using spheres in the Discrete Element Method (DEM) (Zhu. et al., 2006, Ketterhagen. et al., 2008, Anand. et al., 2008, Ketterhagen. et al., 2009, Li. et al., 2009, Yu. et al., 2011, Balevičius. et al., 2011). However, there are limited studies that have focused on the behaviour of elongated particles. Since most materials have complex shapes, it is important to understand the flow, interaction and dynamics of particles other than spheres. The proposed work uses DEM and replicates real-life elongated materials by using sherocylindrical particles that flow through a wedge-shaped hopper. A sensitivity analysis was performed that determined the critical parameters. Different aspect ratios and friction have been employed in this work and the results have been compared against experimental studies as well as correlations reported in literature (Tangri H. et al., 2019). Furthermore, the DEM simulations have been repeated with equivalent multi-sphere and polyhedral particles, investigating the effect of different shape representation methods on the overall flow and discharge rate of the material.

The simulations were in very good agreement with experimental results and previous studies, demonstrating that simulations can be used efficiently to predict the flow through hoppers. Furthermore, the aspect ratio and static friction showed that they have a big effect on the material flow. Specifically the discharge rate is being reduced with increasing aspect ratio whereas changing the coefficient of static friction varies the overall flow as well as the fill height. The results showed that small values of friction cause an increase of the discharge rate, and a critical value of around 0.2 was found to eliminate the effect of fill height.

Virtual optimization of polishing processes based on DEM simulations

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Keywords | DEM, particle simulation, polishing, industrial application

Polishing and surface finishing processes are widely used in industry to meet cleanliness and surface property requirements. Especially

for small components bulk material technologies with a wide range of setups are applied. The surface finishing quality depends on many parameters like the component's geometry, the polishing stones, the process fluid, and technological parameters. Typically, these parameters are optimized based on experience and experiments. As a robust assessment is quite hard due to variable incoming part conditions and scatter, large test series are required.

To save time consuming and expensive experiments and to gain deeper process insight a method based on DEM particle simulations was developed for the virtual investigation of polishing processes. It allows to modify parameters individually and to determine their effects on the polishing quality without being influenced by differing incoming part conditions or other disturbing effects. The assessment of the surface finishing quality is based on the local shear power density. For a robust evaluation method averaging procedures regarding particles, time and surface patches were implemented.

For the DEM simulations computational challenges arise due to the problem size. Hundreds of thousands of polishing stones, thousands of parts (both with arbitrary convex or concave geometries), requirements on the part discretization (which must capture the geometry and the local polishing intensity field), and small time steps result in a huge number of particle vertices and faces and simulation time increments. Therefor it is impossible from an economic point of view to run the simulation for a full model and for the complete process duration. By idealizations and algorithmic means a computationally efficient method was developed to obtain results within reasonable time.

The presentation covers key aspects of the developed method, means for increasing the computational efficiency, and its application to an industrial use case.

10 STED* IN PARTICLE-BASED MATERIALS AND PRODUCTS

* Science, Technology, Engineering and Design

ORAL COMMUNICATIONS

A Water Storing, Flowable Granular Material for Concrete 3D Printing

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Keywords | Additive manufacturing, water storage, dry water, granular material, flowability

Additive manufacturing (AM) in construction can produce free-form structures without special moulds or tools, thereby leading to near independency of production costs and complexity of components.

The project at hand combines two AM processes to create steel reinforced concrete, namely Selective Paste Intrusion (SPI) and Wire Arc Additive Manufacturing (WAAM). SPI is a particle-bed based AM technology, which constructs concrete components by spreading a layer of coarse sand. The cement paste is extruded locally at appointed areas and the steps are repeated until completion. Furthermore, WAAM is used to produce steel reinforcement by arc welding. However, high temperatures during WAAM generate heat transfer into the particle-bed, consequently reducing water in the cement paste and decreasing concrete strength.

For that reason, the present research intends to develop tailored coarse sand particles that store water, and thus, compensate for water loss as well as promote cooling. It is hereby of utmost importance to maintain sufficient bulk properties for SPI to create consistent components regarding strength and shape accuracy. One possible solution arose with dry water, which is classed as a free-flowing, water-rich powder, storing up to 98 wt.% of water. Essentially, dry water consists of water droplets that are encapsulated with hydrophobic fumed silica nanoparticles. Dry water is mixed with coarse sand as an additive, thus, storing water in the bulk with the potential of sustaining initial flow and bulk properties.

The integral part of this study is to investigate the influence of different mass-related variants of dry water and vary the water contents within the bulk material. For process related reasons, the main methods of analysis are the dynamic and static angle of repose as a measure of flowability. Additional analysis addresses critical questions with respect to the progression of dry water capsules during stress events in mixing. As a result, micro-computed tomography (µCT) and scanning electron microscope (SEM) are applied for image analysis. Further research confronts the scalability of dry water, making it feasible as an additive for future large-scale concrete projects.

Analysis of the powder layer quality in the Selective Laser Sintering process from experiments and DEM modelling

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Keywords | Selective Laser Sintering, Powder layer formation, Discrete Element Method, Model calibration, Model validation, Quantitative analysis

Selective Laser Sintering (SLS) is an additive manufacturing process, which uses a laser beam to fuse powder particles together and create 3D objects. Although many are the advantages of SLS, one drawback is the quality of the powder layer, which depends both on the powder flow properties and on the operating conditions, such as the way in which the powder is spread. Therefore, it is important to gather information about the spreadability, that is the ability of powders to be uniformly spread across a plate (Snow et al., 2019), in order to optimize the powder layering.

To investigate on the quality of the layer generated in the spreading process by a moving blade a new approach was developed which included experiments and modelling. For the experiments a purposely made set-up was able to reproduce the spreading conditions of a typical SLS machine. The DEM was applied to the simulation of the spreading of polymeric powders. The interparticle interactions were described by means of the Hertz-Mindlin (no slip) with JKR model. The interfacial adhesive surface energy between the particles was estimated by assuming that the pull off force of the JKR model equals the interparticle force obtained from the isostatic tensile strength. The strength was evaluated through the shear test experiments. A calibration procedure was adopted to evaluate the most appropriate value of the particle rolling friction. Different approaches were assessed to calibrate the model parameters (Lupo et al., 2021) and the one based on the comparison

between the experimental bulk density with that measured from the simulations turned out to be adequate and the most reliable.

The DEM model using the calibrated parameters were validated by comparing the results with the experiments. The comparison was carried out in terms of the wavelet power spectrum applied to one dimensional spatial profiles of greyscale levels in the spreading direction obtained from images of the layers obtained both from the simulations and from the experimental layers. It is found that the obtained DEM model allows to quantitatively evaluate the quality of the powder layer in terms of typical wavelength of the surface asperities.

Heat transfer modelling and in-situ experimental validation of single-layer selective laser sintering of polyamide powders

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Keywords | Laser Sintering, heat transfer, infrared thermography, finite element methods, layer thickness.

Selective Laser Sintering (SLS) is one of the most common types of Additive Manufacturing (AM) process using granular materials in which a three-dimensional object is built layer by layer using laser as a heat source. Delamination of layers in the final product due to lack of fusion and poor laser penetration into powder bed is a major source of a defect in SLS. The temperature distribution along the depth of the powder bed, which is affected by the thermal conductivity of the powder bed is vital in calculating the optimum layer thickness. In this study, a stationary laser sintering experimental set-up equipped with two thermal cameras is built to measure the temperature distribution along the depth and the top surface. A thin layer of polymeric powder is spread on infrared-transparent Zinc Selenide (ZnSe) glass so one of the thermal cameras can see the heat transfer along the depth from the bottom. The powders used in this study are carbon black treated polyamide 12 and polyamide 6.

The study also involves calibration of thermal cameras and calculation of powder surface emissivity at various temperatures. Using both the thermal cameras, the effective thermal conductivity of the powder bed is calculated. The experiment is repeated for various layer thicknesses ranging from 200 microns to 1mm. A three-dimensional finite element model considering the powder bed conduction, convection, radiation and phase change is built and validated against the experiments. The thermophysical material properties are calculated experimentally, which in turn enhances the model. A relation between the process parameters such as laser power, particle size distribution, layer thickness and the mechanical characteristics of the final specimen is established.

DEM simulation of polymeric powders spreading in selective laser sintering at different temperatures

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Keywords | Selective laser sintering, High Temperature, Discrete Element Method, Spreadability, 3D printing

Selective laser sintering (SLS) generates sintered products in any 3D geometry by delivering powders sequentially to the powder bed. The final mechanical properties of products strongly depend on the distributed powder layer quality in the powder bed.

Powder layer quality in the SLS process is significantly sensitive to the temperature experienced during the preheat stage. However, preheating improves the final product properties, drastically makes powder flow behavior worse, and reduces powder spreadability due to the interparticle forces increment. Accordingly, the temperature would be investigated to optimize the SLS efficient condition to obtain high-quality powder layers.

A Discrete Element Method (DEM) could be carried out to simulate the spreading procedure in particle scale, reduce many experimental trials and errors, and reach an acceptable preheat temperature.

To this end, a DEM-based model was developed by considering powder thickness in the powder bed, the temperature of the system, and the spread tool shape and speed as parameters to simulate the powder spreading process. It is important to note that the Anton Paar shear cell experiments were used to calibrate the DEM simulation.

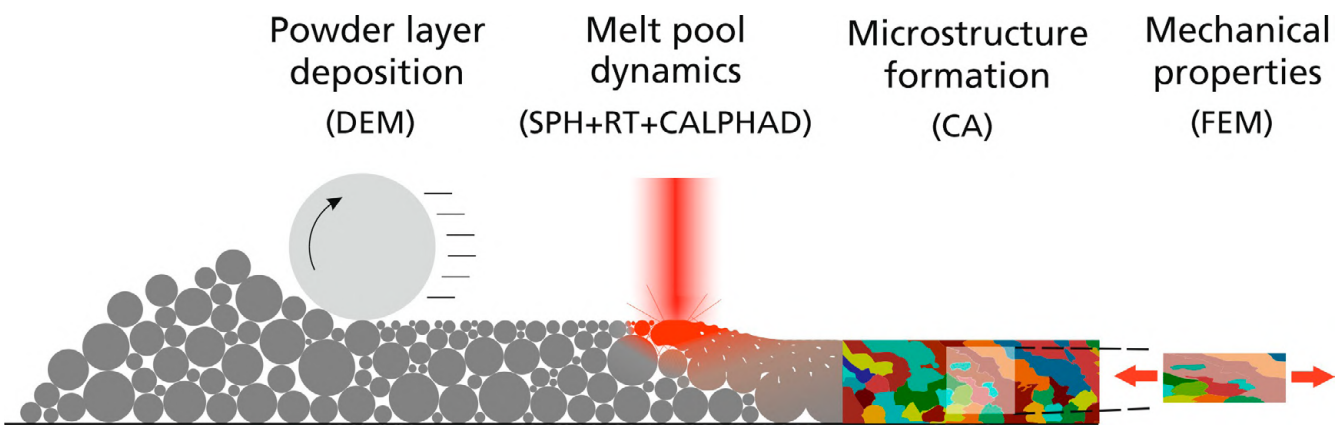
In conclusion, this will help understand the powder suitability used in an SLS process and the best condition, particularly preheating temperature for a specific material, which can simplify the optimization and its layer quality from the ambient temperature to the high temperature approaching the melting point. Furthermore, it made a complete set of experimental and modeling tools to correlate the powder flow properties with the final bed properties at various temperatures.

A holistic simulation workflow for laser powder bed fusion

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Keywords | holistic model, laser powder bed fusion, model combination, data transfer, multiscale modelling

The laser powder bed fusion process is modelled using a sequence of physical simulations. The Discrete Element Method (DEM) is used for powder spreading simulations yielding realistic powder beds. Ray tracing (RT) is employed to calculate the laser energy deposition in the material. Smoothed Particle Hydrodynamics (SPH) simulations are then used to study the thermo-viscous flow in the melt pool considering melting and re-solidification, radiation and heat transfer, thermocapillarity and vaporization pressure. Material properties required for predicting the melt pool dynamics are obtained from thermodynamic CALPHAD simulations. The temperature field of the melt pool is coupled to a Cellular Automaton (CA), which calculates the growth of dendritic grains and, thus, provides a prediction for the microstructure formed during solidification. This microstructure serves then as input for Crystal Plasticity Finite Element Method (CP-FEM) simulations to qualitatively describe texture dependent mechanical properties.



Polymer powders for powder bed fusion via cold wet comminution in stirred media mills

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Keywords | Additive manufacturing, Powder bed fusion, comminution, stirred media mills, Polymers

Fine microparticles of engineering thermoplastics are of high interest for additive manufacturing (AM) processes, like laser powder bed fusion of polymers (PBF-LB/P). PBF allows for manufacture of functional parts of complex geometries that are not easily accessible by conventional manufacturing approaches. Although, the choice of available materials is limited, as the majority of commercialized PBF-LB/P powders are polyamides. Production of novel feedstock materials of good processability is challenging due to high demands that powders have to fulfill in terms of particle and bulk solid properties. Novel thermoplast feedstock powders of good flowability e.g. may be obtained by a process chain approach consisting of a wet grinding step, allowing for very fine products particles down to less than 5 µm size, followed by thermal rounding and dry coating (Schmidt et al., 2012, Schmidt et al., 2016, Dechet et al., 2018). Besides flowability, thermal properties of the thermoplast and rheological properties are essential in PBF-LB/P, thus, degradation of the polymer during powder production and

contamination of the product needs to be minimized.

Within this contribution, cold wet comminution of polybutylene terephthalate and polycarbonate in stirred media batch mills is assessed. The grinding products are thoroughly characterized by vibrational spectroscopy, X-ray diffraction and dynamic and isothermal scanning calorimetry. The dependency of the grinding result on fundamental process parameters (stirring speed, grinding media size, process time, process temperature and system composition) will be discussed with focus on energy efficiency and minimal grinding media wear (Tischer et al., 2022). Moreover, experimentally determined grinding limits for various thermoplasts are discussed in the context of their mechanical properties and continuous production of microparticles by a grinding and classification circuit is briefly sketched.

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Generation of magnetic and plasmonic parts by laser powder bed fusion from nano-additivated polymer powders

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Keywords | Laser powder bed fusion; Polymer powders; Polyamide 12; Thermoplastic Polyurethane; Nanoparticles

Additive manufacturing techniques such as laser powder bed fusion (LPBF) offer the versatility in material design required to produce parts suitable for aeronautic and medical applications. However, the library of materials processable by LPBF is limited, reducing the possibility of manufacturing parts with custom functionalities. To address this limitation, the incorporation of nanoparticles to base powders employed for LPBF has been proved to widen the processable materials and provide additional functionalities.

With this aim, a general route to nanoadditivate polymer powders for LPBF with homogeneously dispersed nanoparticles is proposed. Without the addition of chemical precursors, spherical nanoparticles are generated by pulsed laser ablation (PLAL) or pulsed laser fragmentation (PLFL) in liquids. The nanoparticles adsorption on the polymer powder is conducted directly in the obtained aqueous dispersions after PLAL/PLFL, followed by drying, powder analysis and LPBF processing. The homogeneous nanoparticle dispersion on the polymer powder achieved is shown to transfer the characteristic plasmonic absorption to the produced powder and printed parts with a nanoparticle loading of 0.01 vol% [1].

The process success and versatility in terms of nano-additives and base polymers employed is demonstrated for different nanoparticles (gold, silver, carbon, iron oxide...) and polymers (TPU and PA12). Showing the possibility to widen the selection of processable materials by LPBF (i.e. TPU) and providing additional optical [1] and magnetic [2] functionalities to materials already employed such as polyamide 12 (PA12).

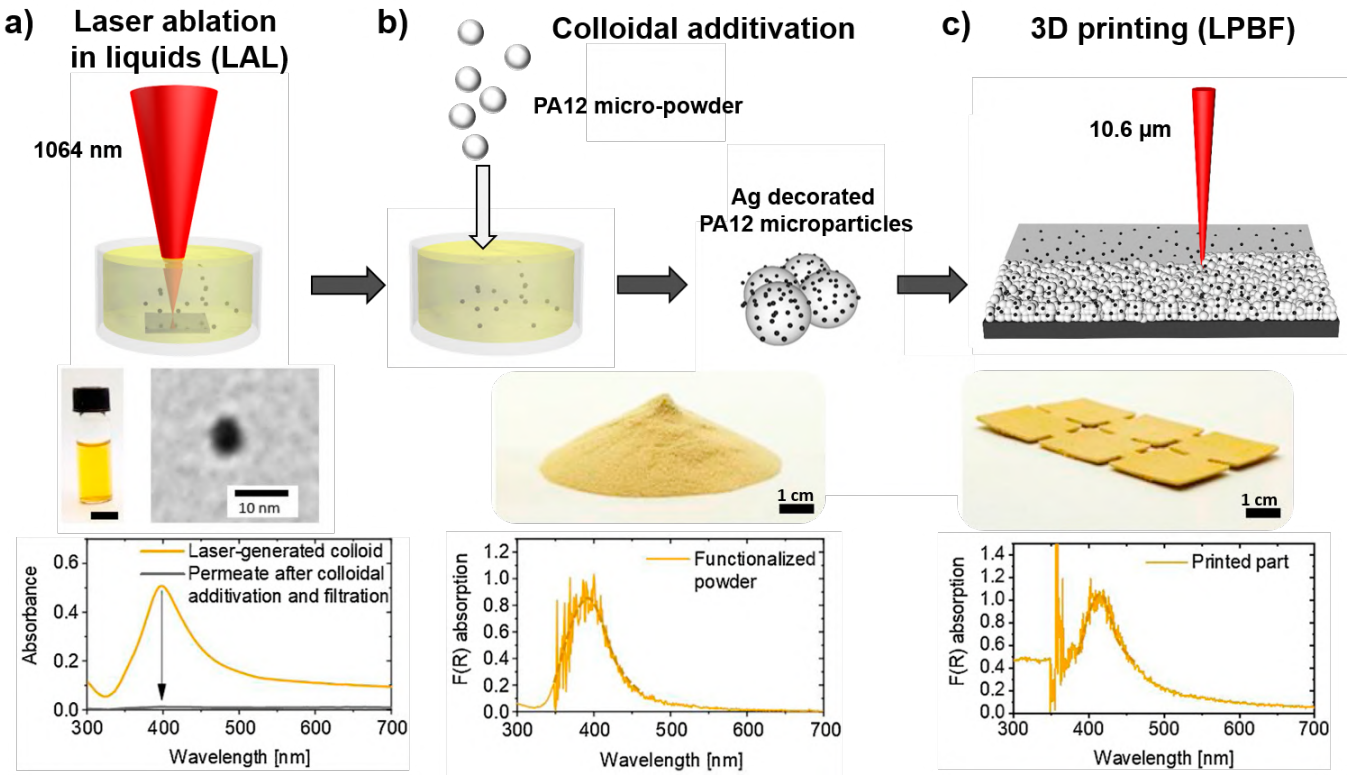


Figure 1. a) Schematic procedure employed for the generation, supporting and LPBF processing of nanoadditivated polymer powders. b) LPBF generated parts employing the nano-additivated polymer powders. c) LPBF produced Ag-PA12 part.

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3D structural characterization and thermal cyclability study of a composite salt hydrate for thermochemical energy storage applications

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Keywords | Composite thermochemical energy storage materials, MgSO4-7H2O, structural characterization, thermal characterization, compaction, extrusion, thermal cyclability

Thermochemical energy storage (TCES) is one of the most promising thermal energy storage (TES) technologies for addressing the supply-demand mismatch. Salt hydrates have attracted considerable attentions in recent years as TCES materials due to their high energy density, low cost. However, one of the major challenges preventing the commercial deployment of such technologies is the material structural stability/integrity and associated thermal cyclability. The structural stability of the material is affected by volume change during hydration-dehydration cycles. This can lead to structural disintegration and blockage of the reactor, and hence deterioration of heat and mass transfer, and reaction processes. The use of a hosting material to form composite salt hydrate has been proposed to address this challenge. With such a method, a semipermeable and structurally stable container is formed to retain the salt hydrate while providing water vapour transport channels. In this study, MgSO4·7H2O is used as the salt hydrate, and both porous and non-porous particles are used a hosting material to

formulate composite TCES materials. The composites are manufactured in different ways including tableting, extrusion and granulation. They are subject to thermal cycling and characterisation for their thermal properties and structural integrity. A 3D X-Ray Tomographic (XRT) method is used for the structural characterisation. Dynamic Vapour Sorption (DVS), Differential Scanning Calorimeter (DSC) and Thermal Gravitational Analyses (TGA) are used for thermal characterisation. The formulated composite materials are found to have an energy density as high as 0.85GJ/m3 and an excellent structural integrity.

Optical and thermal analysis of EVA Doped with luminescent organic dyes as emerging encapsulant material for photovoltaic conversion

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Keywords | PV module; encapsulation process, polymer encapsulant, organic nanoparticles, thermalization technique.

Optical and Thermal Analysis of EVA Doped with Luminescent Organic Dyes as Emerging Encapsulant material for Photovoltaic Conversion

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1. Problem statement

Generally photovoltaic (PV) modules respond poorly to wavelengths less than 400 nm. Only the range 400nm-1150nm is used in current crystalline silicon solar cells. Otherwise, the efficiency of solar cells is limited by the so-called spectral mismatch between the solar spectrum and the solar cell. This spectral mismatch induced the thermalization phenomenon in the PV module. One way to reduce PV module losses by thermalization is the generation of multiple electron-hole pairs from selective incident photon in the polymer encapsulant. Actually, some of the losses can be circumvented by including a luminescent down-shifting (LDS) layer. The LDS layer absorbs UV photons before they are absorbed by the encapsulant, and emits longer wavelength photons that transmit through the encapsulant to the cell [1,2]. The goal of frequency converters is to modify the solar spectrum and organic dye exhibits very high Photoluminescence Quantum Yield (PLQY) in solution, which is a fundamental property for successful LDS.

2. Methods

The most effective method of incorporating organic dyes into the flexible EVA sheets found was soaking in solutions of dyes. In this work, we have described the methodology to include the organic dye in EVA polymer encapsulant, as emerging material.

3. Conclusion

It is revealed, through optical results, an increase of EVA transmittance in visible wavelength range. This increase in transmittance is due to the shift of UV wavelength by LDS technique to visible wavelength.

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Synthesis of nanocomposite coatings via combining an arc reactor and a magnetron sputter device

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Keywords | PVD, nanocomposites, magnetron sputtering, aerodynamic lens

Nanocomposite coatings containing transition metal nitrides exhibit improved mechanical, tribological, and anti-oxidation properties. However, conventional PVD (physical vapor deposition) processes, such as the reactive magnetron sputter process, can only synthesize nanocomposite coatings with restricted material combinations (Tillmann et al., 2019). For instance, one cannot synthesize TiN/CrN nanostructured coatings using a magnetron sputter device, instead, only single- phase coatings of CrTiN would be produced.

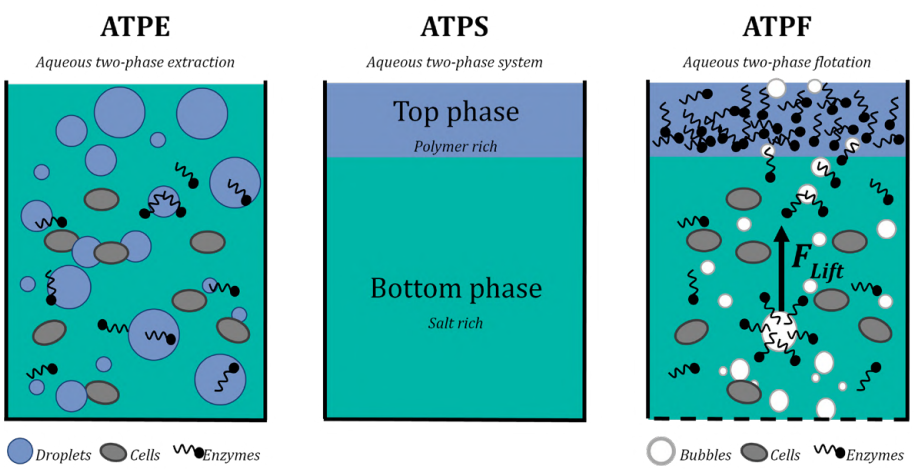
In this work, we developed an approach of combining an atmospheric pressured arc reactor to a magnetron sputter device to obtain coatings consisting of the nc-CrN/nc-TiN nanocomposite structure. Titanium nitride (TiN) nanoparticles were synthesized in an atmospheric pressured arc reactor. The produced nanoparticles were in-situ transferred to a vacuum PVD (Physical Vapour Deposition) chamber employing a designed aerodynamic lens system. The nanoparticle transmission efficiency of the aerodynamic lens system was investigated by model calculations and laboratory measurements. Effect of the PVD process conditions on the properties of incorporated nanoparticles and deposited thin films were also studied.

Our results indicated that the synthesized thin film reveals a two-phase structure consisting of nanocrystalline CrN and TiN phases according to 2D grazing incidence X-ray diffraction (GIXRD) analysis, as displayed in figure 1.

Continuous Aqueous Two-Phase Flotation (ATPF) for Enzyme Purification

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Keywords | Enzyme Purification, Aqueous Two-Phase Flotation, Process Design, Continuous Flotation



Introduction

Industrial enzymes are biotechnological products with many uses as functional additives in foods or washing agents or as catalysts in industrial processes. They are produced by microorganisms in big fermenters with up to 100 m^3 volume. Downstream process for enzyme purification is complex and includes several separation steps which sums up energy costs and product losses. Hence, new technologies that simplify the downstream are in focus of research. In 2009, Bi et .al. introduced aqueous two-phase flotation (ATPF) as a new alternative method combining separation and concentration of penicillin G from fermentation broth.

ATPF is a combination of aqueous two-phase extraction (ATPE) and flotation. Hence, ATPF combines the advantages of aqueous two-phase systems (ATPS), e.g. biocompatibility, environmental friendliness and product selectivity, with the high mass transfer of flotation. This allows biomolecules to be selectively separated from complex biosuspensions and concentrated in a collecting top phase.

Optimization of ATPF through Process Engineering

Despite the application of ATPF for many different biotechnological products from various sources, it is remarkable that almost no process

optimization has been carried out to date. Only recently the important influence of the gas input (volume flow and bubble size) could be shown and possibilities to influence these process parameters could be presented [2]. In process control studies, experiments were carried out with the model enzyme phospholipase A2. It could be shown that continuous phase exchange can significantly increase the space-time yield of ATPF [3].

In this presentation, a strategic approach to process development for the application of ATPF to existing enzyme production processes will be presented. Results for the investigation of an optimized gas input, a suitable apparatus design, a continuous process control as well as an online measurement technique for monitoring the product quality will be presented.

Literature

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[3] L. Jakob, M. Heinzmann, H. Nirschl, Development of a continuous aqueous two-phase flotation process for the downstream processing of biotechnological products, Sep. Purif. Technol. 278 (2021) 119657. https://doi.org/10.1016/j.seppur.2021.119657.

Continuous flow gram-scale synthesis of plasmonic pigments with tailored colour

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Noble metal particles may initially appear prohibitive for large scale optical applications due to bulk material costs being substantially higher than those of traditional colorants. They do however bring numerous competitive advantages which make them interesting contenders in certain niche, high added value applications. One is the fact that a wide palette of visible and near-infrared properties can be provided without changing the constituent material, but rather its morphology. Another advantage is the highly efficient extinction of light by noble-metal containing particles by virtue of their localized surface plasmon resonances (LSPR). This means that far smaller amounts of material would be required to achieve a similar optical effect as standard colorants. Despite these opportunities, the use of plasmonic particles in optical coatings is rarely reported. The most severe bottleneck seems to be the lack of solution processing techniques that are capable of synthesising plasmonic particles with sufficient tunability of the structural parameters (and thus optical properties) at the scale needed for rigorous pigment investigations. This is due to the fact that most synthetic protocols are based on very low volume, low concentration batch reactions.

To solve these problems, we have developed a two step continuous flow process in which silica particles in the form of spheres or flakes in the sub- to low micron size range are partially coated with thin silver patches. In the first process step, the core particles are decorated, using salt-induced coagulation, with a very small number of pre-formed gold nanoparticles. In the second process step, electroless plating is performed whereby the seeded core particles, combined with solutions of silver nitrate and formaldehyde are mixed with ammonia. By adjusting the reagent ratio it is possible to control the size and shape of the silver patches and thus their optical properties. In the present contribution we will show how this process, and subsequent downstream handling, can be used to synthesise pigment powders with targeted colour on the scale of several grams per run. Moreover, we will show how advanced characterisation and electrodynamic modelling can assist in the optimization of the colour properties of the patchy particle pigments.

RoboPharmacist: Automatic Compounding Line for Continuous Manufacturing of Personalized Multi-Component Capsules

Sonntag, Erik (1); Vrba, Jan (1); Štěpánek, František (1)
(1) UCT Prague

Keywords | Personalized medicine, Polypharmacy, Continous manufacturing, Automatic compounding

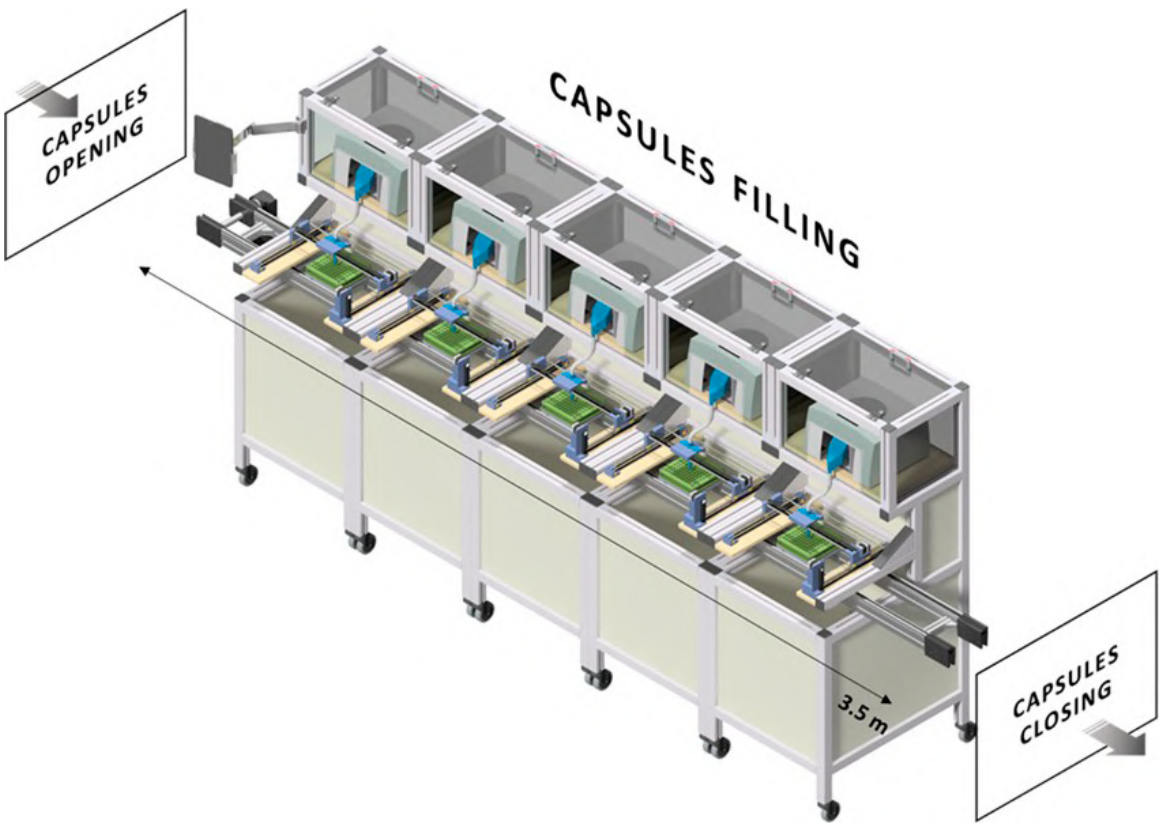
Polypharmacy represents a condition in which a chronically ill patient takes five or more different drugs a day. Surveys in the United States showed that 10% of all patients and 30% of elderly patients suffered from polypharmacy (Sutherland et al., 2015). The medication regimens of such patients are usually very complex and challenging to follow. In 2013, WHO reported that 50% of chronic patients fail to follow the

prescribed regimen. Such a condition is called patient non-adherence. Several studies have been conducted estimating the cost of non-adherence for health care systems. This estimate is $200 billion for the US, while for the EU, it is around €125 billion per year (Viswanathan et al., 2012). A large part of the price is, for example, hospitalization after incorrect use of drugs. Ways to significantly increase adherence include **reducing the number of dosage forms** that patients must take and the **treatment personalization** (Claxton AJ et al., 2001).

"RoboPharmacist" (or "Lekobot" in Czech) is a new technology developed at UCT Prague designed for automatic manufacturing of personalized medicines consisting of multi-component mixtures of several Active Pharmaceutical Ingredients (APIs). This technology works on the principle of precise automatic compounding of sub-units (particles from 0.1 to about 5 mm in diameter) into hard capsules. Each sub-unit contains one type of API and is formulated to meet the conditions of proportionality and bioequivalence with the original single-drug dosage form. By placing the appropriate number of sub-units into the hard capsule, the same therapeutic effect can be achieved as with the original drug. Furthermore, due to the high API content and small dimensions of the sub-units, multiple types of sub-units can be combined inside a single capsule. Using this technology, it is thus possible to reduce the number of dosage forms for the patient and adjust the doses of individual APIs in the capsule according to the individual patient's needs.

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Scale-up of high-shear wet granulation through model-driven design

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(1) Siemens PSE, (2) Roche, (3) The University of Sheffield

Keywords | High shear granulation, scale-up, regime analysis

Wet granulation processes are difficult to scale up because conventional methods require ample experimental data to determine the

most favourable operating conditions. A systematic model-driven framework can facilitate this scale-up process by reducing the number of experiments required, especially at large scale. Therefore, an approach is required which is based on the wet granulation process, key operating conditions and performance indicators. Such an approach helps to design large scale experiments and reproduce desired experimental outcome observed at small scale which is needed for a successful process scale-up.

In this study, a scale-up framework is proposed and applied to a high-shear wet granulation process. At all scales, we emphasised experimental reproducibility during the experimental design based on the previously published regime analysis and process understanding. A limited number of small-scale experiments are used to identify the favourable operating range and determine the necessary formulation-dependendent parameters. Common scale-up rules and regime analysis are applied to successfully scale up to from lab scale to pilot plant. The outcomes of this case study demonstrate that this systematic framework is capable of scaling up favourable operating conditions and predicting the operating range at pilot- plant scale.

FLASH COMMUNICATIONS

Enhanced powder deposition through charge control substances in additive manufacturing

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(1) Institute of Particle Technology, (2) Institute for Particle Technology

Keywords | additive manufacturing; charge control substances; powder deposition; polypropylene, dry coating

Polymers are one of the most important materials of our time. Due to their good intrinsic properties, as well as the diverse processing possibilities, they have become indispensable in many areas of life. Polymers are also of particular interest in powder-based additive manufacturing. However, the polymer powders for this manufacturing process must also have special properties. These include, in particular, good flowability with a rather narrow particle size distribution, preferably below 100 µm. In this size range, the interparticle adhesive forces have a much stronger effect relative to the weight force. This in turn often results in cohesive powders that cannot be processed or can only be processed inadequately. The use of flow aids, such as highly dispersed silicon dioxide or titanium dioxide, can significantly improve the friability of the powders. However, these flow aids also have disadvantages: they can negatively affect the coalescence of the plastic melted during the additive manufacturing process, which can lead to highly porous and/or brittle components. Therefore, it is a desire on the part of manufacturing to limit the amount of additive in the system to the minimum necessary.

This paper focuses on the advantages of using nanoparticulate charge control agents as flow aids in additive manufacturing. The aim is to realize a better powder deposition in the build space and lower mass fractions of additive compared to the state of the art by charge control. Furthermore, the charge control agents make it possible to enable a completely new powder application process for additive manufacturing, electrophotographic powder deposition. For this purpose, the polymer powders are functionalized by dry coating and analyzed with regard to their flowability, thermal properties and the resulting electrostatic surface potential during powder application. Subsequently, selected formulations will be used in the manufacturing process to evaluate the effects of the charge control agents.

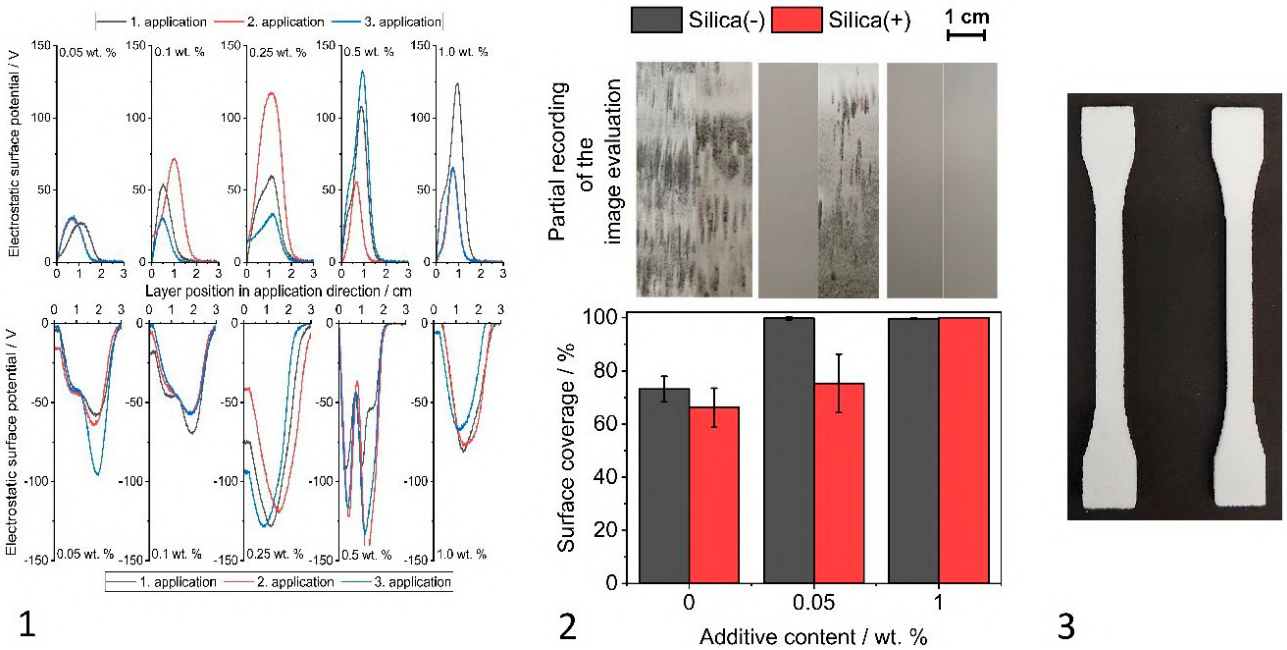


Figure 1: (1) Change of the electrostatic surface potential in dependence to the additive content; (2) Electrophotographic powder deposition of functionalized polymer powder; (3) tensile test specimens

Concrete parts from the powder bed – Material modification for selective cement activation

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(1) TU Braunschweig, Institute for Particle Technology

Keywords | concrete, powder bed, 3D-printing, SCA, flowability, porosity, spring back

Different additive manufacturing processes for concrete are current research topics: Concrete 3D-printing using selective cement activation (SCA) is one of them: SCA is a so-called powder bed process, in which the concrete part is built up layer by layer. For each layer, a mixture consisting of aggregate (sand) and cement is first applied to form a homogeneous and even powder bed, by first spreading it with a counter rotating roller and compacting it with a concordant rotating roller, afterwards. Subsequently, water is selectively sprayed onto the powder bed and hydrates the cement in the desired area. As SCA is a comparatively new process, there are some major challenges, currently: 1) the achievable strengths are still too low for structural use, 2) the shape deviations of the components from the initial design and 3) a high cement content must be used to achieve good component properties.

To meet the above-mentioned challenges of the process, the powder properties of the aggregate-cement mixture for the SCA process are investigated. One reason for the low strength is the high porosity of the printed components in comparison to conventionally produced structural concrete. Therefore, one goal is to improve the flowability of the powder and reduce the porosity of the compacted powder. To achieve this, the surfaces of the starting materials are modified in dry processes using nanoparticles or liquid additives, respectively. The effects of the modifications are characterised by means of the flowability, bulk density and tamped density. To link the experiments with the SCA manufacturing process, the bulk density of the powder in the printer is assessed and test specimen are printed and evaluated. Initial tests have shown that the compressive strength of printed components can be improved by more than 25 %. Furthermore, the application process of the powder is part of investigation. To quantify the stresses, pressure mapping sensors are used. During compaction, a sprickback effect was observed, which is further investigated by linear compaction tests.

Ensuring processability in additive manufacturing: Metrological challenges in assessing quality of aged polymer powder feedstocks

Hesse, Nicolas (1); Peukert, Wolfgang (1); Schmidt, Jochen (1)
(1) Institute of Particle Technology (LFG)

Keywords | powder bed fusion, additive manufacturing, polyamide, bulk solid properties

Additive manufacturing (AM) techniques like powder bed fusion (PBF) allow for the production of polymer parts with a high freedom of design that is unmatched by conventional manufacturing techniques. Due to the layer-by-layer production, complex or customized geometries can be built without the need for specialized tools. However, PBF still faces some shortcomings. Since only a small fraction of the material in the process ends up as finished part, while the larger amount of it merely acts as a supporting structure, it is necessary to reuse the non-molten powder for economic and ecologic reasons. However, in the case of polymeric materials, the long processing times at high temperatures lead to extensive changes in properties of the used powder.

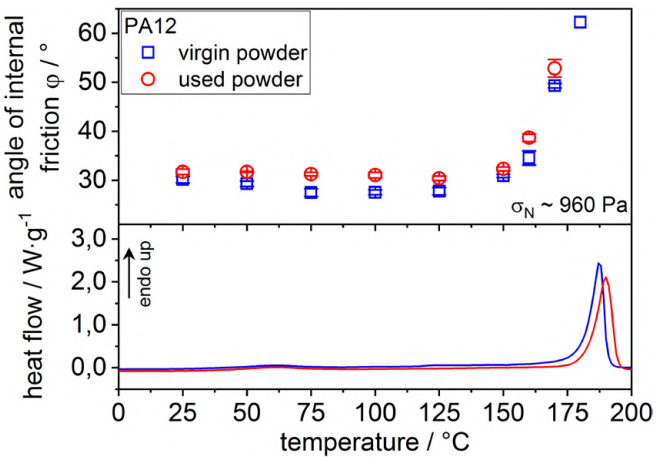


Figure 1 Temperature- dependent angle of internal friction of PA12 powder in shear-experiments (Hesse et al., 2021)

Using the most commonly utilized PBF material polyamide 12 as an example, this contribution examines chemical and physical aging effects which result from thermo-mechanical stress during the AM process and shows possibilities for ensuring the capability of the material to be processed in further production cycles. Both bulk mechanical properties of the powder and rheological properties of the polymer melt, which becomes increasingly viscous due to thermal stress, are addressed. Since even slight changes in the packing properties of a powder can lead to changes in its processing behavior or in the performance characteristics of the parts built from it, special attention is paid to the metrological challenge of detecting corresponding powder characteristics and tracing their origin back to the morphology of the individual particles. In addition to established methods of bulk material measurement, adapted methods based on a customized rheometer setup are also considered in order to better replicate the conditions present in laser sintering (cf. Figure 1).

N. Hesse, B. Winzer, W. Peukert, J. Schmidt; Additive Manufacturing 2021, 4, 101957.

JE. CHALLENGES OF MICROPLASTICS ANALYSIS AND CONTROL - Oral Communications

Extraction and characterization of microplastics from industrial effluents: Continental Portugal analysis

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Keywords | Microplastics, Industrial effluents, extraction, characterization

Microplastics (MPs) awareness has been growing particularly after several reports about "garbage patches" in the world (Magalhães et al., 2020). Plastics do not biodegrade in any meaningful way and, up to now, only a small percentage of plastic waste is recycled, being all the rest dumped in landfills, incinerated or simply not collected. The distribution of MPs within the water ecosystem depends on particle density and size and environmental characteristics, such as winds and currents (figure 1) (Zhang et al., 2019).

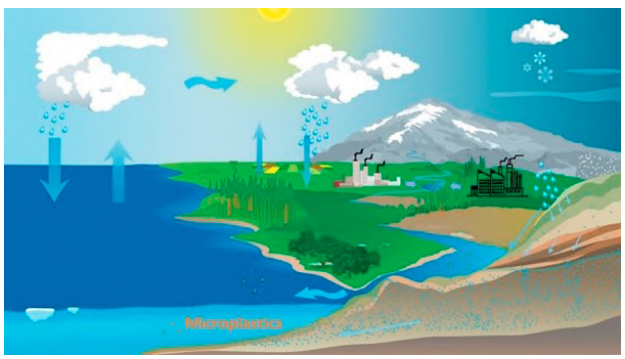


Figure 1: Transportation of MPs to areas

In the present study, different industrial effluents were analysed and characterised to determine which MPs in the treated water released from Wastewater Treatment Plants (WWTP), predominate and contribute the most to the environmental contamination of aquifers which, eventually, will end up in the coast of Continental Portugal. Overall, this work suggests strategies for MPs analysis in WWTP, thus allowing mapping the different types of MPs prevalent in Portugal. The establishment of such database will enable the creation of reliable laboratory models to test new and green removal processes, based on the flocculation by bio-flocculants.

Acknowledgments: This work was financially supported by the Portuguese Foundation for Science and Technology, FCT, via the PhD grant (2020.07638.BD) and the Strategic Research Centre Projects UIDB00102/2020 and UIDB/05183/2020.

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Dynamic modeling to characterize the fate of plastics in the LCA methodology

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Keywords | Life Cycle Impact Assessment, Matrix Model, Residence Time, Micro- Macro-plastics

While LCA is the most common tool used to assess the environmental impacts of a product, some limits constrain its use. Current LCAs do not take into account the plastic leakages in the environment and their impacts (Woods et al., 2021). In life cycle impact assessment (LCIA), emission-related impacts are usually assessed through three parameters: 1) its transport and degradation in the environment (fate), 2) its ability to interact with living species (exposure), and 3) its effect on these species (toxicity). As a first step, we propose to develop fate factors as they are predominant factors considering the persistence of plastics. We base our approach on the USETox matrix model (Rosenbaum et al., 2007) to assess (eco-)toxicity in LCA.

In this modeling approach, the fate of a compound can be interpreted as its residence time in different environmental media (freshwater, ocean/seawater, soil, and air compartments) after an emission in a receiving compartment. The phenomenon affecting the residence time include degradation within each compartment as well as transfers between compartments.

They are represented with their yearly degradation or transport rate (Kd) that are represented in a matrix form and inverted to build the fate factor matrix.

Also, recent studies have shown that degradation of plastics is type-, size-, and shape-dependent (Chamas et al., 2020). Therefore, a fate factors matrix is built for each type of plastic (e.g., macroplastic of PET, cylinder). The degradation rates are computed from recent literature on this topic, considering a dynamic approach to assess the fate at the short and long term.

Finally, the fate factors can be used to assess the persistence into different environmental compartments of plastic leakages generated from a product system assessed in LCA.

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Woods, et al., 2021. A framework for the assessment of marine litter impacts in life cycle impact assessment. Ecol. Indic. 129. https://doi.org/10.1016/j.ecolind.2021.107918

What implications to food sustainability and safety do microplastics have in the fisheries and aquaculture sectors?

Vázquez-Rowe, Ian (1); Aldaco, Rubén (2); Margallo, María (2); Ita-Nagy, Dian (1); Kahhat, Ramzy (1)
(1) Pontificia Universidad Católica del Perú, (2) Universidad de Cantabria

Keywords | fishing; human health; marine debris; marine litter; seafood

Marine plastic waste has become a critical environmental concern worldwide, as high concentrations have been reported in many geographical context, becoming ubiquitous in most marine compartments. Microplastics constitute an important and increasing fraction of this accumulation, in a process that can originate through two pathways. On the one hand, due to direct emissions that occur from the technosphere. On the other, linked to the fragmentation processes of macroplastic waste that occur once the waste enters the marine compartment. The impacts of microplastics on aquatic organisms was vaguely covered in the literature until recently. However, in recent years there has been a push to understand how the exposition and the effect of these polymer particles can affect these marine and freshwater species and, ultimately, fishery and aquaculture activities destined for human consumption. Therefore, the aim of this study was to analyze the effects of microplastics on fishing and aquaculture, identifying the links with food safety and sustainability. Current studies have observed multiple potentially damaging effects of microplastics on marine biota, mainly at lower trophic levels. It is plausible to assume that fishing stocks and aquaculture systems will suffer setbacks due to these damages. However, additional research is needed to understand the potential effects on human health, especially considering that smaller microplastics and nanoplastics, for which data is very scarce, are the particles most likely to be absorbed by human tissues.

Evidence of microplastics accumulation in the ocean in Latin America and the Caribbean

Vázquez-Rowe, Ian (1); Aldaco, Rubén (2); Laso, Jara (2); Fernández-Ríos, Ana (2); Ita-Nagy, Diana (1); Kahhat, Ramzy (1)

(1) Pontificia Universidad Católica del Perú, (2) Universidad de Cantabria

Keywords | microplastics; plastic waste; waste management

Microplastics are released to the ocean through multiple pathways, constituting an increasing global environmental concern. Despite the lack of data, in recent years, however, numerous studies have delved into the fate, effect and damage that plastics, namely microplastics, generate once released into the ocean. Unfortunately, most of these studies have concentrated in littoral areas in Europe and North America, although an increasing number of papers are being published in other regional contexts. In fact, the specific characteristics of the Global South (e.g., widespread mismanaged waste and wastewater) make microplastics an even greater challenge than in the Global North. In this context, this study performs a critical review related to the prevalence of microplastics in the ocean in Latin America and the Caribbean, analyzing also the potential sources of microplastics that generated this release to the marine environment. A high proportion of the studies assessed point towards mismanaged waste, inland or offshore, as well as mismanaged wastewater as the most critical sources of plastic pollution into the ocean in the region. However, there is a lack of results linked to the effects that these microplastics are generating on local biota and human health, which indicates the need to strengthen research in terms of effect and damage in the region.

Development of a method for in-situ real-time monitoring of microplastics in wastewaters using a photo-optical measurement technique

Schuhen, Dr. Katrin (1); Sturm, Michael (1); Klein, Raphael (2); Maaß, Sebastian (2)
(1) Wasser 3.0 gGmbH, (2) SOPAT GmbH

Keywords | Microplastics, Detection, Fluoresecent Dyes, Real-time Monitoring, Photo-optical-sensor, Process control unit

For an appropriate risk assessment of microplastics in the environment and food, it is essential to know the levels of microplastic contamination. Extensive research has been carried out in the field of microplastic detection in recent years. While common methods such as Raman spectroscopy and pyrolysis GC-MS are time consuming and require trained personnel and expensive equipment, there is a need for an inexpensive and easy to use method. Staining microplastics with fluorescent dyes (e.g., Nile Red (NR)) has great potential to meet these criteria, because it is cheap and easy to apply.

Fluorescent dyes are commonly used for microplastics detection, as they show stronger fluorescence signals for plastics than for natural particles. In our work, we tested NR and newly developed derivatives to achieve greater selectivity for plastic particles and more intense fluorescence [1]. Also new fluorescent dyes for in-situ staining of microplastics in water samples are developed.

Another big challenge in microplastics detection is applying the process in-situ for a real-time monitoring. Therefore, in the next step fluorescent staining is connected with a photo-optical sensor using algorithm-based image-analysis [2] for in situ detection of microplastics (SOPAT). The photo-optical sensor uses strobe-light to illuminate multi-phase-system containing particles. A high-resolution camera captures the reflected light and images are then processed in a software using a specifically tailored algorithm for irregular shaped particles based on greyscale gradients. The combination with fluorescent staining allows in-situ detection of microplastics in a continuous process.

Results of this study aim to prepare the permanent monitoring of microplastics contamination in municipal- and industrial wastewaters. Another application of the sensor is for continuous process control for the microplastic removal process Wasser 3.0 PE-X®. Results will be presented.

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JE. III ANQUE-DECHEMA LEADING EDGE CONFERENCE "PARTICLE TECHNOLOGY. SHAPING THE FUTURE" - Keynote lectures

Thermal energy storage in thermal solar power plants by molten salts: past, present and future with nano-particles

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(1) Universidad Complutense de Madrid

Keywords | Thermal Storage, molten salts, high temperature corrosion monitoring, nano-particles, nano-fluids

Molten salts are the present technology for thermal solar power plants, composed by a sodium and potasion nitride eutectic mixture. This molten salt limit the operation temperatures upto 570ºC, and subsequently the efficiency of the plant. In order to increates the operation temperature of the heat and storage transfer fluid, new approaches are considered to increase the efficiency of the plant:

- New molten salts formulations: molten chlorides and molten carbonates.
- Molten nitrates plus nano-particles additions

A complete review of the estate of the art, and the latest results from the UCM research group will be included in this talk. The effects and limitations of nano-paricles additions of copper, silicon and aluminum oxides will be establish.

Importance of Particle Technology for Circular Production of Today´s and Future Batteries

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(1) TU Braunschweig, Institute for Particle Technology

Keywords | Battery recycling, cathode materials, anode materials, electrode production, electrode structure, solid state batteries, ionic conductivity, electric conductivity

A major task achieving a sustainable electro mobility is the energy efficient production of the batteries as well as the circular usage of the materials [1]. Along the circular process chain, from recycling of end-of-life batteries over (re-)synthesis of the cathode and anode materials to the production of the electrodes and cells, particle technology plays an important role.

Within the recycling of lithium-ion batteries, comminution, classification and separation processes are decisive to recover almost 100% of the so-called black powder mass out of cathode and anode materials before the hydrometallurgical treatment [2]. Based on the gained secondary materials, new particulate active materials have to be synthesized and functionalized. For the production of lithium-ion battery electrodes the active material particles like graphite (anode) and lithium metal oxides (cathode) have to be dispersed dry and usually also wet together with a conductive agent (e.g. carbon black) and a binder in an appropriate solvent. By applying a high intensity dry mixing process in advance of wet dispersing the carbon black can be well dispersed and a short wet dispersing process can be achieved [3]. However, more and more fully dry production of electrodes comes into focus. As a good physical parameter to describe the dispersing of the carbon black the internal porosity of the carbon black agglomerates/aggregates was identified. By applying the appropriate stress intensity in the high intensity mixing step an optimum specific capacity at high C-rates can be achieved. Moreover, the drying and calendering processes have to carried out in a way that the demanded electrode structure and by that demanded cell performance can be achieved.

In the future particle technology will also gain much importance for the design and production of all solid state batteries, especially based on sulfides. An effective way to produce the sulfide solid electrolytes is a mechanochemical synthesis in high energy mills. Moreover, the sulfide solid electrolytes have to be composed with the cathode material particles and conductivity additives like carbon black in a way, that

maximum ionic and electronic conductivities are achieved at a maximum cathode material content.

From CO2 to value-added products: particles for the electrocatalytic conversion of CO2

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(1) Universidad de Cantabria

Among the possibilities for CO2 utilisation, the electrochemical route appears as a very attractive option. In addition to converting CO2 into different compounds of interest under low pressure and temperature conditions, the development of electrochemical CO2 conversion processes, coupled with intermittent renewable energy sources, can be considered an excellent way to use the surplus of renewable energy that is otherwise wasted, to reduce the captured CO2 and convert it into value-added products and/or fuels. This talk will present a brief overview of relevant achievements that the use of catalytic particles has allowed in the development of processes for the electrochemical conversion of CO2 into valuable products.

A wide range of different products can be obtained from the electrochemical reduction of CO2. The lecture will discuss those that are currently attracting greater research interest, which include CO, formic acid/formate, and other hydrocarbons (mostly methane and ethylene) or alcohols (mainly methanol or ethanol). Several examples will illustrate that the nature of the particles used as electrocatalyst has a crucial role in the selectivity and efficiency of the process.

Then, focus will be placed on showing that not just the nature of the particles, but also other aspects like particle size and electrode configuration (i.e. the way in which the particles are arranged in the electrode) can have a dramatic effect on the process performance. This will be exemplified with some results obtained on the development of continuous processes for the electrochemical conversion of CO2 into formic acid/formate. These results will also serve to reveal that significant progress has been made in recent years and some excellent promising results have been achieved, but the simultaneous optimization of the requirements for practical implementation is still a challenge.

The keynote lecture will end with some final remarks, highlighting the need to continue research efforts to overcome current limitations, where the use of advanced electrocatalytic particles with innovative electrode configuration will play an important role.

Thermoplast feedstocks for laser powder bed fusion of polymers - novel approaches for production and process-adapted characterization

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Keywords | Additive manufacturing, laser powder bed fusion of polymers, thermoplast powder, flowability, spreadability

Additive manufacturing (AM) processes allow for the production of individualized components of complex geometries without the need for tools or molds. If functional AM-built plastic components of good mechanical properties are desired, typically powder bed fusion of polymers with laser beam (PBF/LB-P), also often referred to as selective laser sintering (SLS), is employed. In this AM process, a powder layer is spread onto the building platform within a heated build chamber and the contour of the part to be produced is selectively fused by a laser. Then, the building platform is lowered, a new powder layer is spread and the next cross-section of the component is fused. The process sequence is repeated, until the build job is finished. The component quality is determined by the interaction of the AM machine with the feedstock powder, i.e. proper AM process parameters need to be identified, that allow for dense parts of good dimensional accuracy. Moreover, the feedstock powders need to exhibit optimal bulk solid properties, i.e. particle size distribution, shape, packing density, flowability and spreadability, as well as appropriate thermal and rheological characteristics.

While there seem to be hardly any boundaries regarding design, there are quite some restrictions concerning the variety of commercially available PBF plastic feedstock powders. By now, the most widely used feedstock for PBF/LB-P is polyamide 12 with a market share of about 90 %. The remaining 10 % market share are mainly made up by other polyamides, polypropylene, thermoplastic elastomers or polyaryl ether ketones. In order to broaden the field of application of SLS, novel polymer powders with good processability are needed.

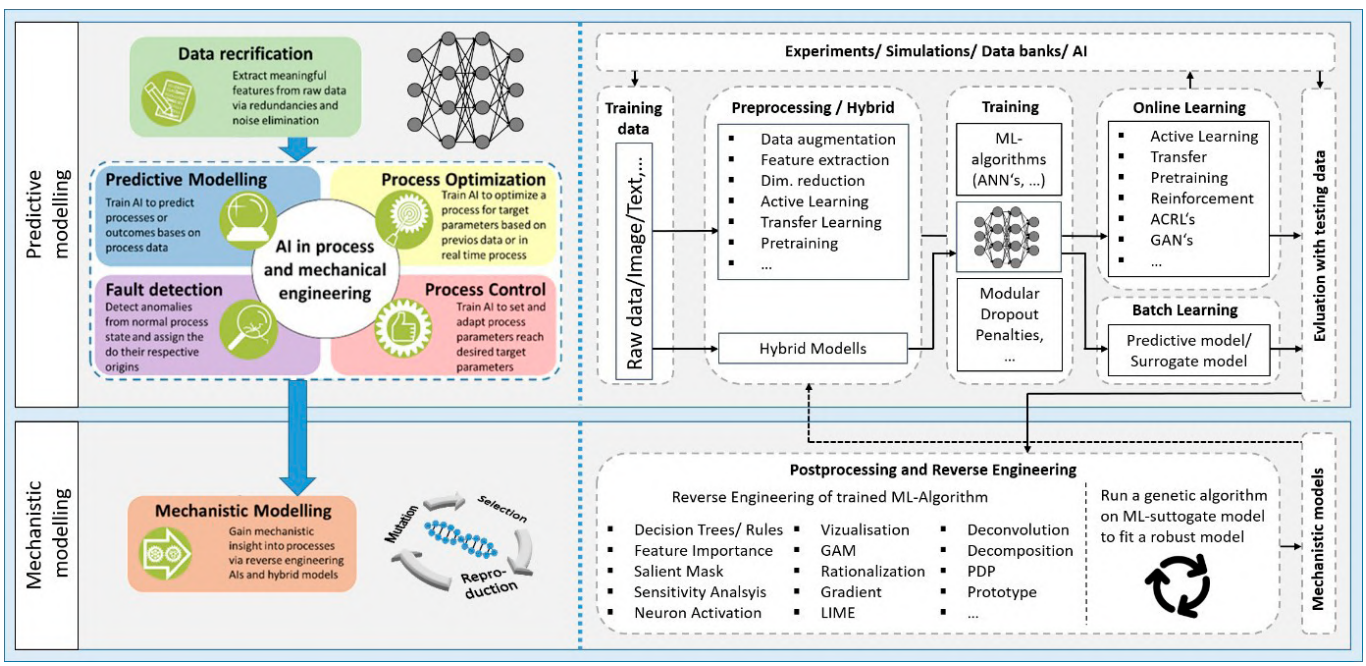
Within this contribution, the state-of-the-art of processes for production and functionalization of novel polymer powders, such as polyesters, for powder bed fusion, including precipitation, grinding, melt emulsification, co-extrusion, rounding and dry coating will be discussed in the context of the feed material's properties. Moreover, modern approaches for process- adapted characterization, for example, measurement of powder flowability at elevated temperatures or determination of thermal properties at high heating rates will be presented. Finally, the effect of powder properties on AM processability and component characteristics will be demonstrated.

Artificial intelligence in the context of particle technology

Schilde, Carsten (1); Hosseinhashemi, Somayeh (1); Röhl, Marvin (1); Böttcher, Ann-Christin (1); Kwade, Arno (1); Thon, Christoph (1)
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Keywords | Artificial intelligence, predictive modelling, mechanistic modelling, genetic programming, Artificial neural networks

Artificial intelligence (AI) is undergoing a boom due to technical breakthroughs in software, e.g., deep neural networks, exponential increases in hardware, data acquisition as well as in generation. AI processes the capability to identifying inherent patterns in large amounts of data which would otherwise be too large and complex for traditional methods of analysis or manual investigation. As key advancements a method for AI training with limited data foundations, as is often the case in academic, as well as industrial use cases, as well as methods for the reverse engineering of black box AI methods will be presented. [1]



This talk will provide a general overview of AI and will demonstrate the application of an AI framework, developed at the institute for particle technology, on various numerical as well as well as experimental use cases such as CFD-DEM simulations or experimental studies in the field of particle technology like mills or a breakage tester, designed for the identification of particle breakage functions. Furthermore, a complex interconnected modular hybrid model of calibration experiments, simulations and a successive mill simulation is presented, as a digital twin for process control or optimization. Reverse engineering strategies will be shown to break down the black box models to more transparent equation systems. Additionally, an outlook to process control, optimization and fault diagnosis as well as to hybrid modelling will be provided, displaying how the trained AI digital twins can be further used to gain mechanistic insights as well as to save time and cost.

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Design of Al alloys for additive manufacturing

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(1) IMDEA Materials Institute, (2) National Center for Metals Research (CENIM, CSIC), (3) LORTEK

Keywords | Additive manufacturing, selective laser melting, aluminum alloys

Additive manufacturing (AM) of robust aluminum alloy components for structural applications has proven challenging due to a high incidence of hot cracking, which results in an impaired mechanical behavior.

In this talk several strategies for the design of high performing aluminum alloys, tailored for laser powder bed fusion AM fabrication, will be presented. Among them, designing new compositions, as well as reutilizing alloys that were initially targeted for rapid solidification studies, and which could not be commercialized further due to the impossibility to produce large volumes, will be considered. In the latter case, unprecedented high temperature properties may be achieved by exploiting the thermal stability of quasicrystalline particles, that can be precipitated in-situ during processing.

A link between the microstructure and the mechanical behavior of the newly designed alloys will be established at a wide range of temperatures in order to provide guidelines for further Al alloy development for additive manufacturing.

Hierarchical, self-assembled particle networks in thin films and in bulk: from transparent electrodes to flexible conductors

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Keywords | Hierarchical Materials Networks Self-assembly Colloids Nanoparticles Composites Hybrid materials

The arrangement of components defines the properties of hybrid and composite materials. Fibers, particles, and macromolecules form networks in rubber tires, battery electrodes, concrete, functional coatings, and other complex materials during liquid processing. The hierarchical network structure translates properties of small-scale components into macroscopic material properties and can lead to emergent functionalities.

In this talk, I will discuss the formation of hierarchical networks from quasi-spherical particles, ultrathin wires, and fractal aggregates on surfaces and in bulk materials. I will outline the underlying colloidal interactions and introduce methods for the in-situ observation of network formation. Challenges include the need for multi-method approaches that combine local electron and optical microscopy, scanning X-ray scattering, and synchrotron techniques to address the multi-scale nature of the problem. I will outline how the analysis of the resulting heterogeneous data can be aided by digital platforms.

The second part of my talk will focus on networks that form in electrically conductive elastomers, transparent electrodes, and spun fibers. I will focus on their structure-property relations and discuss how empirical, industrial knowledge on complex fillers such as Carbon Black can be connected to fundamental concepts such as percolation and fractal dimensions. I will also show how recent, highly defined nanomaterials enable precise control of structure and properties. Finally, I will show how networks with curtailed structures may be used to create new types of multifunctional materials, for example for the integration of sensing and actuation into flexible machine parts and soft robots.

Self-assembly for the formation of highly regular particle structures

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Keywords | Self-assembly Nanostructures Surface patterning bioinspired design

The astonishing variety of functionalities found in nature is almost always based on a self-assembly of relatively simple building blocks over multiple length scales, typical in hierarchical arrangements. This concept continues to inspire scientists and engineers that seek possibilities to create functional materials in a cheap, fast and simple way. Colloidal particles are interesting in that respect as they can be synthesized with high uniformity and precision to yield building blocks with nanoscale dimensions. In the simplest case, such particles are of spherical

shape and do not have any special properties by themselves. However, their uniform size allows such spheres to assemble into ordered crystals, much like oranges or apples on display in a supermarket. In my presentation, I will introduce techniques to assemble such colloidal crystals over macroscopic areas in two- and three dimensions and discuss strategies on how to improve order and homogeneity of these structures. I will then highlight how such colloidal assemblies can serve as masks or templates to create defined surface nanostructures. Translating of the assembly process into the confining element of an emulsion droplet gives rise to supraparticles with a well-ordered internal structure, which are useful components to create hierarchical structuration.

I will introduce how functional, macroscopic properties can emerge from the ordered arrangement of these simple colloidal building blocks, using two examples of the natural world. Structural coloration, responsible for the flamboyant optical effects in many different animals, emerges from the self-assembled colloidal crystals as light waves impinging on the assembly constructively interfere in the visible range. Non-wetting and liquid-repellent surfaces, mimicking the Lotus leaf or the pitcher plant, can be created by controlling the surface topography and surface chemistry. The combination of these two effects can be used to provide simple sensor platforms that distinguish different liquids by their different wettability colorimetrically.

On optimization of particulate products: From synthesis to colour

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Keywords | Process optimisation, Optimisation of particulate systems, Inverting process to design mapping, Optical properties, Colour

In this talk we discuss holistic model based optimisation ranging from nanoparticle synthesis to the optical properties of particulate systems.

First, model based optimisation of time dependent process controls for nanoparticle synthesis are presented taking advantage of sensitivity information. This will be illustrated for the synthesis processes of nobel metal nanoparticles modelled by one and two dimensional population balance equations. The main idea therby is to make us of the newly developed exact method of moments to refomulate the population balance equation in terms of a scalar fixed-point problem.

Second, optimising optical properties of nanoparticles and nanoparticle assembiles by topology and shape optimisation will be discussed. This renders in problems, in which a single evaluation of the objective requires thousands of state problems, namely time-harmonic Maxwell's equation, to be solved. In this presentation, we show how these difficulties can be approached efficiently. One key idea thereby is that while optimising one can use previously computed information to speedup gradient approximations on the fly.

Finally, a proof of concept is presented: Combining the optimisation of the synthesis process based on the optical properties of the respective particulate product result in tailored properties. This proof of concept will be based on the seeded growth process of gold- silver alloy nanoparticles.

Bridging the valley of death: The role of particle technology in bringing functional materials into technical electrochemistry on industrial scale

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Keywords | Electrochemical functional materials, nanoparticle processing, electrode manufacturing

New, micron and nano-sized functional materials play a key role for future technologies that are currently developed in the field of energy conversion (electrolysers, fuel cells) and electrocatalysis (N2 fixation, CO2 reduction). While new materials with outstanding properties are continuously developed, they rarely find their way into – urgently needed – large scale production and industrial applications. Overcoming and bridging this "valley of death" is an interdisciplinary endeavor for which chemical engineering and in particular, particle technology is indispensable (Fig. 1).

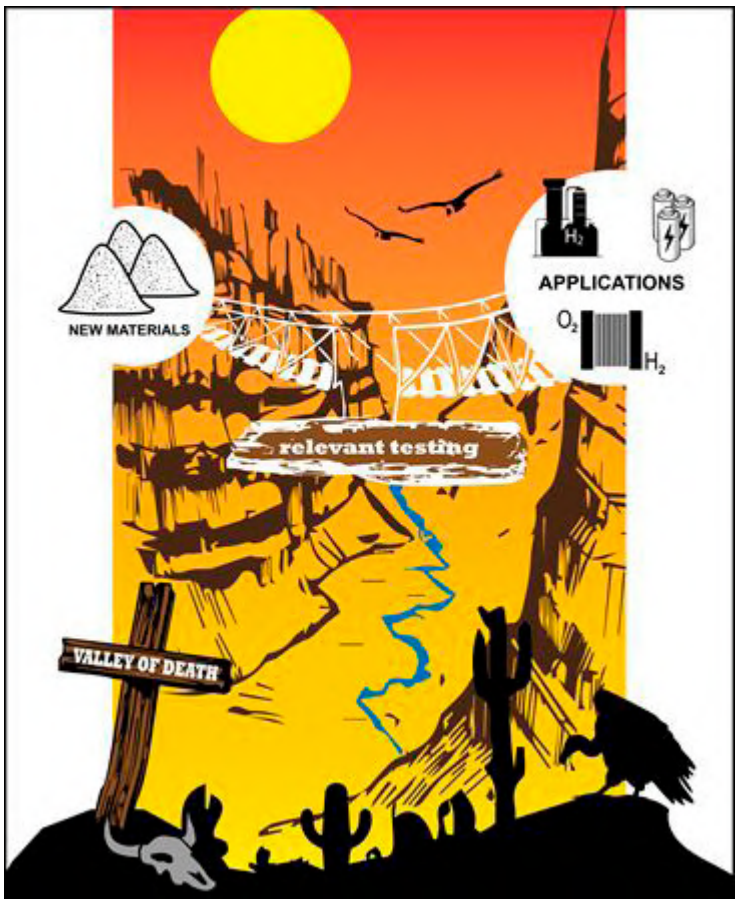


Fig. 1: Valley of death that must be crossed to bring new materials into energy applications (with permission of [1]).

To fulfil this bridging function, the field needs to develop in such a way that we i) collect and make efficient use of more data and develop standard procedures that allow us to better understand process-structure and structure-property relationships,

ii) replace idealized conditions and model formulations by technically relevant scenarios (testing at application concentration, full complexity of a formulation mixture) and iii) apply relevant processes (scalable dispersion, R2R-coating) already on lab-scale as integrating research tools. In my talk, I will introduce concepts that we developed and apply in my team and in collaborations with colleagues to speed up materials development and generate hierarchically structured electrode layers that can be integrated into full cells.

[1] D. Siegmund, S. Metz, V. Peinecke, T. E. Warner, C. Cremers, A. Grevé, T. Smolinka, D. Segets, U.-P. Apfel, JACS Au 2021 Vol. 5 Pages 527-53.

JE. MULTIDIMENSIONAL PARTICLE PROPERTIES: CHARACTERIZATION, SEPARATION, APPLICATION - Oral Communications

Novel concepts in equipment and process engineering for highly specific and multidimensional separation of fine particle systems

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Keywords | Separation, Multidimensional

Separation methods for particle systems depend not only on particle size but also on form, density, and material-specific properties (polarizability, magnetic susceptibility, triboelectric properties). These dependencies can specifically be addressed in order to obtain a material-specific separation, or to solve possible ambiguities which deteriorate the separation performance. In this presentation, we present novel concepts for particle separation equipment as well as process engineering of particle systems with multidimensional characteristics. The forces applied to obtain the separation can be classified as being based on field distortion, on particle inertia in a flow field, on electrophoretic forces or on centrifugal forces.

Field distortion can be used for particle fractionation by dielectrophoresis (DEP), which bases on frequency-dependent polarization of particles and the surrounding medium in an inhomogeneous electric field, with the inhomogeneity caused by the presence of non-conductive elements in the flow field. Particle trapping and subsequent release allows fractionation.

Deterministic lateral displacement (DLD) bases on flow distortions induced by flow obstacles (microposts) which are regularly displaced one after each other. Smaller particles will follow the streamlines, particle larger than a critical diameter will bump against the posts and move to another streamline (“displacement mode”). Fractionating by electrophoretic forces relies on triboelectric charging, which can occur as a result of particle-wall collisions. Different triboelectric properties of particle mixtures can be applied to obtain a material enrichment. Aerodynamic lenses, in which the optimal Stokes number can be selected via the process pressure, can be applied to obtain a narrow transfer function based on the particle relaxation time. Here, ambiguities can occur as the relaxation time is depending on size and density but can be solved when adding a subsequent separation step at another pressure. Tubular centrifuges allow high centrifugal acceleration and semi- continuous operation, a real-time process monitoring allows the continuous evaluation of separation efficiency in multi- component density fractionation. Analytical ultracentrifugation can apart from size fractionation also be used to determine two-dimensional particle size distributions. Magnetic counter-current chromatography allows the fractionation of complex mixtures based on their susceptibility, continuous fractionation is possible by means of simulated moving bed chromatography.

Highly specific and multidimensional separation of fine particle systems with technical relevance: Influencing the separation feature

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Keywords | Separation features, selective agglomeration, electrostatic separation, electrophoresis, Separation at interfaces

For achieving highly specific and multidimensional separations of fine particle systems, clear separation features that enable high yields and separation sharpness are needed. As it is our endeavour to separate not only according to size, but size and shape and zeta potential (Antonyuk), size and shape and surface properties (Garnweitner), size and zeta potential (Breitung- Faes), but size and composition, i.e. surface

properties (Peuker), as well as size and composition and surface properties (Segets), particles must be manipulated to achieve sufficiently distinct properties to enable multidimensional separation. In addition, in-depth characterization was established to assess multidimensionality inline or online and on the ensemble level under consideration of the mass balance to analyze multidimensional separation functions.

In terms of modeling, a multiscale approach for cross-flow filtration, starting from computational fluid dynamics coupled with discrete element methods and ending at a macroscale flowsheet simulation was generated. On the experimental side, gradient gels enabling multidimensional separation in 2D electrophoretic chambers were developed and binary nanoparticle mixtures were separated at high throughput [1]. Interfaces of particles and between non-miscible liquids are tailored to repel, immobilize or allow to penetrate individual particles [2]. In terms of surface properties, pH-induced charge effects were used to tailor the phase transfer properties of particle mixtures and to analyze results with single particle scattering approaches. A similar principle was applied to induce selective agglomeration of wear during fine grinding accompanied with a development of a measurement method for particles mixtures [3]. Finally, stability of binary colloids in solvent mixtures was investigated via stability maps to separate by size and surface properties.

In conclusion, with the described methods, new concepts for multidimensional separation from sub-10 nm up to 1 µm are developed [4]. Though often restricted to 2D separation, generated understanding and developed methodological toolbox paves the way to true multidimensional separation of colloids including modelling, experimental approaches and characterization.

[1] Barasinski et al., J. Phys. Chem. C 2020, 124, 5157.
[2] Peppersack et al., Advanced Powder Technology 2021, 32, 4049.
[3] Menter et al., Advanced Powder Technology, 2019, 30, 2801.
[4] Weber et al., Frontiers in Materials, 2020, 7.

An Introduction to Multidimensional Separation Functions in Particle Technology

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Keywords | particle size; particle shape; particle characterization; classification; sorting; TROMP-curve;

High-tech materials require modern manufacturing processes combined with very precisely defined particle systems of high purity. Within the Priority Program PP2045, which is funded by the German Research Foundation (DFG), a new class of separation processes for highly specific and multidimensional properties of particles in the size range below 10 µm is about to be established.

The understanding and formal description of a separation considering the multidimensional properties is essential for a profound process description and translation into practical process optimization strategies. When considering multidimensionality, a clear distinction must be made between the multidimensional particle properties themselves and the separation forces acting on the particles. Both are influencing parameters effecting the multidimensionality of the separation step itself.

To describe the multidimensional separation, it is necessary to develop or define a multidimensional separation function, which is able to quantify the separation of a multidimensional particle system according to more than one property. This can be done using the data of the particle system prior and after the separation process. Furthermore, based on a multidimensional separation function it is also possible to define the characteristic separation parameters like a median of the separation function, which actually becomes a line in the two-dimensional case. We show how this set of graphs can be used and what methods exist to validate the analysis.

Based on the multidimensional separation function it is possible to deduct a one-dimensional (marginal) separation function for a multidimensional particle system as well, which describes the separation according to one property.

For illustrative purposes, we present the method using a case study of selected particle systems and show the advantage of a coupled multidimensional approach compared to conventional established methods. Together with the methodology for multidimensional characterization of particle systems, the possibility of multidimensional mapping of separation processes opens the door to a new way of process understanding. Ultimately, it represents an efficient tool to quantitatively compare such processes and can thus be the basis for the evaluation of existing but also of new separation processes in the future.

Optimal fiber fractionation for wood-based biorefineries

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Keywords | fibers, hydrodynamic fractionation, pressure screen, empirical model, optimization

Fiber fractionation technology offers the possibility to separate fibers (i.e., elongated flexible cellulose particle) and small particles (fines; smaller and more spherical organic or inorganic particles) in pulp mills. Thereby, the ultimate goal is to perform a shape-based fractionation, i.e., to retain comparably small fibres and reject comparably large spherical particles from the primary product stream. Optimized fibre fractionation technology can be seen as a key breakthrough technology for wood-based biorefineries due its potential to boost product value and widen the product portfolio.

Here we present an empirical fractionation model for a novel fiber fractionation technology (miniFrac, [1]). This model, combined with an optimization procedure, is then used as a design tool to synergize competing fractionation performance characteristics, i.e., product quality, product yield and energy demand. Based on this model, miniFrac is compared to state- of-the-art fiber fractionation technology in the light of (i) long fiber-short fiber fractionation and (ii) fines-fiber fractionation. A comparison with the main competing technology (i.e., pressure screens) shows that in terms of fines-fiber fractionation, miniFrac is outperformed by classical µ-hole pressure screening regarding the purity of the fines fraction. However, miniFrac is capable of outperforming pressure screens regarding product quality and energy demand at a comparable product yield.

We conclude that if - in case of fines-fiber fractionation - reject purity (i.e., fines exclusion) is more important than fines purity (i.e., long fiber remain in the reject), miniFrac can be seen as an appealing technology for wood-based biorefineries. Also, our model can be implemented in any fractionation-based stock preparation concept to support future investment decisions regarding fractionation technology.

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Multidimensional distributions and their separation

Peukert, Wolfgang (1); Frank, Uwe (2); Walter, Johannes (3)
(1) Institute Particle Technology Friedich-Alexander-Universität Erlanden-Nürnberg, (2) Institute of Particle Technology, Friedrich-Alexander-Universität Erlangen-Nürnberg, (3) Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU)

Particle technology evolves towards increasing complexity with respect to multidimensional particle properties. We present the theoretical background for multidimensional particle property distributions by transferring the concepts known from 1D particle size distributions (PSDs) to multidimensional particles comprising of at least two different properties such as size and shape or size and composition. Particle formation and processing lead to the evolvement of nD property distributions [1,2].

In general, particles are separated by acting external force and velocity fields, e.g. external gravitational, centrifugal, electric or magnetic fields. These field forces control particle motion, which allow their separation into different fractions. In addition, short-range force fields induced by particle interactions can be employed as well and used for fractionation. In this special case, the particles interact either selectively with external surfaces, e.g. in flotation with bubbles, in particle interaction chromatography with the stationary phase material, or with themselves for property-selective agglomeration. The proper handling and characterization of multidimensional separation processes acting on multidimensional PSDs is presented.

Within this general framework, we distinguish different cases according their dimensionality. In a 1D separation only one external field drives particle motion, whereas in a 2D separation two external fields, e.g. a centrifugal field and an electrical or magnetic field, act on the particles. We discuss centrifugal separations acting on 2D particle populations. Technical realizations could be a (hydro-)cyclone, a disc separator or analytical (ultra-)centrifuge. Even in a 1D separation the 2D PSDs must be carefully characterized. This is demonstrated for the separation of cylindrical from spherical particles with respect to size and shape [3].

Then, we move on to 2D separations with two acting force fields. Here we present model studies for centrifugal and electric fields. The

(electrophoretic) separation of 2D populations in centrifugal fields serves as example. For nanoparticle characterization in particular, analytical (ultra-)centrifugation is the gold standard in terms of sample statistics, reproducibility and accuracy.

Support of PP2045 and CRC1411 is acknowledged.

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JE. THERMOMECHANICAL BEHAVIOUR OF GRANULAR MATERIALS (MATHEGRAM) - Oral Communications

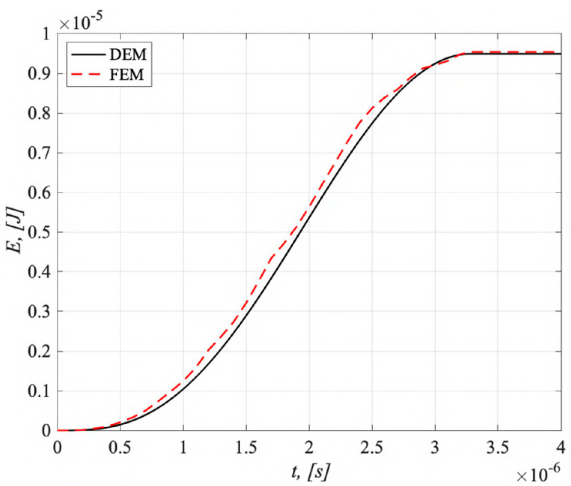
Modelling and validation of energy dissipation due to the plastic deformation of an elasto-plastic sphere during normal impact

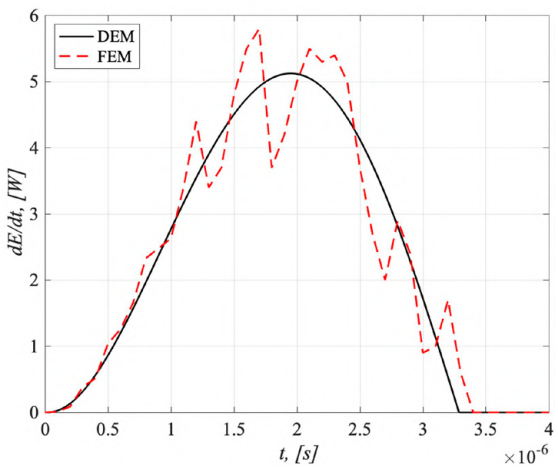
Morimoto, Tokio (1); Kisuka, Francisco (2); Wu, Chuan-Yu (2)
(1) Imperial College London, (2) University of Surrey

Keywords | DEM FEM Elasto-plastic contact model Heat generation

An increase in the temperature of a granular media alters the rheological behaviour and its shear resistance. Such a temperature change due to external heat sources and chemical reactions has been well-discussed; however, there are few studies examining the amount of heat generation due to the plastic deformation of granular particles. A discrete element method (DEM) coupled with a thermal network model (TNM) is a useful tool to examine how heat is generated and transferred within granular materials to address this gap. To run a DEM simulation considering the heat generation due to the plastic deformation of particles, the calculation of the dissipated energy in each time increment is essential.

Nonetheless, such dissipated energy calculations are poorly documented. This paper introduces the equation calculating the energy dissipation within a given time increment based on the elasto-plastic contact model developed by Vu-Quoc and Zhang (1999). The derived equation of the incremental energy dissipation enables to track the time history of the energy dissipation during the normal impact of an elasto-plastic sphere on a rigid wall with various initial normal impact velocities. The time history of the energy dissipation using the elasto-plastic contact model is compared with the results obtained using the finite element method. An excellent agreement is obtained for the impact velocities considered, implying the developed model can accurately describe the energy dissipation during elstoplastic normal impacts.





A DEM Study of Heat Transfer Mechanisms in Dense Granular Materials

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Keywords | Discrete Element Method, dense granular flows, thermo-mecanical analysis.

In this work, the DEM is used to explore different aspects of heat transfer in granular materials. Therefore, a computational tool was developed to consider thermal effects in the classical soft-sphere approach of DEM (Cundal & Strack, 1979). This tool is able to simulate different mechanisms of heat transfer, by means of conduction, convection, and radiation. In addition, heat generation by friction, collision and plastic deformation between particles are also considered. Although not coupled with a Computational Fluid Dynamics (CFD) solver, a 1-way thermal coupling between the particles and the surrounding fluid is possible. For each type of heat transfer and generation mechanism, several numerical models, as reviewed by Peng et al. (2020), were implemented to account for the respective effect. All these implementations were carried out within the open-source framework Kratos Multiphysics (Dadvand et al., 2010).

The focus of this numerical study is on dense particulate systems with static or dynamic behavior, such as packed beds and granular flows. For the former, Representative Volume Elements (RVEs) are employed for simulating the static behavior. For the latter, a model of a rotating drum is taken as reference for performing the numerical investigations. An experimental validation of heat generation due to the particles interactions has been conducted for this type of application. Furthermore, the results provided by all the implemented numerical models are compared, at the same time that the contribution of each mechanism to the total rate of heat transfer and generation is analyzed. Conclusions and discussions based on the obtained experimental and numerical results are drawn in order to achieve a better understanding on the thermal behavior of granular materials.

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Experimental investigation of heat generation in granular mixes

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(1) University of Surrey, (2) Johnson Matthey

Keywords | Energy dissipation, Frictional heating, Plastic heating, Rotating drum, Thermal imaging.

Particle transportation plays a vital role in industrial applications, agriculture, civil engineering, and many other fields. In such processes, the particles are subjected to collisional, rolling, and/or sliding motion with each other and with the boundaries. Consequently, energy is dissipated especially by friction and plastic deformation, and eventually, most of this energy is converted to heat. Despite several numerical studies to explore this area, there is still little knowledge on the underlying mechanisms of heat generation in granular flows and its effects on the global flow behaviour of the granular medium. In addition, the existing numerical studies that are based on theoretical assumptions, lack more realistic data to calibrate the flow parameters. Moreover, there are still not enough experimental outputs to validate such models.

In the present work, we propose and conduct experiments to explore this problem. A rotating drum is used to agitate the grains where their rise of temperature is monitored by both thermocouples and a thermal camera. The use of the thermal camera is more reliable because it provides full-field information about the temperature in real-time. However, this comes with the challenge that the rotating drum should be equipped with an observation window whose materials can allow the thermal camera to access the heat signature of the particles inside the drum. Such materials e.g., quartz and NaCl are notoriously expensive and mechanically fragile hence not suitable for such aggressive experiments. To curb this problem, a stainless-steel sieving cloth of size 425 mm is used for an observation window. Preliminary tests showed that the metallic sieve can partially transmit infrared radiations from the particles inside the drum. With the right calibration of the camera, the temperature of agitated particles is accurately quantified.

With such a robust experimental setup, the next step is to explore the heat generation patterns at the various system and material parameters such as flow regimes, filling ratio, and material properties. This is followed by a systematic calculation of the corresponding heat energy using basic principles of thermodynamics. Then, the total energy losses and thermal- mechanical energy conversion coefficients can be estimated.

The Multi-Level Coarse-Grain Model used in CFD-DEM Simulations of Iron Ore Reduction

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Keywords | DEM, Coarse-Graining, USCM

Applying the discrete element method (DEM) or CFD-DEM simulations to large-scale industrial systems such as blast furnaces or direct reduction shaft furnaces quickly reveals the limits of this simulation strategy. To reduce the computational cost it is inevitable to decrease the level of detail of the method. A potential candidate is the coarse-grain (CG) approach to the DEM (Bierwisch et al., 2009) which lowers the computational demand by using coarser (pseudo) particles to represent a certain amount of original particles. However, due to the violation of geometric similarity, this simple coarse-graining approach fails to capture effects that inherently depend on particle size.

Recently, a multi-level coarse-grain (MLCG) model of the DEM (Queteschiner et al., 2018) has been proposed to alleviate the deficiencies and increase the applicability of DEM coarse-graining. In this model multiple concurrently simulated coarse-grain levels are coupled to adjust the resolution of the system as needed. The MLCG model can also be applied to fluid-particle systems using CFD-DEM. To fully picture industrial plants involving the reduction of iron ore, however, the ability to consider heat transfer and chemical processes is also indispensable. In this work, the reduction process encountered is described via a three-layer unreacted shrinking core model (Valipour, 2009) considering the different iron oxides hematite, magnetite and wüstite as well as the surrounding gas properties. For the MLCG model this means that additional information needs to be transferred between the differently resolved DEM CG-levels as well as between the DEM and the CFD components, i.e. particle temperature and reduction state. In this study we have verified this additional functionality using a silo filled with iron ore pellets undergoing reduction at elevated temperatures.

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Viscous sintering of melting grains

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Keywords | viscous sintering, granular materials, food grains

Viscous sintering occurs when two grains, upon contact, coalesce to reduce their total surface area. The driving force is the surface energy, while viscous and inertial forces hinder the bridge growth. This phenomenon could give rise to agglomeration problems in granular materials such as caking (Rando et al., 2022), yet, at the same time, the mechanism is exploited in additive manufacturing processes, e.g., 3D printing of glass, polymers or food (Rando and Ramaioli, 2022).

Despite all these applications, this phenomenon is still poorly understood and not sufficiently characterised experimentally, particularly when the sintering flow is coupled with heat or mass transfer. The aim of this work is to experimentally investigate the effects of heat transfer and grain size on the sintering dynamics of partially melting fat based food products,i.e. dark chocolate.

Two spherical particles were heated through forced convection using an air stream at[A1] different temperatures. Optical and IR thermal cameras were employed to quantify the sintering dynamics and surface temperature evolution.

Subsequently, the optical images were analysed to extract the bridge size (x) between the grains. Figure 1 shows the typical evolution of the surface temperature and of the dimensionless sintering bridge size.

The sintering dynamics shows an initial lag time, during which no bridge growth is observed, followed by a rapid growth and by a slower growth. Preliminary results showed that the lag time and sintering dynamics are size dependent.

FIGURE 1 Time evolution of the bridge size (x/Dp0, in red) and surface temperature (in blue) of chocolate spheres having an initial size of 3 mm heated through forced convection (Tair= 38°C velocityair = 0.4 m/s)

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MEMORIAL SESSION. ROLF K. ECKHOFF

KEYNOTE LECTURES

Lessons learned from testing of dust explosion protective devices and systems

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Keywords | Dust explosions, protection, venting, isolation

To protect industry from the consequences of dust explosions several protective systems and devices have been developed especially during the last 50 years. These systems include passive devices such as vent panels (including flameless venting devices), self-closing valves including explosion isolation flap valves and diverters, and active systems such as explosion suppression systems, fast-acting valves and extinguishing barriers which are activated upon detection of a dust explosion. Assurance of optimized functionality, explosion resistance and where necessary capability for preventing flames to transmit is established through the ATEX directive 2014/34/EU. This directive has been transposed into national law within the EU and associated countries and demands approval of these systems and devices by so-called notified bodies. The approval in most cases implies testing of the systems according to standards developed for this purpose. The testing has revealed some interesting phenomena which will lead to improved testing procedures to be implemented in some of the standards and also improved guidance with respect to design of dust and powder handling facilities. In this paper two such phenomena will be highlighted and discussed. The first phenomenon concerns the strong increase of reduced overpressures seen during vented dust explosions in vessels when these vessels are connected to ducts in which an explosion isolation device is installed and activated. The overpressures seen can exceed those predicted by guidelines to size explosion vents by a factor of up to 4.

The strong pressure increases are seen when the valve closes.

The second phenomenon concerns strong pressure oscillations occurring in ducts connected downstream of explosion isolation flap valves upon the closing of these valves, potentially causing these valves to open again (allowing for flames to transmit) and exposing the valves to much higher net pressure loads. This can be seen from the pressure-time histories measured in front and directly behind an explosion isolation flap valve when tested with a duct mounted on the downstream side of the valve.

The paper explains the phenomena and discusses necessary remedies.

Strength of knowledge in risk assessments for dust explosions

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Keywords | Dust explosion; Risk assessment; Strength of knowledge

Abstract

Accidental dust explosions pose a threat to people and property in industries that produce, process, handle, or transport combustible dusts (Eckhoff, 2003). Although there has been significant development in the technologies, methods and standards for safe design and operation of process facilities over the last century, severe dust explosion accidents continue to cause severe losses in the process industry (Eckhoff, 2020). Whereas conventional approaches to risk analysis treat risk as an expectation value, there is increasing awareness of the importance of uncertainty (Aven, 2010). To this end, Skjold (2018) reviewed some of the practical aspects associated with risk assessments and risk management for dust explosions in the process industries, with emphasis on consequence modelling.

The present study extends the analysis from Skjold (2018) by exploring the concept of strength of knowledge (SoK) in risk assessments for systems where dust explosions in complex geometries represent a significant hazard. In its simplest formulation (Aven, 2013), the SoK is considered 'weak' if one or more of the following conditions are true: 1) the assumptions made represent strong simplifications; 2) data

are not available, or are unreliable; 3) there is lack of agreement or consensus among experts; and 4) the phenomena involved are not well understood, or models are non-existent or known/believed to give poor predictions. The discussion emphases the importance of aspects such as risk awareness and the inherent limitations in the predictive capabilities of consequence models for dust explosions in complex systems.

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ORAL COMMUNICATIONS

Influence of bends in the functionality of passive explosion isolation valves

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Keywords | Dust explosion, explosion mitigation, explosion isolation

Passive explosion isolation valves are widely used in dusty industrial processes to isolate explosions.

They prevent the propagation of flame and pressure to the rest of the installation, mitigating the consequences of an explosion. However, the valves only work under specific conditions, defined by the manufacturer as intended use. One of these conditions is the installation of bends between the protected equipment and the isolation valve, since it can generate turbulence and pressure drops that can influence the speed of flame propagation and the pressure wave.

This paper presents the methodology and findings of the Adix explosion tests.

Explosion tests have been carried out in a 2m3 vessel together with a DN 500mm straight pipe, as well as tests introducing 3 three 90º bends. These configurations have been tested with low and high kst fuels.

As a result, it is shown correlations between Kst, the presence of curves, flame propagation speed, and pressure wave propagation.

Reviewing Particle Size Influence in Biomass flammability and Explosibility

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Keywords | Biomass flammability, Particle size distribution, explosivity, dust explosions

Due to the new policies implemented to mitigate and reduce climate change effects, the use of biomass has significantly increased in the last years, and so, the number of accidents related to biomass storage, use, transport, and handle. Several authors have focused their research on biomass flammability and explosion severity properties, together with the influence of chemical and physical characteristics, finding out that particle size (PS) presents a major effect on those properties (Eckhoff, 2009; Guo et al., 2012). In order to increase biomass safety knowledge, PS has been widely studied, however, most of the published research determines PS using common methods that do not properly define this parameter. As biomass presents elongated and fibrous shape, granulometry methods that approach particles to spheres do not properly characterize biomass particles, which leads to misunderstandings when assessing relationship between granulometry and flammability properties (Gil et al., 2014). The aim of this study is to collect published information regarding biomass granulometry and flammability so assessment can be carried out. Indeed, it was found out that most of the published research determines PS using laser diffraction whose error when testing fibrous particles is quite significant. The obtained results were compared to coal samples, as their

characteristics are further studied. From data, it was noticed that biomass samples present a wide particle size range and, even when considering biomass with similar particle size distributions, important deviations were found when assessing flammability and explosibility properties. Furthermore, it was clear that there is no standard method for particle size determination which can unify results in order to carry out comparisons between samples. Moreover, the public existing data regarding biomass industrial safety is not extensive and should be increased to help understand biomass behavior.

MEMORIAL SESSION. UĞUR TÜZÜN

VS

Dynamically structured fluidized beds: Scalable, homogeneous, and responsive mixing

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Keywords | Fluidization; Intensification; Mixing; Pulsation; CFD-DEM

Dynamically structured fluidized beds use vibration and/or pulsation of the inlet gas flow to create an ordered flow characterized by the organization of gas bubbles into a triangular lattice, where they maintain a constant size and separation. Dynamic structuring was discovered over two decades ago but its relation to the applied perturbation has only been clarified recently. In this work, we explore its potential to intensify particle manufacturing operations. We investigate the stability of pulsation-induced structured beds and the associated characteristic contact pattern, solid mixing and stresses. We report bubble dynamics and local solid velocity fields, Fig. 1a, gathered experimentally in (quasi-2D) beds of Geldart B particles at various oscillating frequencies, and we replicate their behavior via CFD-DEM. High-speed visualization and analysis of the dispersion of a tracer in CFD-DEM simulations reveal important and unique qualitative features. The macroscopic mixing of the solids in a structured bed is slower than in traditional units, and dispersion is no longer diffusive. Mixing is driven by the advection of solids across well-defined mixing regions or compartments, Fig. 1b, that appear in response to a stable ordered bubble motion. As a result, micro-mixing in the solid phase, associated to vertical velocity fluctuations within each region, is decoupled from macro-mixing established through advection across neighboring mixing regions. Such a compartmentalization offers three advantages: homogeneity, scalability, and control. In contrast to the broad range of time and spatial scales involved in a traditional bed, mixing results from multiple repetitions of the exact same flow structure, Fig. 1c, thus exposing the solids to a narrow stress history and a homogeneous gas-solid contact pattern. This flow structure is reproducible in a broad range of particle sizes to any bed width and heights up to at least 20 cm, and, critically, it is responsive to external actuation. Modulating changes in gas velocity, one can tailor the bubble size and separation and therefore control the size of the mixing compartments.

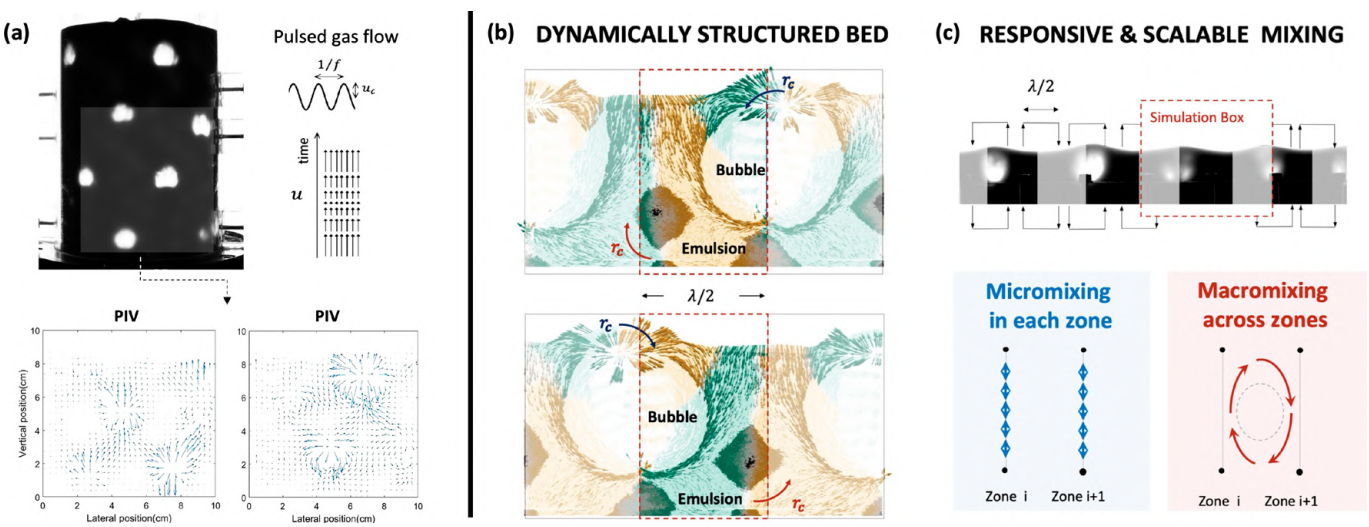


Figure 1 - Dynamically structured, pulsed fluidized bed. (a) Structured bed and local PIV detail. Example of (b) CFD-DEM simulations and (c) solid mixing patterns in a 2D bed.

A contribution to visualization of industrial processes by Electrical Tomography

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Keywords | Electrical Tomography, Industrial Processes, Suspensions flow

Understanding the behaviour of suspension flows continues to be a subject of great interest considering its industrial relevance, regardless of the long time and effort dedicated to it by the scientific and industrial community. Information about several flow characteristics such as, flow regimen, relative velocity between phases, or spatial distribution of the phases, is essential for the development of exact models describing the processes involving suspensions' flow. Pedro Faia, Maria Rasteiro and Uğur Tüzün, together, integrated a team that edited a Special Issue of Applied Sciences dedicated to those issues, named "On Visualization and Simulation of Microstructural Dynamics of Complex Fluid–Particle Systems: Process Industry Applications".

Figure 1.: i) ET adjacent injection and measurement protocol for the first (A) and second (B) projections (Reproduced with permission from Malmivuo et al. [1]); ii) ET images obtained with a concentration of 1% (w/w) of Pine fibres, a), b), c) and d) respectively for 0.5 m/s, 1m/s, 1.5m/s and 2.5m/s [2].

Among the contributions, one was dedicated to the usage of a non-invasive technique that allows to obtain experimental data about suspension flow in different processes, Electrical Tomography, ET [2]. ET presents perhaps the best compromise between cost, portability and, above all, safety of handling. A brief review and comparison between existing technologies for pulp suspensions flow monitoring was presented, together with their strengths and weaknesses. Emphasis was given to ET, not only because it offers the above-mentioned compromise and also because it was the strategy adopted by the authors to characterise different flow processes (solid-liquid, liquid-liquid, fibres-liquid, etc). The produced portable ET system was described, and examples of results of its use for pulp suspensions' flow characterisation were reported and discussed.

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Granular Flow in Hoppers: from Theory to Practice

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How to develop a general theory to describe the granular flow in hoppers under different conditions is a long-standing topic in particle science and technology. Current practice is still largely based on the classical work of Jenike. Here we report an alternative which is based on a continuum theory for granular flow, in memory of Professor U Tuzun's contribution in this area.

The theory considers the elasticity, frictional plasticity and flow rheology of granular materials. Its governing equations, closed by suitable constitutive relations, can be solved by advanced finite element model (FEM) to generate numerical solutions under different conditions that can be reflected by material properties and boundary conditions. The outcomes can therefore be used to aid the design, control and optimization of a bulk solids handling process. The success of this Computer Aided Technology (CAT) is verified by the facts: 1) the model can be used to simulate the dynamics of particles in a hopper;

2) it can reproduce the mass/funnel flow patterns proposed by Jenike, and 3) it can well describe the ratholing phenomena of highly cohesive particles. Moreover, the mass flow rate predicted not only conforms with the Beverloo equation but also agrees with the discrete element method (DEM) simulations when the DEM (micro) and FEM (macro) parameters are linked using a shear cell test. By combining with the Genetic/Gradient Decline algorithm, the CAT can generate some optimally curved hopper shapes that can almost double the mass discharge rate of conventional conical hopper. The so designed hopper is confirmed by experiments. The application to hoppers is just an example from theory to practice. The proposed CAT is promising in solving design and control problems associated with different granular processes.

A novel model for liquid bridges and coefficient of restitution in wet fluidisation

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Keywords | wet fluidisation; liquid bridges, coefficient of restitution

Adding small amounts of liquid into gas fluidized beds is a common operation in industry. For instance, in fluidised catalytic cracking reactors, the sprayed droplets introduced through atomisers may spread on the surface of hot catalyst particles and vaporise to enhance the cracking efficiency. The addition of liquid can give rise to strong cohesion between particles, imposing a marked impact on fluidisation.

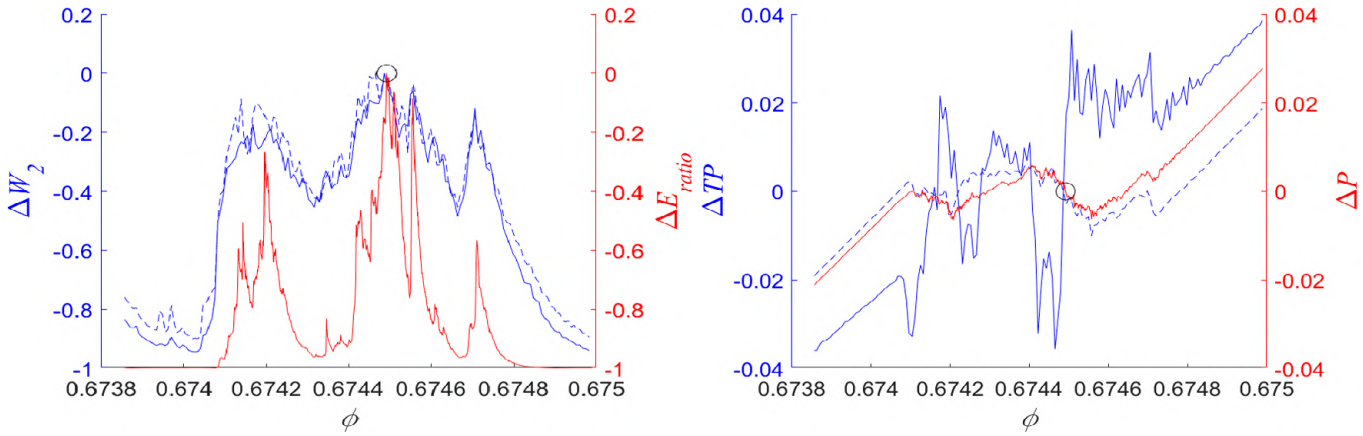
In this paper we present a novel model framework for liquid-bridge force and coefficient of restitution to improve CFD-DEM simulation of wet particle fluidisation. We integrated a temporal-scale criterion into the original spatial-scale threshold for liquid bridge force. Thus, a liquid bridge is allowed to form and remains stable when the normal relative velocity of two particles is lower than a critical value, in addition to the critical distance criterion. The liquid bridge force increases with vnc. The comparison between the predicted bubble behaviour and the measurements strongly indicates that there exists a critical particle velocity to impact lifetime of liquid bridge. With the new model, the predictions of bubble properties including bubble centre, shape, and volume agree well with the measurements reported in the literature. Furthermore, using more than 800 data points from the experiments in literature, an artificial neuronal network (ANN) tool is developed to estimate the coefficient of restitution for wet particles. Capillary, Reynolds, Stokes, Bond, and Weber numbers can be used as AI model inputs. The new model framework is expected to enhance the modeling and simulation in the processes of fluidised bed conversion.

Understanding slow compression and decompression of frictionless/frictional soft granular matter by network analysis

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Keywords | Discrete Element Method, Persistence analysis, Slow deformation, Force networks

We consider dense frictionless and frictional granular systems in three spatial dimensions exposed to slow compression and decompression, below, during, above and well above jamming. The evolution of granular systems under slow deformation is non-trivial and involves smooth, continuous, reversible (de)compression periods, interrupted by fast, discontinuous, irreversible transition events. These events are often, but not always, associated with rearrangements of particles and of the contact network. How many particles are involved in these transitions between two states can range from a few to almost all in the system. An analysis of the force network that is built on top of the contact network is carried out using the tools of persistent homology. Results involve the observation that kinetic energy is correlated with the intensity of rearrangements (Wasserstein distance, W), see Fig1.a, while the evolution of global mechanical measures, such as pressure, is strongly correlated with the evolution of the topological measures quantifying loops (total persistence, TP), in the force network, see Fig.1b. Surprisingly, some transitions are clearly detected by persistent homology even though motion/rearrangement of particles is much weaker, i.e., much harder to detect or, in some cases, not observed at all. Future work is devoted to investigating the evolution of granular systems under slow deformation prepared with cohesive and non-spherical particles.



(a) (b)

Fig1: (a) $/Delta_{W\_2}$ distances both for $/beta_0$ and $/beta_1$ as well as relative change of kinetic $/Delta$ energy E_ratio, and the arrow is pointing to the reference value used for normalization. (b) Normalized values for the pressure change $/Delta_{p}$, $/Delta_{TP0}$ and $/Delta_{TP1}$; all shown quantities are normalized by their value at the centre of the event; for example, pressure is plotted as $/Delta_{p}$ = (p - p*)/p*, where p* is the reference value shown by $/bigcirc$. Here $/Delta_{TP0}$ and $/Delta_{TP1}$ are total persistence for components and loops, and $/Delta_{W\_2}$ is the distance, see [1] for details.

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Effect of Electro-Clamping Force on Enhancing the Resistance to Shear Deformation of Granular Materials

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Keywords | electro-clamping, cohesion, electrical, granular, flow, control

Application of an electric field to a granular medium produces electro-clamping forces at contact points. By designing a suitable electrode systems, this mechanism was used as an actuating mechanism for flow control of granular materials (Ghadiri and Clift, 1986). However, the response of the granular medium to the electric field produced an intriguing pattern, i.e. the flow rate did not initially change significantly with increasing the electric field strength until a certain field strength was reached, beyond which it rapidly declined (Ghadiri et al., 1992). To analyse this trend, a novel translational shear cell equipped with a set of electrodes was developed in collaboration with the late Professor Tüzün. Bulk cohesion increased with the field strength, but interestingly, the internal angle of friction remained constant. The analysis of the experimental results also showed that the models of electro-clamping force did not reliably predict the magnitude of the generated force (Martin et al., 1991). The novel measurement of the single contact electro-clamping force showed consistency with the bulk cohesion trends, but differed from the predictions of the electrical clamping models (Ghadiri et al., 2006). This was attributed to the inadequacy of the models in accounting for contact deformation. Recent work on the analysis of the rheology of cohesive powders sheds some light, as it is shown that the shear resistance to flow increases only when Granular Bond Number exceeds a value around 10. The prediction of cohesive powder flow is still very challenging, requiring further work to develop a reliable rheological model of flow influenced by an electric field.

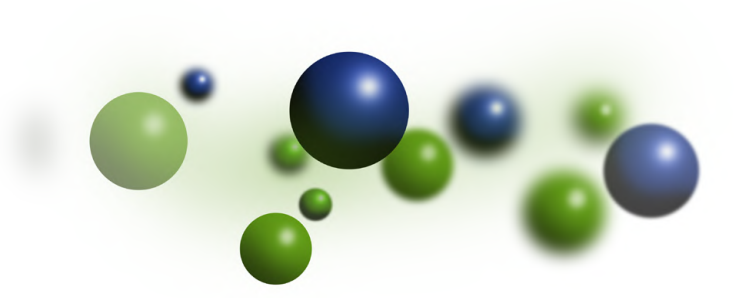
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POSTERS



1 Particulate solids handling

Concept for the simultaneous dust release and separation by means of electrostatically assisted spray nozzle systems

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Keywords | Dustiness, Dust Separation, Wet Scrubbing, Water Spray, Electrostatic

The use of various raw materials in the form of bulk materials takes place in the building materials industry, agriculture and the food industry, among others. Bulk material handling, transport and storage result in the particle emissions, referred to as dusting.

The aim of the project is to demonstrate the effectiveness of the dust reduction measure “use of spray nozzles” in combination with the effect of electrostatic charging. The special feature here is that the investigations are oriented to the type of stress and the material and thus take into account the character of the dust emissions. Different test set-ups are used to realistically illustrate the handling operations during which dust is released. The effectiveness of the reduction measure is investigated using different operating parameters for dust emission as well as separation. The subsequent electrostatic charging of the spray mist allows the use of water and compressed air to be reduced. An evaluation method for spray nozzle systems is being developed, which considers the economics of the used resources and the material-related efficiency of the reduction.

First of all, planning work has to be carried out to choose the test materials and suitable test set-ups. Subsequently, suitable test parameters are specified. The previously selected bulk materials are characterized regarding their material properties and tendency to dust. Furthermore, different material handling set-ups are tested in a wind tunnel. The experimental verification of the dust reduction measure with and without the use of an electrostatic influence on the water spray mist then takes place. The measure is quantified by mass balances using the dust concentration with and without use of the spray nozzles. Based on the test results an evaluation method for the dust reduction measure is developed. The special focus is on the interdependence of resources used (water, compressed air, power flow and equipment complexity) and the separation efficiency of the particle fractions PM2.5 and PM1.

Capillary Water Transport in Biomass Pellets and its Influence on Structure and Stability

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Keywords | Biomass Pellets, Water Uptake, Swelling, Storage

Constantly rising prices for heating oil and natural gas in households and their environmental impact lead to a rising interest in renewable raw materials. Wood pellets are small cylindrical compacts from natural wood, primarily from sawdust and wood shavings. They are used as a versatile biofuel with a significant lower emission of the climate-relevant carbon dioxide compared to fossil fuels. These pellets can be burned using a variety of technologies, such as home heating stoves or institutional boiler systems.

To ensure the burning efficiency of the pellets, their quality must remain constant during preceding processes such as transport and storage. However, an increased water content due to storage under conditions with high humidity can lead to a reduction in quality, e.g. by swelling and a decrease of their mechanical strength. Together with the stresses and friction caused by the subsequent transport, an undesirably high level of fines is produced, which can block the conveying systems. According to DIN EN ISO 17225-2, a critical water content of 10 wt.-% should therefore not be exceeded. When considering storage processes, there is a limit of investigations on the capillary water transport in wood pellets so far.

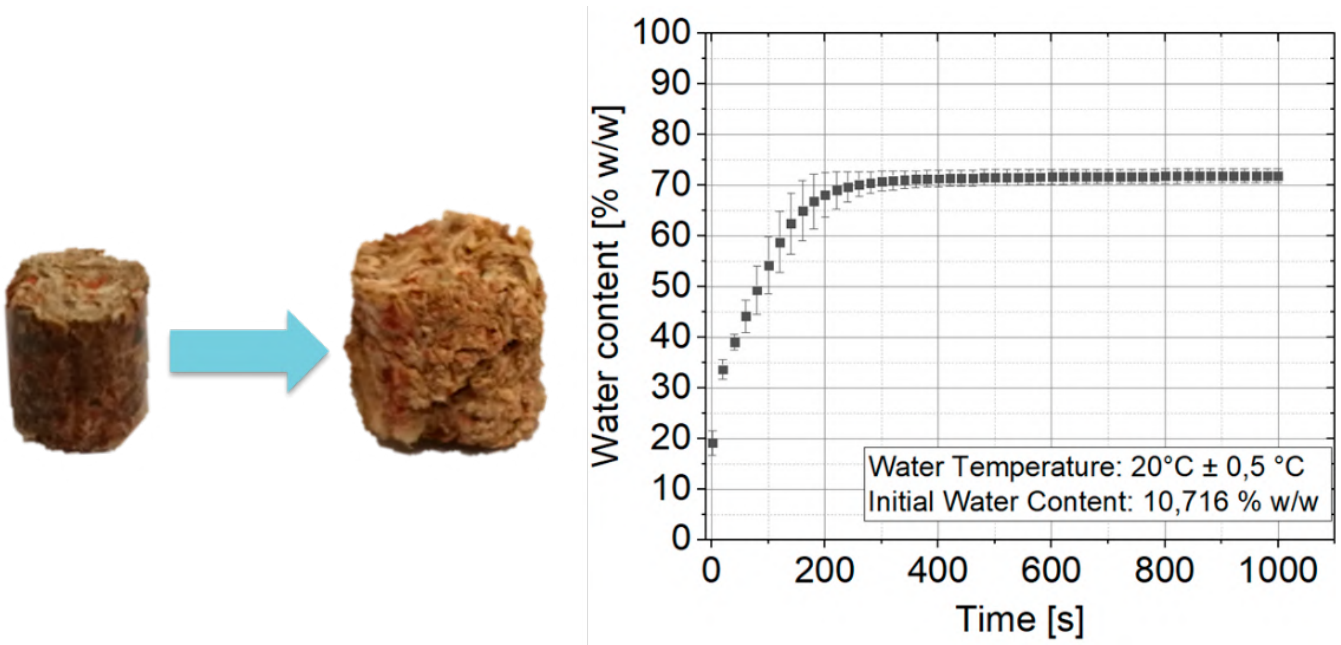


FIGURE 1. Example of the water uptake over time in wood pellets (right) and swelling of pellets (left).

Exceeding the saturation vapor pressure during a long period of storage time can lead to condensed water, which come into contact with wood pellets. The capillary properties of wood pellets are investigated by varying their water content, porosity and the temperature of the adsorbed water. Thus, it could be shown that the water uptake rate varies for pellets of different wood species and different water content. In addition, the water uptake rate increases with increasing porosity of the pellets. The water uptake in the pellets is reflected in a strong swelling (FIG. 1), a reduction in mechanical stability and consequently an increase of the fine content.

Acknowledgment: The German Federation of Industrial Research Association (AiF). AiF Project 20565 N/2

Estimate the impact of extent of lubrication on downstream tablet properties.

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Keywords | Lubrication, Tensile strength, Porosity, Tablet, Model

Lubricants are significant tablet excipients that are included in pharmaceutical formulations. The extent of lubrication plays an important role during the downstream compaction processes and in the overall performance of the drug products. Magnesium stearate is a commonly utilised lubricant in the pharmaceutical industry. Although it helps the compaction process by lowering wall friction during tablet press ejection, minimising the potential for the pharmaceutical formulation to stick to exposed metal surfaces during powder compaction, enhancing powder flow, and increasing bulk powder density. Overmixing during lubrication, on the other hand, diminishes tablet hardness, lengthens disintegration time, and slows dissolving rates. Since these effects are impacted by formulation type and process dependent factors, it is critical to understand their interdependence.

In this work, the impact of the extent of lubrication on downstream tablet properties such as tensile strength and porosity has been investigated. Using a Bin blender, a systematic batch-wise experimental study was carried out. We examined the effects of different formulations and degree of lubrication on tablet characteristics. Additionally, lubrication process dependent parameters such lubrication blending duration and speed, as well as downstream compaction pressure during tableting, are examined for each formulation. The interplay between formulation and process conditions has been shown to have a considerable effect on tablet characteristics. We compare our experimental findings with the model prediction by extending the previously proposed model by Nassar, J et.al., 2021 for a wide range of formulations and lubrication process parameters. The quality of correlation between predicted and experimental tablet strength and solid fraction is analysed and presented.

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Sensitivity analyses of binary mixture parameters on blast furnace segregation and bed permeability

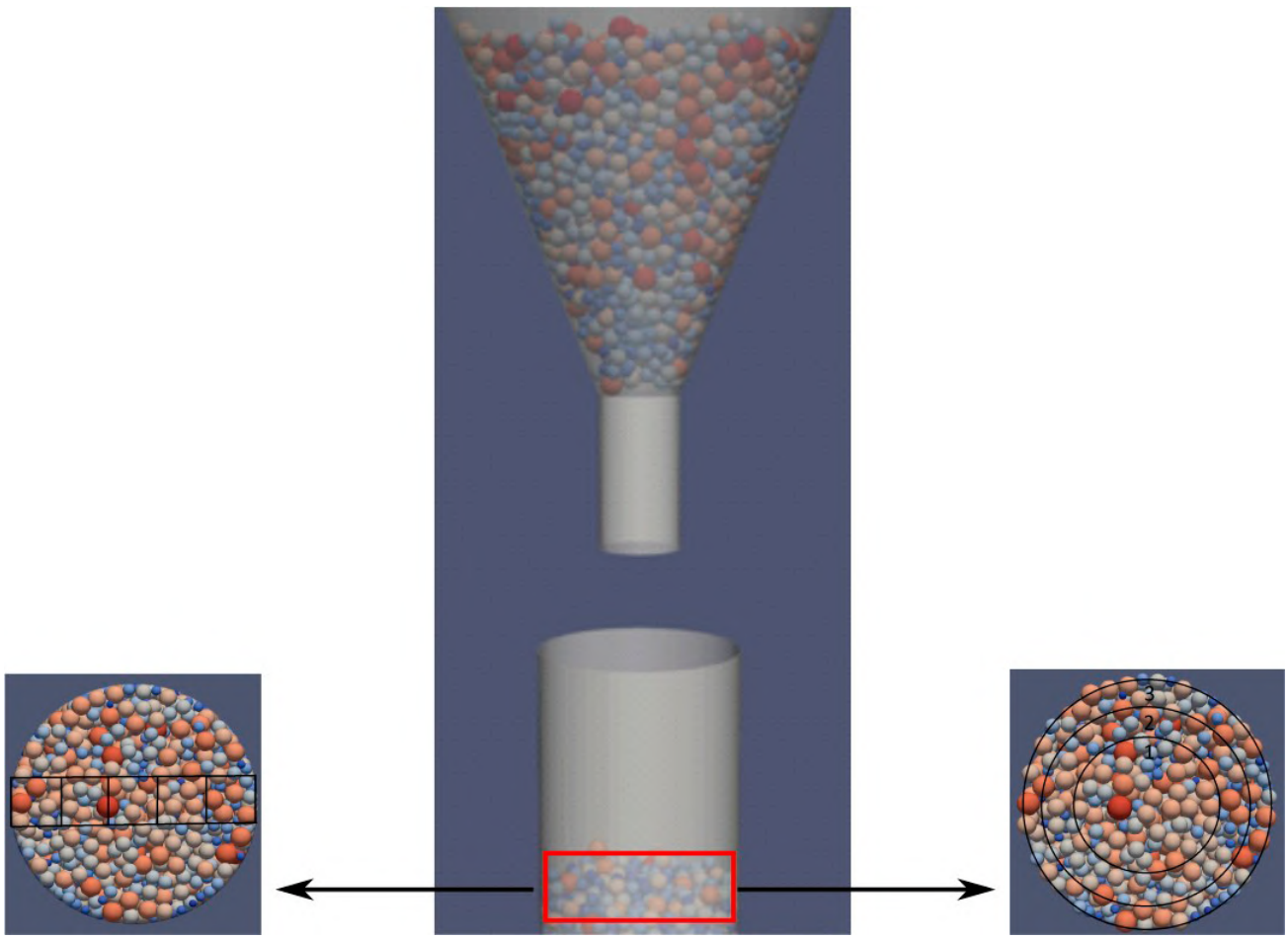
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Keywords | Charging, segregation, permeability, DEM

A blast furnace is a counter-current reactor which produces liquid iron through a series of chemical reactions occurring between ascending hot reduction gas, which is injected through nozzles at the bottom of the furnace, and a descending packed bed of raw materials (pellet, sinter, lump ore and coke, collectively referred to as the “burden”) which are charged at the furnace top. Bed permeability is a crucial factor in blast furnace efficiency and stability and depends on the structure of the packed bed and therefore, the material distribution which is achieved through charging. Due to the differences in raw material properties, segregation is expected to occur during furnace charging. It is therefore of importance to investigate segregation during the charging process and to assess its effect on the permeability.

In this work, the Discrete Element Method (DEM) is used to model the raw material behavior using a simplified discharging setup. Since we are dealing with a mixture, a relatively large number of model parameters must be determined through calibration. As a first step, we consider a binary mixture of pellet and sinter and perform sensitivity analyses to determine the importance of different mixture interaction parameters (coefficient of sliding friction, coefficient of rolling friction and restitution coefficient) on the degree of segregation and permeability of the packed bed of materials after being discharged. This allows us to determine which parameters can be excluded from the timely calibration process.

We perform sensitivity analysis for the effects of individual factors and factor combinations by employing analysis of variance (ANOVA) in conjunction with factorial experimental design. The segregation and permeability analysis are performed in different directions and using different virtual volumes. Our initial results show that all three interaction parameters are important when considering both segregation and permeability. Further research will be performed to rank the importance of the parameters.



Research on multiphase fluidized bed separation technology for waste mixtures

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Keywords | powder, mechanics of particulate solids, separation, particle shape, particle size, bulk densities

The treatment and recycling of waste materials from various industries is an increasingly hot topic worldwide. Research is focused on the separation of the different components of waste bulk mixtures. Waste mixtures such as electrical wire pulp or a mixture of contaminated abrasive material for the industrial surface blasting process contain large amounts of material components such as copper or abrasive, which have a high market value in their pure state. From this point of view, commercial customers require maximum purity of these material components with a minimum of impurities as well as maximal effectivity during their recycling and re-use. Separation in fluidized bed takes advantage of the different properties of the individual components of the bulk mixture, such as particle shape, particle size or different bulk densities. The first stage of the technology aims to a fluidized sieve separator, where large particulate impurities and other undesirable elements are separated from the mixture. The second and subsequent stages of separation are carried out on a vibrating fluidised bed with multi-level vacuum outlet pipes going to particle separators. The proposed equipment for multiphase separation of waste bulk materials has proven to be very efficient, especially for the mixture of electrical grit from which it is required to obtain copper wires in maximum purity, free also from the dust fraction. The separation efficiency is very sensitive to the optimum setting of the operating conditions of all functional separation stages, which must be tuned individually for each waste mixture to be separated. The advantage of the proposed technology compared to another solutions is seen mainly in the simplicity of the technological unit, which can also be significantly reflected in lower production costs compared to competing solutions.

Optimisation of Ball Indentation flowability measurement technique: bed preparation and indentation conditions

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Keywords | Powder Flowability, Low-stress Techniques, Ball Indentation Method

Optimisation of Ball Indentation flowability measurement technique: bed preparation and indentation conditions

The ball indentation method has been developed recently and showed reliable results for assessing powder flow behaviour under low and high-stress levels using a very small amount of material (few milligrams) (Hassanpour, A., & Ghadiri, M., 2007). In the ball indentation test, the powder sample is first consolidated to the desired stress and is then penetrated by a spherical indenter to a specific depth. The indenter is finally unloaded at the same loading speed. Powder flowability is determined by calculating the bed hardness, resistance to plastic deformation, from the maximum applied force and the projected area. Using the bed hardness, and the pre-established constraint factor of the powder being examined, the unconfined yield stress can then be determined.

The technique was adopted by several researchers where some studies analysed the operation window of the technique and it is being utilised to infer the unconfined yield stress of cohesive powders at low stresses, however, bed preparation and filling methods could be further optimised in order to achieve a uniform packing state at low stresses.

In this work, the filling and bed preparation methods were optimised using titanium dioxide powder, in an attempt to achieve a uniform bed packing. Two sieve filling approaches were adopted: partially-filled and fully-filled beds. Additionally, three different indentation parameters were investigated, where the effect of penetration depth, indentation position and indenter radius on the inferred unconfined yield stress was studied. In addition, X-Ray tomography scans were carried out for several samples to investigate the bulk density around the indention zone, before and after compression and indentation.

Results showed that the fully-filled beds provide uniform packing density and stress distribution, in contrast to partially filled beds (Figure 1). Also, no significant effect on inferred unconfined yield stress is observed on using different indenter sizes for the fully-filled beds.

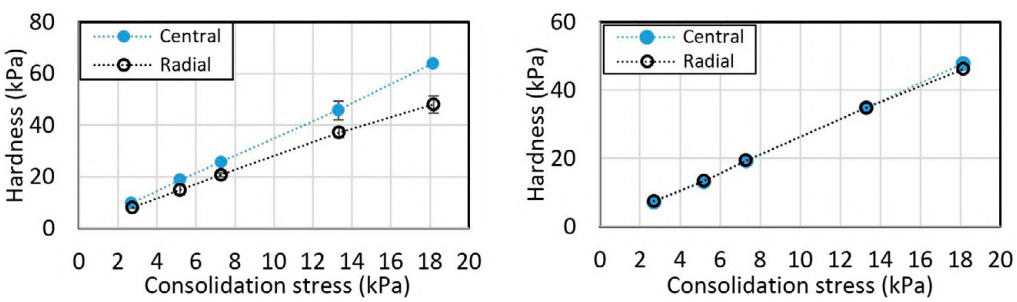


Figure 1. (a) partially filled bed (b) fully filled bed References

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An aerated virtual Couette powder rheometer: A CFD-DEM analysis

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Keywords | Granular rheology; Intermediate flow; Couette Flow; CFD-DEM

Despite the industrial relevance of rapid granular flows, powder flow characterization is often restricted to flow initiation.Few devices can operate within an intermediate regime and flowability data are often comparative in nature.Experimental measurements are hard to transport across shear cells due to inherent assumptions regarding their internal flow and difficulties in maintaining comparable wall conditions and consolidation. Couette-type testers were introduced decades ago as a simple alternative, but earlier units suffered from a

poor slipping wall contacts and poor reproducibility. Modern devices, termed aerated Virtual-Couette rheometer VCR address the problem substituting the inner cylinder by an impeller such that the powder rotating within acts as the shearing wall. The cell can operate at a wide range of inertial numbers using gravity to compact the powder and aeration to reduce consolidation, Fig 1a.Although the axial distribution of normal stress is often considered hydrostatic, recent experimental work suggests that wall effects can create secondary flows akin to a Taylor vortex. Bulk measurements however show excellent reproducibility studying the rheology of glass beads, and a good agreement with μ-I models. Furthermore, the simplicity of the geometry leads to a characteristic radial position that makes rheological measurements comparable at any scale, thus improving standardization. In this work, we describe for the first time the dynamics of an aerated VCR through CFD-DEM simulations. Computing the macroscopic Eulerian variables from the Lagrangian data allows us to describe the formation and extension of a well-defined shearing region, Figs 1b & 1c.The full- scale model is validated with experimental measurements, and we report the local flow pattern and stress distributions, identifying characteristic structural changes associated to the departure from a quasi-static into intermediate flow with increasing aeration.This digital twin will now be used to incorporate cohesive particle-particle forces and study the interplay of Bond number and aeration in the rheology of cohesive mixtures under very low consolidation with the intention of linking advanced rheological models with bulk characterization techniques.

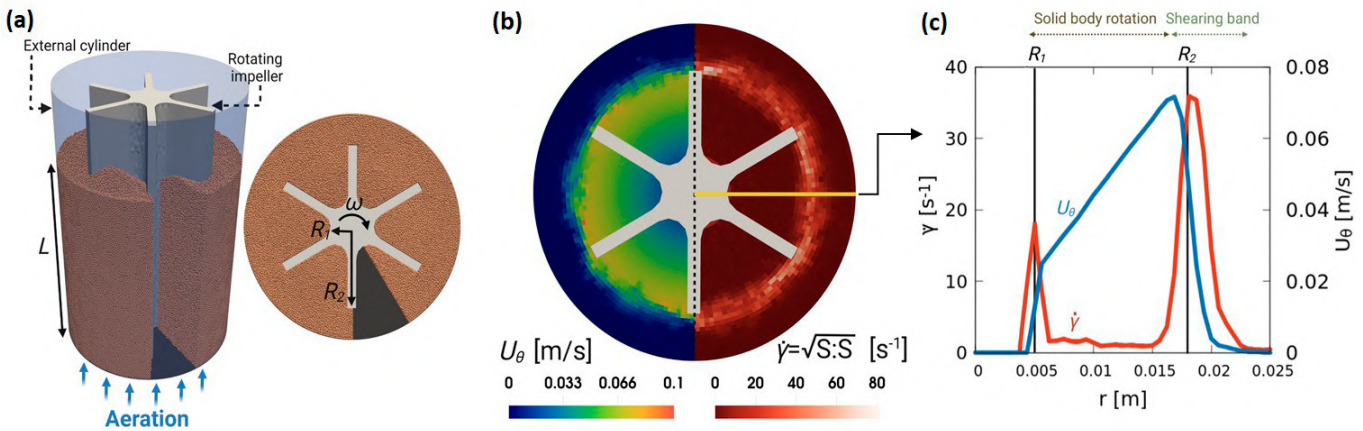


Fig. 1 - (a) An aerated VCR, (b) and (c) CFD-DEM simulations showing averaged azimuthal velocity DD, and local shear rate Ḋ , where S is the coarse-grained strain rate tensor.

2 PARTICLE AND PARTICULATE SYSTEMS CHARACTERIZATION

Effect of Particle Shape on Void-Fraction and Flowability

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Keywords | Sphericity, Void Fraction, Density, Flowability

Recent papers by the author presented a thorough analysis of particle free and tapped bulk densities or void fractions and also the effect of moisture content and various mixtures. The main thing is that it was shown that Archimedes number is appropriate to describe the behavior and therefore to include measurements conducted in various liquids. However, all the above are a function of the particle shape, which is the aim of this investigation. By conducting tests with many materials, some having various sizes the behavior as a function of the particle sphericity became clear. The experiments included, spheres, cylinders, disks and flakes, irregular shapes and other specific shapes. The range of tested Archimedes number was between 1 and 10^8. It was found that the normalized value of the Void fraction (normalized by the spheres data) can be nicely presented for all material by a shape factor. However, for angle of repose and tilting a critical Ar was found (minimum angle value). For decreasing Ar below the critical value, the angle value was increased in a similar matter, although at higher values, as the spherical particles. For increasing values beyond the critical Ar value, the angle was increased, while for the spherical particles it was stabilized.

Influence of moisture content on the mechanical properties of three granular materials usually stored in agricultural silos

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(1) University of Extremadura

Keywords | wood pellets, wheat, corn, moisture content, angle of internal friction, dilatancy

Traditionally, the design of agricultural silos has been carried out following classical design theories. Among them, Janssen's theory is the most used throughout the world. These theories are based on the determination of several mechanical properties of the materials stored within them, such as the angle of internal friction or the bulk density among many others. These properties determine the loads generated under static conditions. For many granular agricultural materials these properties have been determined for years. However, under dynamic conditions, loads differ from those corresponding to the static ones. In these cases, numerical methods are quite useful. With these techniques, additional parameters must be considered apart those applied in the classical theories. Although it is known that the values of all these parameters strongly depend on the moisture content, just few studies have been conducted up to date to determine the variation experimented by granular materials when moisture content is varied. For this reason, the main goal of this research is to determine the variation of the values of some of these parameters, such as the angle of internal friction, the apparent cohesion and the dilatancy angle, when moisture content is varied, too. For this purpose, several direct shear tests have been conducted.

Samples of wheat, corn and wood pellets have been tested using a direct shear test device. Loads of 10, 20, 50, 100, 200 and 300 kPa have been applied in the different tests conducted. Tests were replicated twice. With the help of a climatic chamber five different moisture contents have been applied to the samples of wheat and corn before they were tested, whereas for wood pellets just two different moisture contents could be applied. Preliminary results show that for wheat and corn, the angle of internal friction increased as the moisture content of the samples was increased, too. In addition, for wheat and corn the dilatancy angle reached a maximum value at an intermediate moisture content and it decreased later as moisture content was increased. For wood pellets results must be analyzed for the two moisture contents tested to obtain significant conclusions.

Characterization of the physical and flow properties of non- conventional solids for pneumatic conveying (PHOBARS project)

Quezada Henry, Manuela (1); Leturia, Mikel (1); Daouk, Elias (1); Destoop, Thierry (2); Bonny, Fabrice (2); Flament, Antoine (2); Morin, Mathieu (3); Tebianian, Sina (3); Saleh, Khashayar (1)
(1) Université de Technologie de Compiègne, (2) NEU-JKF Process, (3) IFP Energies Nouvelles

Keywords | Irregular particles, Biomass, Fluidization, Permeability

As a consequence of the present environmental issues, the development of waste recovery processes has increased. Due to the heterogeneity of the raw materials used in these processes (plastics and biomass) in terms of physical properties, these solids are considered as "non-conventional solids" (Fig. 1). These non-conventional solids must be transported efficiently through pneumatic conveying between each step of the process to prevent malfunctions. However, while the transport methods for conventional materials have been widely studied and are well controlled (Cui et al., 2006), there is a pressing need to better understand and model the behavior of non-conventional materials during pneumatic conveying and improve its performance.

This work is framed in the PHOBARS project (Pneumatic Handling Of Bio And Recycled Solids), whose focus is the study of the pneumatic transport of non-conventional solids. Both the system hydrodynamics and the electrostatic phenomena that occurs during transport are considered and the aim is to gain a better understanding of the mechanisms involved in order to describe, control, optimize, and predict their behavior. The project has two axes: the determination of the properties of the materials and the relevant descriptors to characterize the transport, and the multi-scale modelling of the experimental results using different approaches (CFD, MP-PIC, DEM-CFD). Within the first axe, several tests are carried out on ideal solids (various spherical and cylindrical plastic granules) and non-conventional solids (straw, plastic flakes, etc.). Particle size, shape, density and sphericity, among other parameters, are measured for each material using morpho-granulometry and pycnometry techniques. The flow properties of these solids are assessed using lab scale equipment, in order to determine their permeability (74 mm internal diameter) and fluidization (97.5 mm internal diameter) characteristics. The results are compared and analyzed to identify the effect of physical properties and operating conditions on the hydrodynamic behavior of these solids.

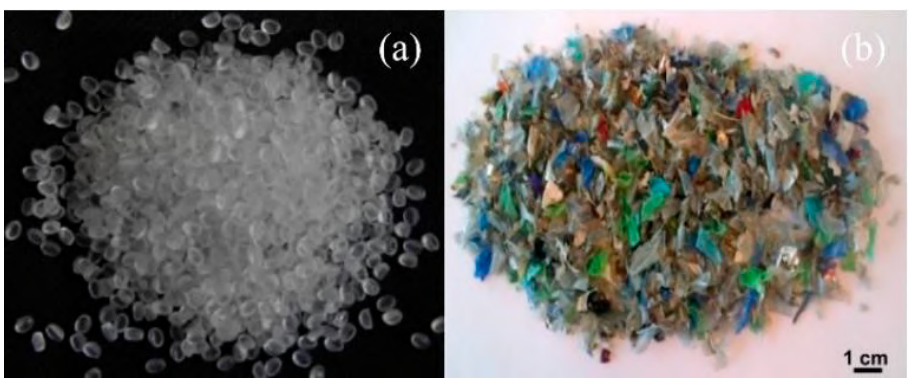


Fig. 1. Example of conventional (a) vs non-conventional (b) plastic solids.

Acknowledgement: The authors acknowledge the support of the Agence Nationale de Recherche (ANR) under contract ANR-21-CE50-0032-01.

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The Use of X-ray Computed Tomography to Quantify Packing and Consolidation Behaviours of Mefenamic acid and D-mannitol

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Keywords | X-ray Computed Tomography powders consolidation packing compaction pharmaceutical tablets tablet manufacture

Successful tablet formation is dependent on physicochemical and mechanical properties of drug powders. The anisotropy of crystalline materials, poor flow and compressibility, cause processing difficulties. The drug:excipient ratio in formulations also impacts their processability and the quality of tablets. Poor flow and consolidation can be overcome by milling and granulation before compaction. The ability to quantify

and control packing and consolidation of formulated powder mixtures is critical for manufacturing high-quality solid-dosage forms. X-ray Computed Tomography (XCT) is one non-destructive method that can be utilised to quantify packing and consolidation.

In this paper, XCT was employed to study the consolidation and internal microstructure of powder beds of mefenamic acid (MA) and d-mannitol (D-M), individually and in a 50:50 mixture before also tabletting. This system exhibits well-known processing issues related to particle cohesion. The powders were scanned after 0, 10, 20 and 30 taps. Furthermore, MA and D-M were compacted in a 5mm die at 205 MPa, then scanned. The density and porosity levels of both powders and tablets were quantified. XCT measurements were related to powder flow and compressibility properties observed at lab-scale.

It was found that D-M possesses superior packing than MA (Hausner ratios (HR) of 1.30 and 1.67 respectively). The 50:50 mixture resulted in better packing (HR:1.24). Segregation of the two in the mixture was possible by application of the Zeiss phase retrieval filter. The data revealed that mixing the two improved the packing of both materials MA (HR:1.26) and D-M (HR:1.20).

The tablets also verify the better consolidation of D-M having an even density distribution, unlike MA, suggesting the potential of a polymorphic transition. This study demonstrates the unique ability to distinguish and quantify two pharmaceutical powders of very similar densities, within a mixture.

Subsequent studies will involve analysis of different powder and tablet compositions. Expanding on the knowledge of how the two compounds influence each other's consolidation behaviour, which can be employed in the manufacture of higher quality dosage forms. The XCT results will also be correlated to the compounds' solid-state and surface properties for understanding the interparticle/intermolecular interactions involved.

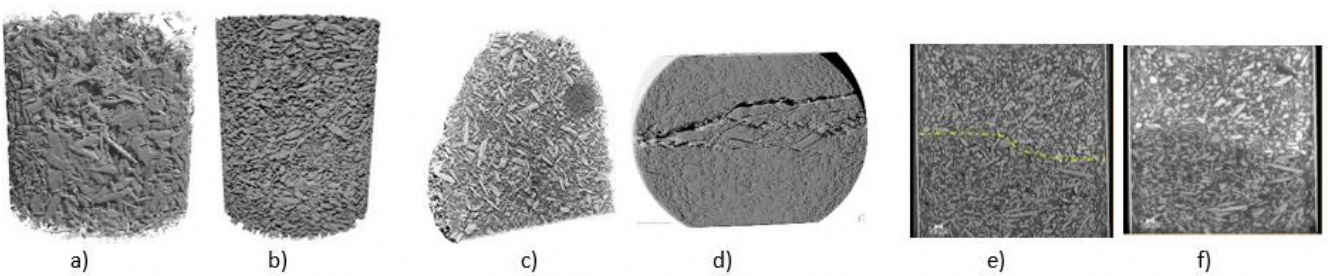


Figure 1. 3-D reconstruction of a) mefenamic acid powder bed at 0 taps, b) d-mannitol powder bed at 0 taps, c) mefenamic acid tablet, d) d-mannitol tablet, e) 2D reconstructed slice of d-mannitol on top of mefenamic acid and f) 2D reconstructed slice of d-mannitol on top of mefenamic acid after application of the Zeiss phase retrieval filter

Auto-agglomeration of dry ibuprofen powder: effect of surface chemistry as a result of recrystallisation

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Keywords | dry API powder, auto-agglomeration, surface composition

Dry Active Pharmaceutical Ingredient (API) auto-agglomeration is a type of unwanted powder transformation that can occur without any additives. Uncontrolled, formation of hard, i.e., capable to withstand drug manufacturing process, API agglomerates may lead to an inadequate API distribution within the final formulation exhibiting the real risk to safety, efficacy and processability of drug products. Despite the key role that the API has in the drug product performance and manufacturing, the number of studies addressing its auto-agglomeration is currently limited and the underlying root-causes remain largely unknown.

The aim of this paper is to explore correlations between particle properties, its processing/handling conditions and auto- agglomeration phenomenon. The experimental work was carried out to test the following hypothesis: API surface chemistry impacts its auto-agglomeration tendency using ibuprofen as the main model API. To create samples with different surface composition ibuprofen was recrystallised from four different solvents, resulting in materials with comparable particle size distributions, but with different particle aspect ratios, hence, with different particle surface chemistry. Auto-agglomeration tendencies of recrystallised ibuprofen batches were tested using mechanical vibration that corresponds to drug handling conditions. The increase in particle size upon mechanical vibration was detected but only in

the case of ibuprofen recrystallised from ethanol and from acetonitrile. Interestingly, the triboelectrification of the samples was found to inversely correlate with ibuprofen auto-agglomeration tendency: the samples that did not auto-agglomerate were shown to gain the greatest tribo-electric charge-per-surface area. This inverse correlation was thought to be linked to the competitive advantage of adhesive forces over cohesive forces when the samples of relatively hight polar component were vibrated. In addition, a novel method (Agglomerates Strength Index) was proposed to assess the strength of auto-agglomerates.

Fullerene and silica nanoparticle diffusivities in air from molecular dynamics simulations

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Keywords | Particles, fullerene, silica, atomistic simulation, molecular dynamics, diffusivity

Molecular dynamics (MD) can provide significant insight into the molecular mechanisms governing major physicochemical processes dominating aerosol dynamics (Mavrantzas & Pratsinis, 2019), which can facilitate process design, process optimization and eventually process innovation (Pratsinis, 2010). Here MD simulations are employed to study the diffusivity of fullerene and silica nanoparticles (NPs) in air and compared with analytical expressions from the kinetic theory of gases or the Stokes-Einstein equation including corrections for the friction coefficient (i.e., the Cunningham correction factor, *C*). The kinetic theory predicts well the diffusion coefficients of small nanoparticles characterized by diameters down to the size of small molecules, while corrected Stokes-Einstein-based correlations bridge the gap of dynamics from the transition to the continuum regime. However, hardly any analytical correlation describes the diffusion coefficients of the tiniest NPs, and discrepancies have been reported between different corrections for the friction coefficient regarding the dependence of diffusivity on NP diameter and/or temperature (Rudyak et al., 2002).

The MD simulations are conducted in the NPT ensemble at practically infinite dilution by employing a fully atomistic model, thoroughly validated for its capability to accurately capture the diffusion coefficient. NP diffusion coefficients are obtained by averaging over many independent simulations to lower statistical noise and narrow the error bar.

The MD simulations show that for the same particle diameter (and within the error bars of the simulation), the diffusion coefficients are the same between the hollow fullerene and the solid silica NPs. The MD results are compared with the predictions of the kinetic theory and the modified Stokes-Einstein relation incorporating the Cunningham slip correction factor for the friction coefficient with numerical constants as proposed by Rader (1990). A new expression for the correction factor *C* for NP diffusivity in the free molecular regime is proposed, while work is extended to investigate the temperature dependence of NP diffusivity.

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Characterization of complex colloidal nanomaterials by means of analytical ultracentrifugation

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Keywords | analytical ultracentrifugation (AUC) InP QDs size-bandgap relation photoluminescence quantum yield

Characterization of complex colloidal nanomaterials by means of analytical ultracentrifugation
Lisa Stiegler, Johannes Walter, Wolfgang Peukert Institute of Particle Technology (LFG), FAU, Germany

Characterization of complex colloidal nanomaterials require a detailed knowledge about the size, shape, composition, functionalization as

well as related optical properties and is the key for understanding of many multiphase products, for instance pigments, pharmaceuticals, cosmetic preparations, coatings and inks. Within our group we already demonstrated that analytical ultracentrifugation (AUC)[1] is a powerful tool to determine particle size, shape, composition and optical properties.

Our recent developments significantly extend the possibilities of AUC for multidimensional property characterization of organic, inorganic as well as hybrid colloidal systems. We will show how AUC coupled with extinction detection is used to study the size-bandgap relation of hybrid InP-ZnSe semiconductor quantum dots (QDs) while coupled with emission detection[2], it can be used to gain information about the size-dependent photoluminescence quantum yield. Such hybrid architectures have a high potential as light-emitting diodes (LEDs) and can be used for novel devices and nanomaterials for bioimaging, display, and lighting.[3] However, ex-situ characterization of size-dependent optical QD properties is a tedious task which involves multiple pre-processing steps and is often limited with respect to sample statistics. AUC can overcome the limitations due to its excellent accuracy and reproducibility and thus provides novel insights into the optoelectronic properties of narrow size-selected fractions.

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Method of measurement & analysis of individual crystal dissolution rate using ultrafast tomography imaging

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(1) University of Chemistry and Technology, Prague, (2) Paul Scherrer Institute

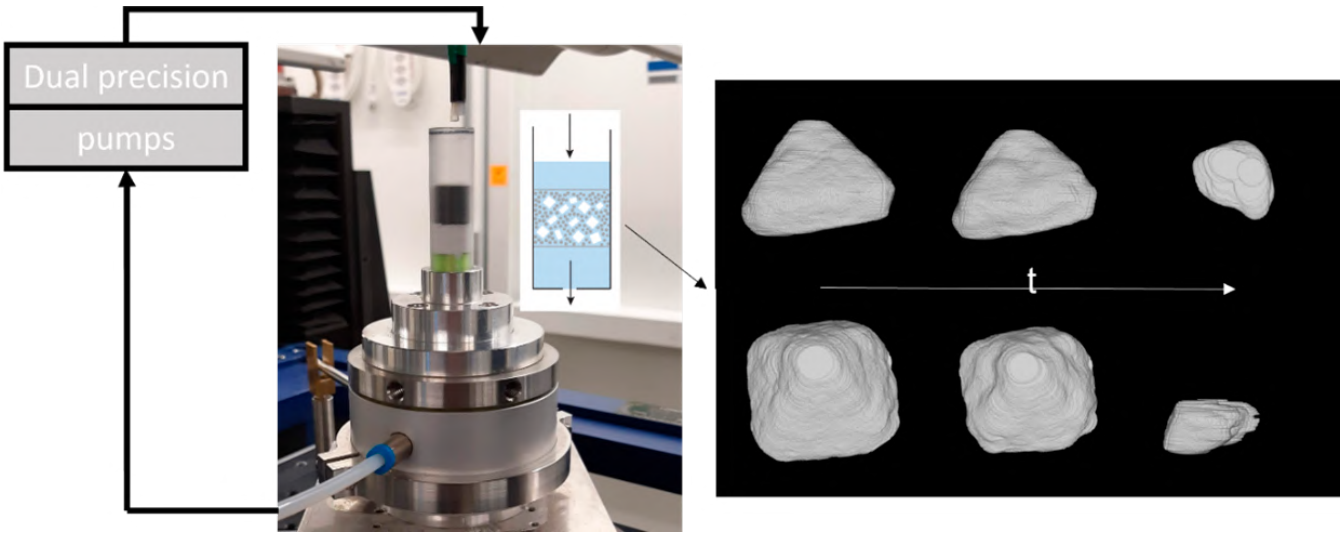
Keywords | ultrafast tomography, dissolution, particle analysis, 3D tracing

Intrinsic dissolution rate (IDR), defined as the amount of drug dissolved per unit time per unit area, is commonly used to describe the dissolution characteristic of APIs (Active Pharmaceutical Ingredients). However, since IDR is measured on compacted pellets, it is inherently inaccurate as the surface changes during the dissolution.

Therefore, a method of measurement of the individual dissolution rate (INDR) of crystals is proposed. By measuring the dissolution rate of each individual crystal and taking the change in volume and surface of the crystals into account, the dissolution rate of the bulk API can be described more accurately compared to the traditional method. Whereas IDR describes dissolution speed per unit area as a single value, INDR describes dissolution speed as a distribution of such speeds.

The experimental setup consisted of linear precision pumps and a flow-thru dissolution cell filled with a mixture of observed soluble salt and insoluble “spacer” powder. The bulk dissolution of particles has been measured under an ultrafast tomograph placed in the PSI synchrotron.

The bulk of particles was scanned via ultrafast tomography and methods for image analysis and 3D particle tracking were developed. Developed methods allowed for tracing of each individual crystal within the observed bulk, calculating its volume and surface as well as its INDR, which can be then calculated for the bulk. Multiple logical and data quality related filters had to be introduced to negate the generation of falsely traced particles and mitigate errors. Data analysis was done via a custom algorithm and obtained information was confirmed via image analysis of the samples measured by ultrafast tomography. The successfully developed method of data analysis allows a more accurate description of dissolution characteristics.



Stability of carbon particles suspended in a high viscosity solution

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(1) University of Birmingham

Keywords | Carbon particles, aqueous solutions, suspensions, dispersing agents, stability

Suspensions of particles in a liquid are widely used in biological, chemical, minerals, materials and medical industry. As a result, there have been numerous studies in the area. However, the stabilisation of hydrophobic particles in an aqueous suspension remains a challenge. Such instability is mainly associated with the aggregation of particles due to interparticle interactions, leading to the formation of large agglomerates and hence phase separation and settling under gravitational actions. Main methods to resolve this problem can be divided into physical and chemical categories. An example of the physical methods is the use of ultrasonic treatment, which, although has been widely used in both laboratory and industrial settings, has limitations due to the limited reflection of ultrasonic wave at the gas-liquid interface was limited. Another example of the physical methods is the use of plasma and irradiation technology. Such a method has led to improved dispersion of plasma-treated particles. An example of the chemical methods is the application of surfactants and dispersants. Many problems remain in terms of the design of surfactants as the exact interaction mechanisms between particles and surfactants are still unclear.

This work concerns with the suspension of carbon particles in viscous solutions. We explored the dispersion of carbon particles with different shapes, sizes, and microstructures in a highly viscous aqueous solutions with an aim for long shelf-life applications. Both physical and chemical treatment methods were used to enhance the stability of the suspensions.

Inorganic solid nanoparticles were used to surface modify the suspended carbon particles. The suspensions were characterised for their physical and chemical properties and stability was investigated. We shall present these most recent findings at the conference.

Analysis of a rotary granulator process by Magnetic Particle Tracking

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Keywords | Magnetic Particle Tracking; CFD-DEM simulation; Rotary Granulator; Particle dynamics;

Analysis of a rotary granulator process by Magnetic Particle Tracking

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The Magnetic Particle Tracking (MPT) method is an in-situ, online measurement method, where a tracer particle (permanent magnet) follows the gas-solid flow without interference, and the spatial location as well as the orientation of the tracer can be measured. MPT was successfully used in previous works at TUHH (Neuwirth et al., 2013 and Oesau et al., 2022). The schematic and a photograph of the plant are illustrated in Figure 1.

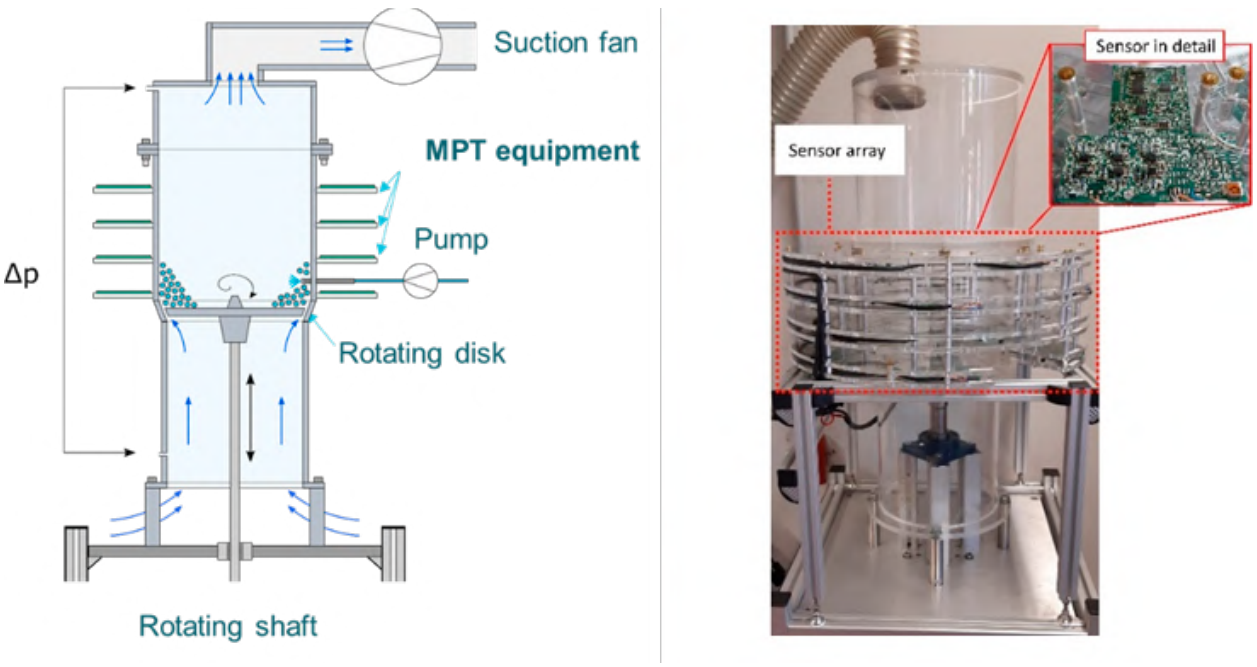


Figure 1: Schematic drawing and a photograph of the plant and the MPT equipment.

The experiments were performed with different air velocities, disk rotation rates, particle sizes (down to 2.8 mm) and particle shapes. Furthermore, the influence of the addition of water into the process was examined as well. The resulting phenomena, dead zones, velocity distributions and several dimensionless numbers are compared to results obtained by

CFD-DEM simulations, e.g. with the results by Grohn et al. (2020). As exemplary result, the radial and axial distribution of the tangential velocity is shown in Figure 2.

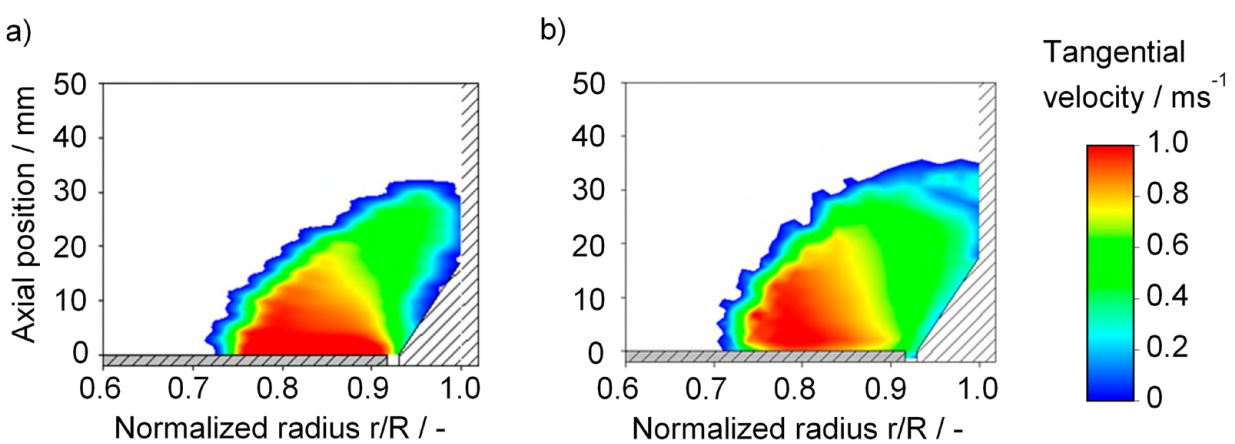


Figure 2: Visualization of the tangential velocity obtained by the simulation (a) and MPT measurements (b). Shown are the results for 5 vol-% of added water.

The authors would like to thank the Deutsche Forschungsgemeinschaft (DFG) for funding the project via HE 4526/25-1 and AN 782/12-1

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Oesau, T., Grohn, P., Pietsch-Braune, S., Antonyuk, S., Heinrich, S. 2022. Novel approach for measurement of restitution coefficient by magnetic particle tracking, Advanced Powder Technology, 33 103362

3 PARTICLE PROCESSING

Modelling specific energy requirements for knife-milled wheat straw and beech chips at different moistures and mill variables

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Keywords | beech chips, comminution law, knife mill, size reduction, specific energy requirement, wheat straw

The mechanical size reduction of lignocellulosic biomass belongs among the crucial initial biorefinery processing steps. When reducing biomass particle size, then increasing the particle's surface, and improving process efficiency for transport phenomena during subsequent biomass biorefining steps. The contribution deals with overview experimentally identified values of specific energy requirements for knife-milled woods chips and wheat straw concerning biomass moisture, screen sieve size and peripheral rotor speed. E.g. regarding initial wheat straw size D90 of 8.1 mm, it was found that specific energy requirement is inversely proportional to particle size D90 (mm) powered to the parameter r (-), as presented in Figure 1. D90 value means a characteristic particle size at the cumulative mass fraction of 90 wt %. Regarding the general mathematic solution of a differential equation, both an integration constant C and r parameters were identified by the least square methods and critically discussed concerning biomass moisture and knife mill variables. Finally, the gained original modelling approach is discussed with conventional comminution models known as Rittinger, Bond, or Kick laws.

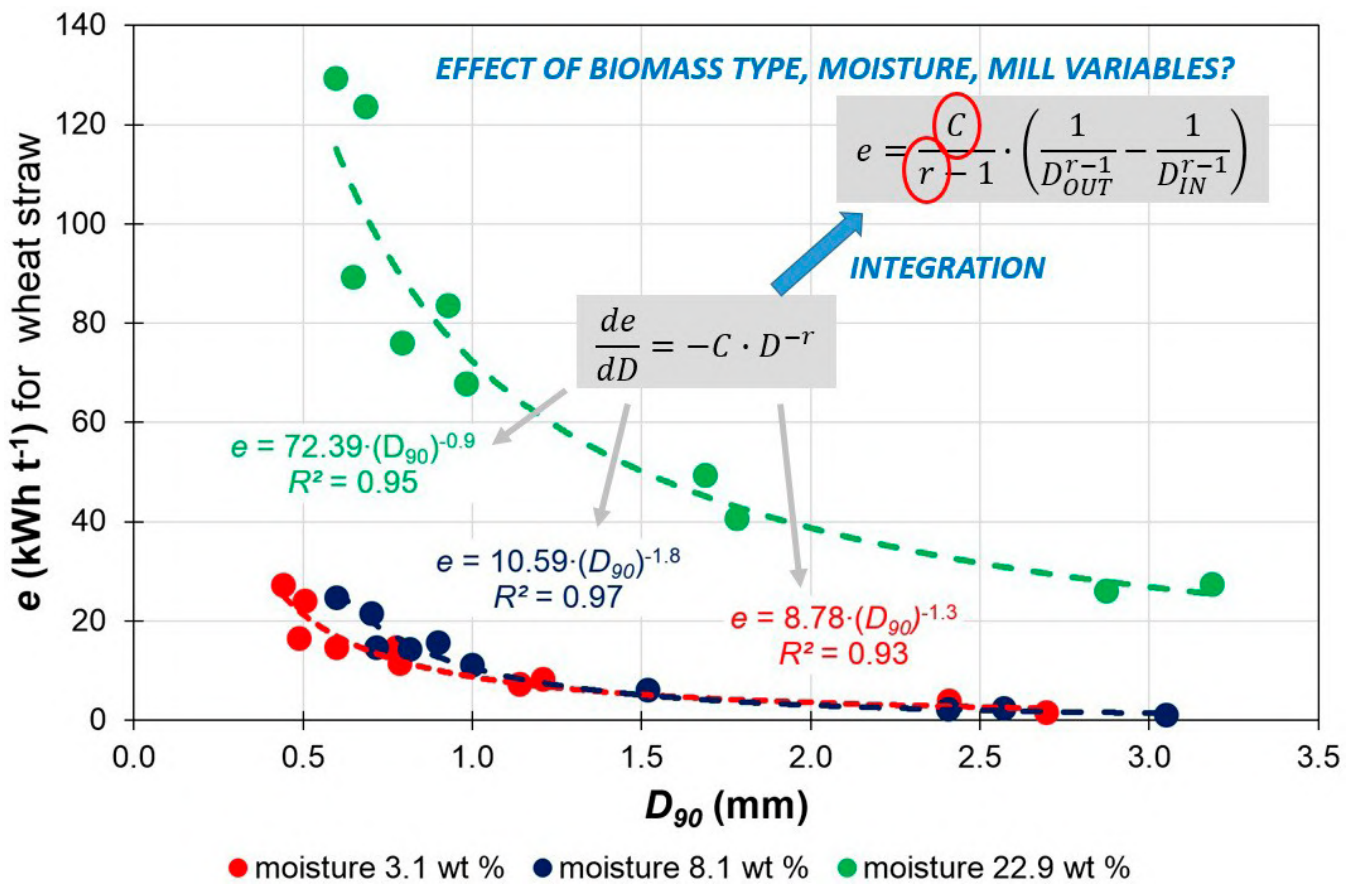


Fig. 1 Specific energy requirement dependent biomass moisture (point – experimental value, line – fitted curve). Acknowledgement:

This research was supported by the Ministry of Education, Youth and Sports of the Czech Republic under OP RDE grant number CZ.02.1.01/0.0/0.0/16_019/0000753 "Research centre for low-carbon energy technologies".

Geopolymers as potential adsorbents to remove water-based flexographic inks

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Keywords | Eucalyptus ash, geopolymer, flexographic ink, adsorption process.

Geopolymers as potential adsorbents to remove water-based flexographic inks

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The increased use of water-based or flexograhic inks in paper and plastic industries creates significant environmental concerns since they are not removed by conventional deinking technologies such as flotation (Balea, 2017). Therefore, they remain dissolved in water and alter the recycling processes and the possibilities of water reuse. This work describes the preparation, characterization, and utilization of geopolymers using metakaolin ans eucalyptus ash to remove copper phthalocyanine blue from aqueous effluents. Different ratios ash metakaolin were tested, varying the concentration of NaOH and the volume of H2O2 used to prepare the geopolymer. The samples were characterized by X-Ray Fluorescence, X-Ray Diffraction, Field Emission Scanning Electron Mcroscopy, Fourier Transform Infrared Spectroscopy and BET Surface Area. The influence of adsorption time, pH, ink initial concentration and adsorbent dosage on ink removal were evalued. Figue 1 shows the variation of the ink removal as a function of the reaction time al two pHs.

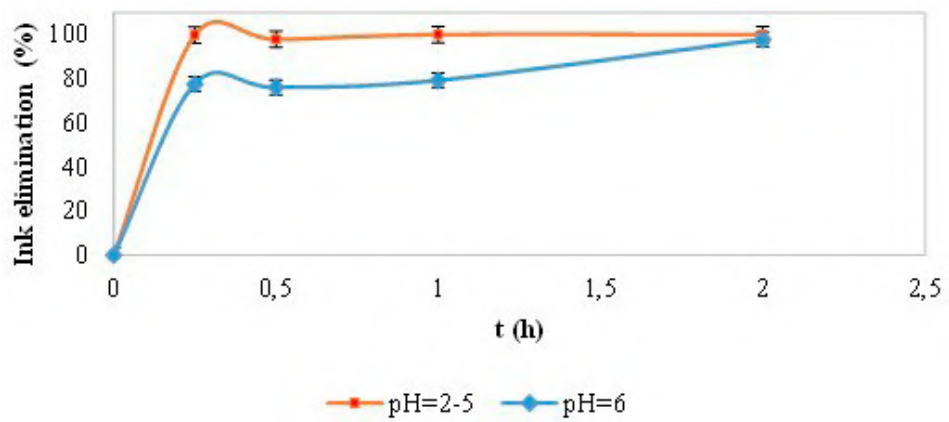


Figure 1. Ink removal as a function of adsorption time and pH (geopolymer dosage 2 g/L, ink initial concentration 5 mg/L).

Results show that the ink can be totally removed after 2 hours for both pHs tested. The ink adsorption is more favourable at acid pH values since the ink is adsorbed in less than 30 minutes. It is conclued that geopolumers are potential efficent adsorbents for flexographic inks.

Acknowlogments

Thank to Community of Madrid for the project RETROPROSOST-2-CM, S2018/EMT-4459.

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Characterization of the deformation behavior of metal powders at different stress intensities in wet and dry operated mills

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Keywords | Wet-operated grinding media mill, stress behavior of metals, physical material properties

Additive manufacturing is well established in both research and industry. In the field of metallic materials, Selective Laser Melting (SLM) has prevailed over Selective Laser Sintering, enabling defined components to be produced in a short time by melting the metallic raw materials.

Metallic raw-materials involve a considerable challenge for process preparation steps, since the powders used in the SLM process require a defined shape- and size distribution. In addition to the dry top-down powder design processes already investigated for various metals and the ideally shaped materials obtained from synthesis (bottom up), a wet top-down process route is being evaluated in this study. In a first step, the metal powders, which are usually irregularly shaped and often broadly distributed in size, are stressed in a wet-operated grinding media mill. Varying the rotational speed, the grinding media size and the grinding media density, the used metal powders suspended in ethanol are stressed in a planetary ball mill. Copper, molybdenum and tungsten powders were used as raw materials. At defined time steps, samples are taken and the particle size distribution is measured as well as the shape is examined by SEM-images. As an example, Figure 1 indicates the stress behavior of tungsten particles in a planetary ball mill. After initial deagglomeration, the size of the particles increases over the duration of the stressing period. SEM-images reveal that with increasing stress time the particles form flake-like thin layers. In this process, the edges of flakes are separated and subsequently cold welded onto the surface of larger flakes to form new layers. This appears to be a continuous cycle of comminution and reunification whereby the crystal lattices are displaced into each other by stress events, leading to material unification. The metal particles do not seem to store the energy required for fracture, but instead convert most of this energy into plastic deformation. From this, it is concluded that there is a relationship between the stress-intensity and the physical material properties for the metal particles used resulting in different flake formed particles.

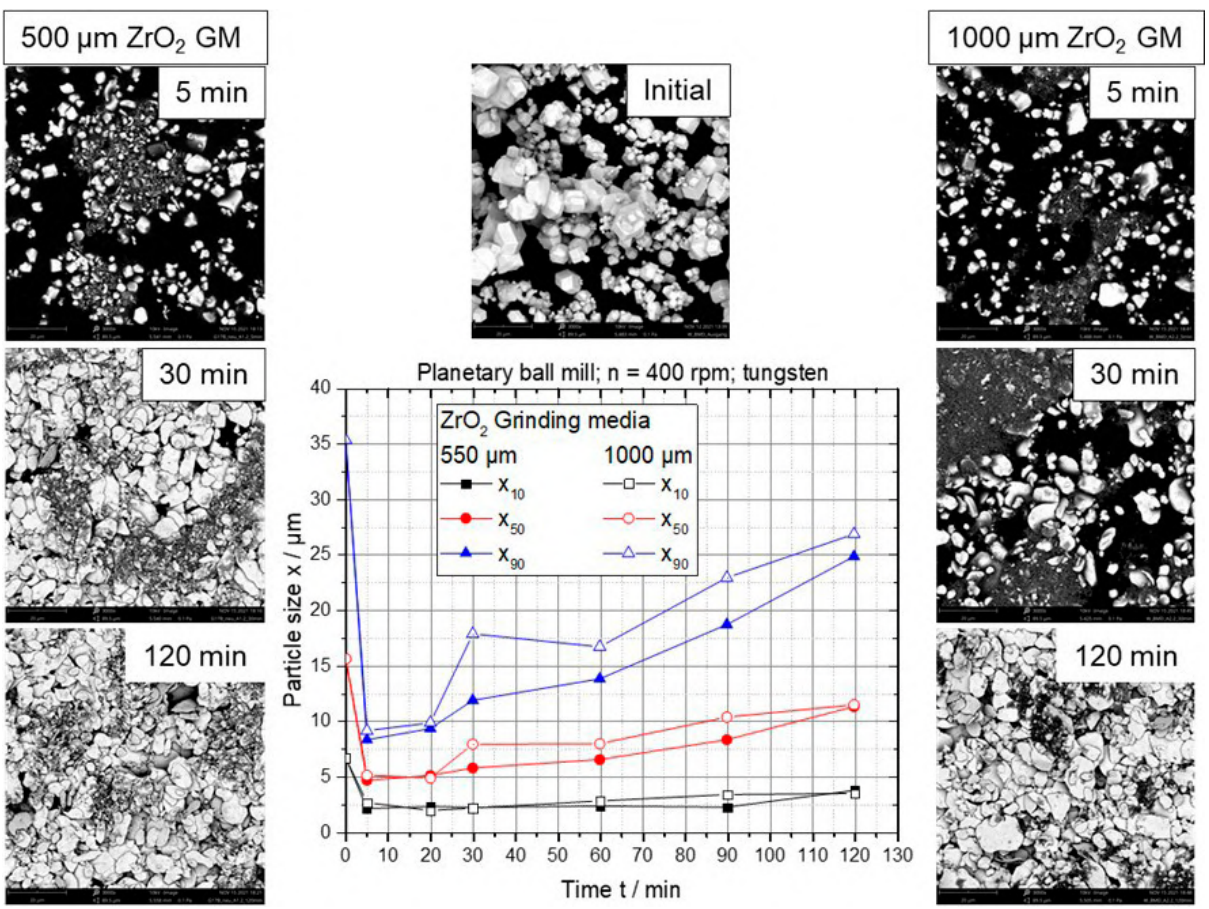


Figure 1. Particle size distributions and deformation behavior of tungsten particles at different stress-intensities.

Drying of avocado wastes from guacamole production in a conical spouted bed dryer

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(1) University of the Basque Country

Keywords | Avocado wastes, conical spouted beds; drying; renewable biomass

Avocado is an edible fruit, with global consumption over 7 million tons in 2019 (FAOSTAT, 2021), because of its nutritional properties. Industrial manufacturing of guacamole, the main processed product, generates a large amount of wastes, mainly the skin and the seed, suitable for energy valorization due to its high heating value. Thermal efficiency of avocado wastes improves by remove moisture content by means of prior dying.

Drying process of avocado wastes was conducted at 105 °C in a plant designed at purpose, Figure, provided with a conical dryer based on the spouted bed technology, successfully applied for thermal exploitation of biomass wastes (San José et al., 2013a, 2013b, 2014a, 2014b, 2017, 2018, 2019).

Biomass used consists of binary mixtures of avocado seeds and skin wastes of density, 1143 and 611 kg/m3, respectively and moisture content of 117 wt% (db), grinded by a mill up to particles of mean Sauter diameter of 3.75 and 4.7 mm, respectively.

Beds of binary mixtures of avocado wastes present low segregation experimentally determined by the mixture index. Moisture content of avocado wastes particles decreases with the time to the equilibrium moisture, more pronounced at short times. The feasibility of the conical spouted bed dryer for drying of avocado wastes has been proven with short drying times.

Acknowledgements.

This work was performed with financial support from the Spanish Ministry of Economy and Competitiveness and co-funded by the European Union through ERDF funds (Project CTQ2017-89199-P).

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Low-cost Microplastic Detection and Identification using Microfluidic ATR Platform for Ocean Application

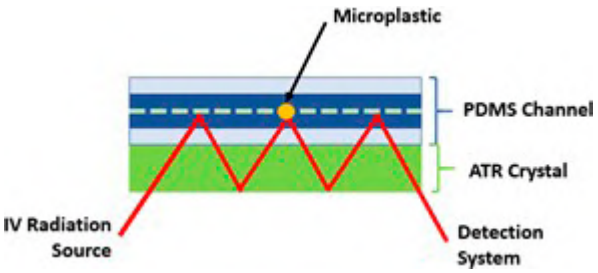
Ramos Aires Moreira Penso, Camila Maria (1); Ribeiro, Clarisse (1); Paiva, Maria Conceição (1); Gonçalves, Luís Miguel (1)
(1) Universidade do Minho

Keywords | Microplastics; ATR; Microfluidic; PDMS

Abstract

Pollution due to plastic degradation has originated an increasing need to create robust and autonomous systems able to detect microparticles (<5mm) in the ocean. (Ivleva, Wiesheu, and Niessner 2017) Several technologies have already been developed, although most require sea samples to be filtered. During this process, plenty of particles are lost, due to filter size and misidentification. Raman technologies represent one of the most promising technologies to detect microparticles. However, when in seawater, microparticles develop biofilm and this originates fluorescence in the Raman spectra. (Shim, Hong, and Eo 2017) Fluorescence overpowers the Raman signal and the polymer footprint is lost.

In this work, we propose a detection system based on attenuated total reflection (ATR) combined with a PDMS microfluid platform. ATR is an attractive methodology for in-situ sensors since it's a low power, small size, and long lifetime technology.



As can be seen in figure 1, over the traditional ATR system we constructed a microfluidic channel, made of PDMS, where seawater and particles travel. Particle separation per size is done with a centrifuge system based on a spiral microfluidic channel,[CMRAMP1] before crystal interaction. (Al-Faqheri et al. 2017) Particles travel in the middle of the channel to always allow the same penetration depth of light. Reference light collects only the signal due to superficial reflection. Both signals are detected and processed to eliminate fluorescent interference.

With this system, we hope to provide a worldwide solution to microparticle identification using low-cost technology, allowing for an in-situ microplastic identification and quantification while using simple and robust technology.

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Transition to a new circular economy model by sludge-based activated carbons for the sustainability removal of organic compounds

Gutiérrez Sánchez, Pablo (1); Sanz Santos, Eva (1); Álvarez Torrellas, Silvia (1); Larriba Martínez, Marcos (1); Águeda Maté, V. Ismael (1); García Rodríguez, Juan (1)
(1) Complutense University

Keywords | Activated carbon; adsorption; neonicotinoid pesticides; sludge-based adsorbents

In the EU, around 13.0 million tons of sewage sludge (in dry matter) are produced annually (Havukainen et al., 2022). Additionaly, the presence of emerging and priority pollutants in the aquatic environment represents an environmental problem of increasing concern. These compounds have low biodegradability, which makes it difficult to eliminate them in wastewater treatment plants (WWTPs) and leads to their frequent presence in the treated effluents. Given their continuous introduction into the environment, micropollutants have been detected in surface water, groundwater, soil, and even in drinking water (Sanz Santos et al., 2021).

This study is focused on the synthesis of four activated carbons (ACs) with different activating agents (ZnCl2, FeCl3.6H2O, Fe(NO3)3.9H2O, and Fe(SO4)3.H2O), using a pharmaceutical industry sludge as precursor, to be used for the removal by adsorption of three neonicotinoid pesticides included in EU Watch List (Decision 2018/840): acetamiprid (ACT), thiamethoxam (THM), and imidacloprid (IMD). The prepared ACs showed micro–mesoporous properties, obtaining relatively slow adsorption kinetics to reach equilibrium, but despite this, high values of adsorption capacity (qe) were obtained. In the case of AC-ZnCl2 (SBET = 558 m2/g), high adsorption capacities of qe = 128.9, 126.8, and 166.1 mg/g for ACT, THM, and IMD, respectively, were found. In most cases, the adsorption isotherms showed a multilayer profile, indicating an important contribution of the mesoporosity of the activated carbons in the adsorption process.

Acknowledgements

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Characterization of new heterogeneous catalysts obtained from urban and industrial sewage sludge. Application in emerging contaminants removal

Gutiérrez Sánchez, Pablo (1); Sanz Santos, Eva (1); Álvarez Torrellas, Silvia (1); Larriba Martínez, Marcos (1); Águeda Maté, V. Ismael (1); García Rodríguez, Juan (1)
(1) Complutense University

Keywords | Sludge catalysts; catalityc wet air oxidation; ciprofloxacin; emerging pollutants

Sewage sludge is generated both in municipal and industrial wastewater treatment plants in increasing amounts. It is waste material that is very difficult to manage because of the variability of its composition, pollution by pathogens and micropollutants, and high organics and water content. The current practice of sludge management has considerably changed during the past twenty years. Improved biogas production, advanced sludge dewatering processes, controlled land filling and thermal processes are increasingly applied in practice. This is accompanied with an increase in costs of sludge management. The costs often represent more than fifty percent of the total wastewater treatment costs. Due to the high costs, but recently also due to the urgency to develop more sustainable scenarios for sludge treatment, an increasing growth is observed in research into innovative more sustainable sludge treatment processes (Domini et al., 2022).

In this context, both the urban and industrial sludge were characterized and used to synthesize iron-based catalysts. These catalysts were characterized with XRD, XRF, BET, TG/DTG and SEM. It was confirmed that the prepared catalysts are essentially mesoporous (type IVa isotherms) and the supported Fe particles in the pores of activated carbon are needle shaped.

Acknowledgements

This work has been supported by the Spanish MICINN through the project CATAD3.0 PID2020-116478RB-I00. In addition, the authors thank the financial support from the Comunidad de Madrid (Spain) through the Industrial PhD projects (IND2017/AMB-7720 and IND2019/AMB-17114) and REMTAVARES Network (S2018/EMT-4341) and the European Social Fund.

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Electrocatalytic reduction of CO2 to formate: assessment of particles with different nature

Díaz Sainz, Guillermo (1); Merino-García, Iván (1); Fernández Caso, Kevin (1); Albo, Jonathan (1); Álvarez-Guerra, Manuel (1); Irabien, Ángel (1)
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Keywords | CO2 electrocatalytic reduction; formate; SnO2 nanoparticles; Bi carbon-supported nanoparticles; Gas Diffusion Electrodes

The “Development of Chemical processes and Pollution Control” (DePRO) research group, led by Prof. Angel Irabien, of the Department of Chemical and Biomolecular Engineering at University of Cantabria has made great efforts in the field of continuous CO2 electroreduction to formate for over a decade, working with nanoparticles of different nature and electrode configurations, as a promising approach to reduce the CO2 emissions to the atmosphere and, at the same time, to generate value-added products. In this context, this communication aims

at presenting a rigorous comparative assessment of different experimental results obtained in our laboratory facilities (same operating conditions) using Pb, Sn, SnO2, and Bi- based (nano)particles in different electrode configurations for the continuous conversion of CO2 to formate, employing a filter-press electrochemical reactor with a simple pass of the reactants in the cell.

Table 1. Summary of our results obtained in the continuous CO2 electroreduction to formate.

Figures of merit	Pb plate	Sn plate	Sn PE (150 μm)	Sn PE (150 nm)	Sn GDE (15 nm)	Sn CCME (15 nm)	SnO_2 GDE (2 nm)	Bi GDE (10 nm)	Bi CCME (10 nm)	Bi MEA (10 nm)
Formate rate ($mmol \cdot m^{-2} \cdot s^{-1}$)	0.45	0.44	1.4	3.2	4.8	1.1	7.0	4.6	1.2	2.0
Faradaic Efficiency (%)	40	70	70	70	45	49	45	45	54	89
Formate concentration ($g \cdot L^{-1}$)	0.05	0.14	1.35	1.5	16.9	19.2	27	25.9	25.9	337
Current density ($mA \cdot cm^{-2}$)	22	12	1.40	90	200	45	300	200	45	45

It is important to highlight that the best results obtained up to date have been achieved with SnO2 (Merino-García et al., 2021), and Bi carbon-supported (Díaz-Sainz et al., 2021) nanoparticles with an average particle size of 2 and 10 nm, respectively. Both nanoparticles are deposited in the form of Gas Diffusion Electrodes (GDE) by airbrushing techniques. Firstly, the use of SnO2 nanoparticles allowed for achieving a maximum formate concentration of 27 g·L-1 (stable up to 10 h) in a liquid-liquid configuration, while the Bi-based cathodes reached remarkable results in terms of formate concentration of 340 g·L-1 and Faradaic Efficiency for formate (89 %) as a function of the amount of water in the CO2 input stream (gas-liquid configuration). These studies carried out in DePRO's research group represent a step forward in the development of continuous CO2 electroreduction to formate processes to get closer to practical applications.

Acknowledgments

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Effect of surface modification of CaCO3 nanoparticles by a silane coupling agent on the stability of foam and emulsion

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(1) Dongguk University-Seoul

Keywords | CaCO3 nanoparticles, Surface modification, Silane coupling agent, Emulsion stability, Foam stability

In this study, the surface modification of calcium carbonate (CaCO3) nanoparticles was performed by using a silane coupling agent methyltrimethoxysilane (MTMS) and effect of hydrophobicity of nanoparticle surface on the stability of foam and emulsion was investigated. In order to confirm surface modification of CaCO3 nanoparticles by MTMS, Fourier infrared spectroscopy, differential scanning calorimetry, and thermogravimetric analysis have been carried out. Atomic concentration of CaCO3 nanoparticle surface treated by MTMS has been identified by using x-ray diffractometer and x-ray photoelectron spectroscopy analysis. The active ratio for the CaCO3 nanoparticles modified by MTMS was found to be 97.6% at 15 wt% concnetration of MTMS where the contact angle of water on the CaCO3 surface modified by MTMS was found to 89.6. Both stable foam and emulsion were formed at 15 wt% concnetration of MTMS, suggesting that the CaCO3 nanoparticle treated by a silane coupling agent MTMS is a strong candidate for the potential applicability as a stabilizer for foam and emulsaion.

Theoretical and Experimental Investigation of Acoustic Agglomeration of Aerosol Particulates

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Keywords | acoustic agglomeration, aerosol particulates, DEM, CFD

This report aims to investigate the theoretical and experimental aspects of Acoustic Agglomeration. Phenomenon, where small particles under periodic motion in the fluid approach each other forming large agglomerates. This process is governed by different hydrodynamic mechanisms, and the efficiency of agglomeration depends on the frequency of the acoustic pressure.

Our investigation is restricted to SiO2 particles with diameter from 0.3 to 10 μm subjected to a frequency of 500 to 3000 Hz. Various interaction mechanisms comprising binary interaction of particles, contribution of third particles, and collected behaviour of multiparticle systems are investigated numerically in the framework of DEM. A comprehensive study of very fine particles in DEM was performed that included orthokinetic agglomeration and acoustic wake effects. The same selected samples were tested by applying the CFD approach.

The analogous agglomeration mechanism was also investigated experimentally. The experimental facility adopted to investigate particle emission in real conditions was located in an industrial chimney system.

The acoustic effect on PM agglomeration was determined by sampling before and after the agglomeration zone. The change of particle concentration obtained for different parameters of acoustic exposure are shown in Figure 1. The blue curve indicates the initial particle size distribution before acoustic pretreatment. The remainder curves illustrated particles size after acoustic pretreatment of different sound pressure frequencies. The graphs show that two different agglomeration regions were observed. In the smaller size range, the concentration of particles with a diameter of 0.2 μm to 3.0 μm decreases by almost 80% (500 and 3000 Hz), and the number of particles with a diameter of 5.0 μm to 10.0 μm increased to 80% (at 500, 750, 1500 and 3000 Hz).

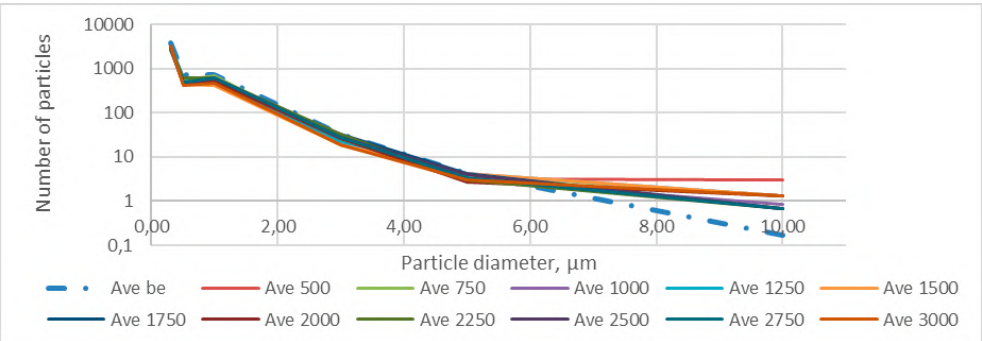


Fig. 1 Results of particle number measurements with and without acoustic effects

The experimental results confirm the theoretically predicted agglomeration character and provide a better understanding of the behavior of the particles in the acoustic field. Further investigation by applying higher frequencies is required to describe the agglomeration of smaller particles.

Funding.

This research has received funding from the European Social Fund (project No. 09.3.3-LMT-K-712-19-0026) under grant agreement with the Lithuanian Research Council (LMTLT).

Acoustic Agglomeration of Microparticles in a Humid Environment

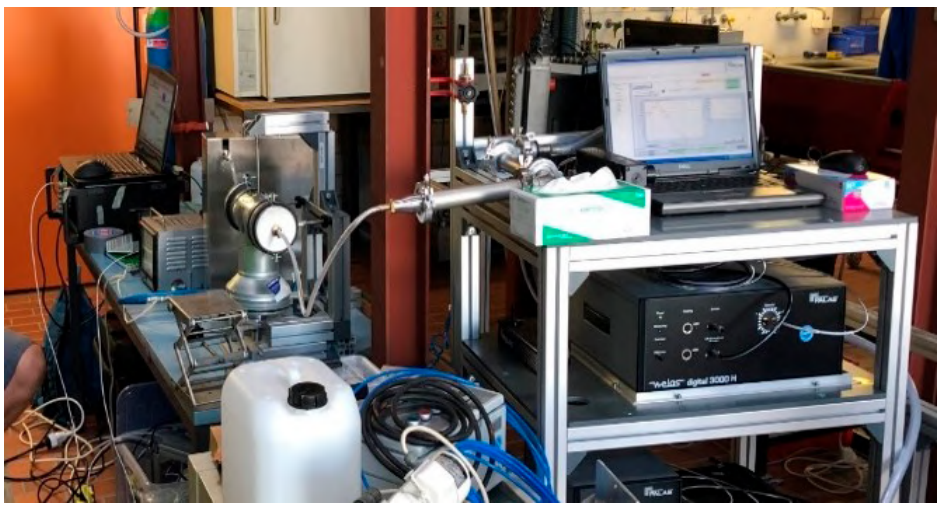
Kilikevičienė, Kristina (1); Kačianauskas, Rimantas (2); Kerner, Maximilian (3); Antonyuk, Sergiy (3); Vainorius, Darius (2)
(1) Institute of Mechanical Science,, (2) Institute of Mechanical Science, Vilnius Gediminas Technical University, Vilnius LT-10223, Lithuania, (3) Technische Universität Kaiserslautern, Institute of Particle Process Engineering, 67663 Kaiserslautern, Germany

Keywords | Acoustic agglomeration, microparticles, humid environment

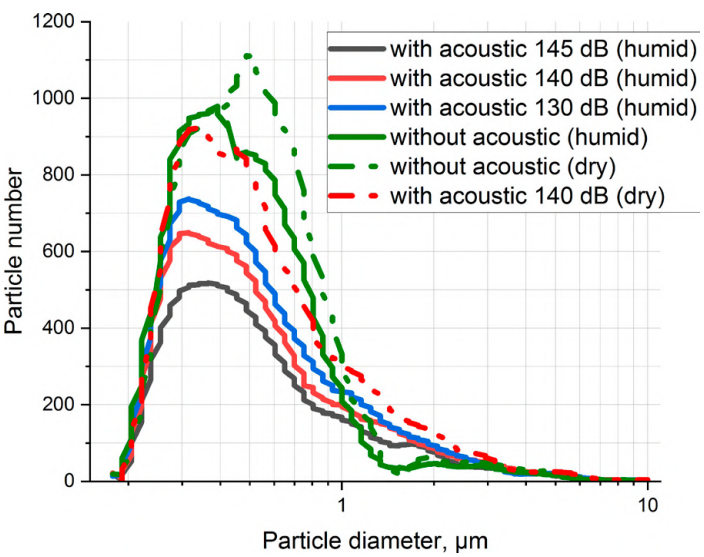
The reduction of the emission of fine micrometer-sized particles into the atmosphere is an important measure to prevent pollution of the environment and the harmful effect on human health. The development of reliable particle removal technology is based on the fundamental properties of particle mechanics. Agglomeration is one of the most important phenomena that make fine particles aglomerate in a large size distribution. The combination of acoustic agglomeration and humidification in the pretreatment process is investigated in this report.

Experimental equipment was used to pretreatment small particles. The main part of it consists of an agglomeration chamber (containing horizontal cylindrical based (korpusas) with controlled piezoelectric acoustic generator), particle dosing device (Palas RGB 1000) and particle measuring equipment (Welas digital 3000H) (Fig. 1a). The generator was designed to generate a controlled high pressure level reaching ab 145 dB at a fixed frequency equal to 20 kHz. The required humidity (60 ± 5 %) is specified and maintained during the experiment.

A series of tests was conducted to evaluate the effect of pretreatment. Fixed doses of glass particles spaced in the acoustic chamber were considered and the concentrations of the particulates were measured. The selected results of the concentrations of particulates in the humid (solid curve) and dry (dashed curves) environment in addition to the acoustic field (130, 140 and 145 dB) are presented in Figure 1b.



a)



b)

Fig. 1. View of the experimental facility stand (a) and results of particle number measurements (b): black, red, green, and blue solid curves - humid environment; red and green dashed curves - dry environment

The particle agglomeration in the humid environment was observed for range (0.2 - 1.0 μm) in diameter, which decreased from 38 to 55 %.

Particle concentration increased significantly from 205.1 to 314.6 % in the range (1.0 - 3.0 μm).

The results indicate that the combined effects of sound fields and a humid environment lead to agglomeration of small particles, and therefore reduce the emission of small particles.

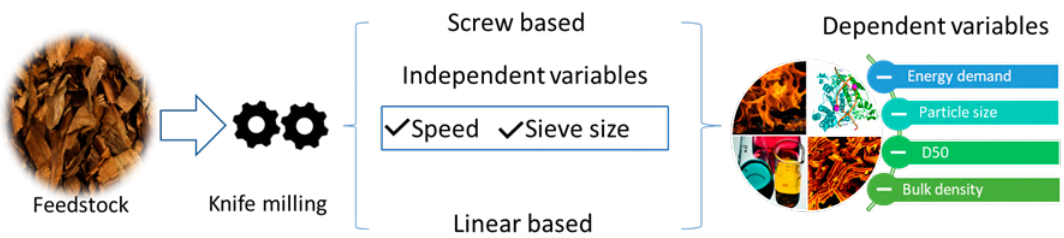
Funding

This research has received funding from the European Social Fund (project No. 09.3.3-LMT-K-712-19-0026) under grant agreement with the Lithuanian Research Council (LMTLT).

Effect of rotor geometry on physical properties and energy consumption of beech wood chips

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(1) Czech Technical University. Faculty of Mechanical Engineering. Department of Process Engineering

Keywords | Comminution, Energy Consumption, Biorefinery, Particle size



Lignocellulosic biomass (LCB) is the largest carbon source on Earth and through the biorefinery concept, can be used to obtain several value-added products, with the potential of replacing the use of fossil fuels, shifting industry to a more environmentally-compatible sector.

This material needs pretreatment to be valorised and mechanical treatments have proven to be compatible and effective enough to be used with this kind of material. These treatments reduce the particle size of LCB making it easier to handle by the reduction of the particle size and increasing the accessibility of the sugars contained in the lignocellulose matrix. The selectivity of this process is not high but mass yield is.

However, energy consumption is the biggest drawback this technology presents. Therefore, a thorough study regarding this factor, with the objective of reduce it as much as possible without negatively affecting the following processes, needs to be addressed.

Among all configurations that can be used for mechanical size reduction, knife milling has been successfully used by several authors in the treatment of LCB to increase the effectivity of the subsequent treatment either biological (Chuetor et al., 2021; Garuti et al., 2022) or chemical (Bokhari et al., 2021) and will be the one studied is this research.

Experimentation wise, two different rotors have been chosen (linear and screw based), and as independent variables, rotational speeds (1000, 2000 and 3000 rpm) and several sieves from native to 0.75 mm. As dependent variables, particle size, D50, bulk density and energy demand were analysed to obtain the most suitable configuration regarding energy consumption and physical properties.

Bokhari, S.M.Q., Chi, K., Catchmark, J.M., 2021. Structural and physico-chemical characterization of industrial hemp hurd: Impacts of chemical pretreatments and mechanical refining. Ind. Crops Prod. 171. https://doi.org/10.1016/j.indcrop.2021.113818

Chuetor, S., Ruiz, T., Barakat, A., Laosiripojana, N., Champreda, V., Sriariyanun, M., 2021. Evaluation of rice straw biopowder from alkaline-mechanical pretreatment by hydro-textural approach. Bioresour. Technol. 323. https://doi.org/10.1016/j.biortech.2020.124619

Garuti, M., Sinisgalli, E., Soldano, M., Fermoso, F.G., Rodriguez, A.J., Carnevale, M., Gallucci, F., 2022. Mechanical pretreatments of different agri-based feedstock in full-scale biogas plants under real operational conditions. Biomass and Bioenergy 158. https://doi.org/10.1016/j.biombioe.2022.106352

Development of a hetero-aggregation process of submicron particles by mixing and desublimation in a supersonic flow

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(1) TU Kaiserslautern

Keywords | Aerosol generation, particle morphology, supersonic, desublimation, CFD, coating

In many industrial processes the production of particulate products is an important process step. Particulate functional materials like high-quality pigments or catalysts often consist of different particulate materials that can be aggregated by different adhesion mechanisms. The chemical, physical and morphological properties of submicron hetero-aggregates are unique and critical for the quality of the products. In this work, mixing and desublimation mechanisms of submicron particles in a supersonic flow are described to develop a hetero-aggregation process. Fig. 1 shows the experimental setup (Urazmetov et al., 2021) that consists of a disperser, tube furnace, Laval nozzle and expansion chamber. The gaseous copper- phthalocyanine phase with high supersaturation bonds with the TiO2 core particles by a forced desublimation process.

Due to strong influence of the fluid dynamics on the mixing behavior the processes in the Laval nozzle and expansion chamber were analyzed using computational fluid dynamics (CFD). The energy equation and the k- ω SST model are used to study the compressible flow in detail. Experimental studies provide information about process parameters leading to the formation of the different hetero-aggregate structures. To analyze the properties of the generated aggregates, token samples of a nano aerosol sampler (NAS) are characterized by scanning electron microscopy (SEM). Comprehensive information about particle shape is obtained by measurements with the Light Scattering Sensor (3D-LSS) (Pitz et al., 2018).

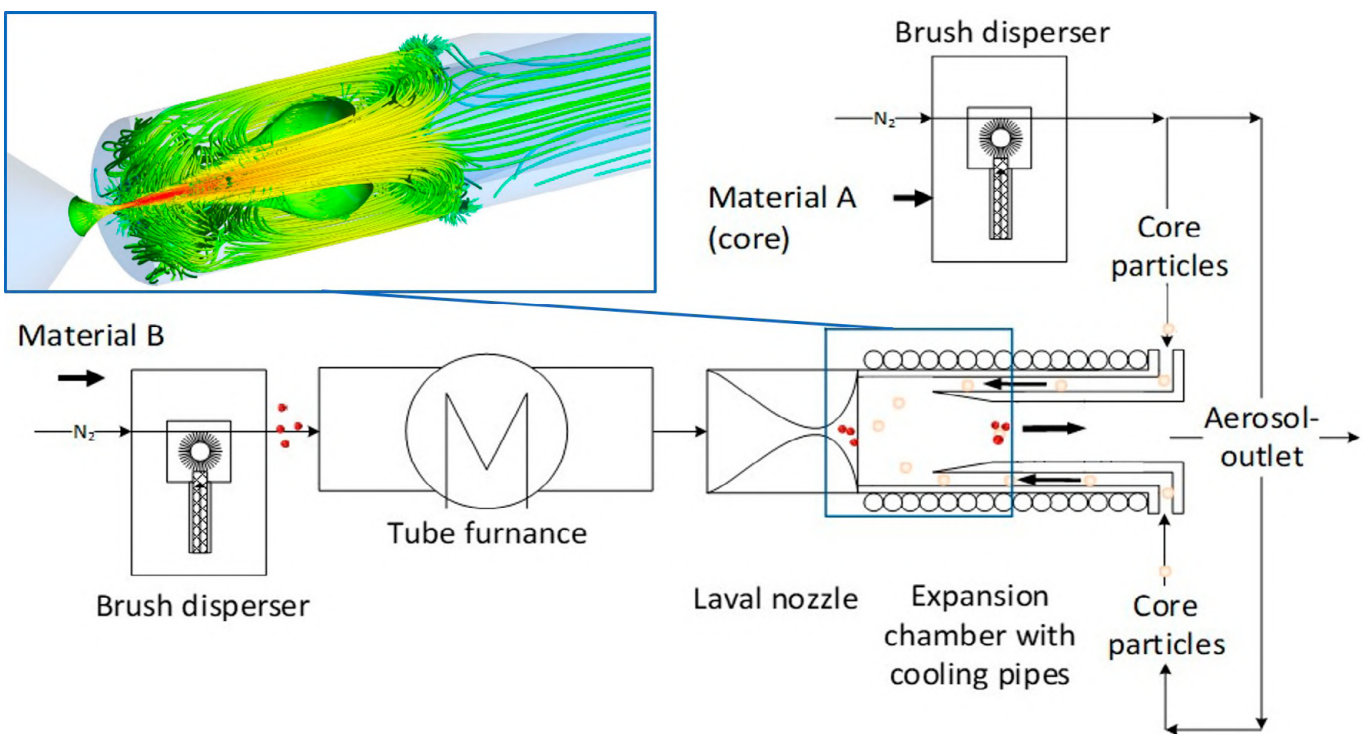


Figure 1: Experimental setup for the hetero-aggregation of submicron particles in supersonic flow (Urazmetov et al., 2021)

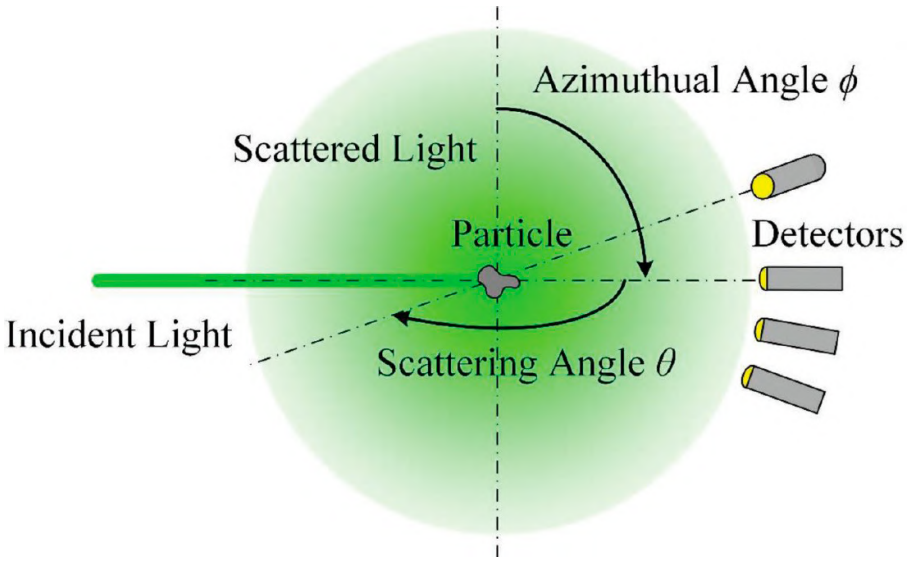


FIGURE 2: Principle of the 3D-Light Scattering Sensor for the particle shape measurement (Pitz et al., 2018)

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Pitz, M., Hellman, A., Ripperger, S., Antonyuk, S., 2018. Development of a 3D Light Scattering Sensor for Online Characterization of Aerosol Particles, Particle & Particle Systems Characterization 35

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Immobilization of amylase and protease in iron oxide microparticles. Application in food cleaning

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(1) Universidad de Granada

Keywords | Enzymes Microparticles Immobilization Detergency

Introduction:

The application of enzymes for the cleaning of food environments provides an interesting alternative for the classical Cleaning-in-Place (CIP) treatments involving chemical agents, reducing energy and water consumption, while maintaining the required hygiene levels. Proteases and amylases are the most commonly used enzymes. The covalent immobilization of enzymes on magnetic supports is a suitable option for the control of enzymatic catalysis in industrial applications, since allows the removal and subsequent reuse of the enzyme simply using the action of a magnetic field, reducing the total cost of production and the time of the cleaning process.

Methods:

Amylase (Termamyl®120) and protease (Savinase®16.0L) were immobilized separately on iron (II, III) oxide microparticles (MPs) that were functionalized with amino-silane functional groups through the silanization reaction using 3- aminopropyltriethoxysilane (APTES). The activities of free and immobilized protease and amylase were measured using a modification of Anson's method and the iodine-iodide method (Jurado et al., 2015), respectively. Enzymatic stability was also analyzed when both enzymes were acting jointly.

Results:

The cleaning of starch and protein adhered to stainless-steel was compared using a BSF (Bath-Substrate-Flow) device simulating a laboratory-scale CIP system. The influence of flow rate, starch load, free and immobilized enzymes, MPs concentration and magnetic field on the detergency results was analyzed. The recovery of immobilized enzymes on MPs was also analyzed using a magnetic field. The use of

immobilized enzymes in MPs allowed achieving acceptable detergency values similar to those obtained by enzymes in free state, allowing their reuse.

References:

Jurado, E., Herrera-Márquez, O., Plaza-Quevedo, A., Vicaria, J.M., 2015. Interaction between nonionic surfactants and silica micro/nanoparticles. Influence on the cleaning of dried starch on steel surfaces. J. Ind. Eng. Chem. 21, 1383–1388.

4 PARTICLE-FLUID SYSTEMS: FLUIDIZATION AND MULTI-PHASE FLOW

A packed-bed based gas-solid thermochemical system for thermal energy storage - an experimental study

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(1) Birmingham centre for Energy Storage, University of Birmingham, (2) University of Oxford, (3) Birmingham Centre for Energy storage (BCES), University of Birmingham

Keywords | Thermochemical energy storage system, packed bed, gas-solid system, efficiency

Abstract:

This work is concerned with thermochemical energy storage (TCES). The TCES technology is often badged for long-term energy storage, but it can also serve for medium and short-term applications. Despite significant efforts over the past decade or so, the TCES technology remains at a low technology readiness level. Various technological challenges exist across TCES materials, devices, and systems. Here we report an experimental study on gas-solid packed-bed based TCES system (water-sorbent pair) with a rated capacity of 7.5kWh (Figure 1). Both pure sorbent (silica gel) and a composite TCES material (magnesium sulphate salt) were studied. Although the experimental system was designed as a closed system, the experiments were mostly done with the system operated as an open cycle due to long charging/ discharging processes. Figure 2 shows typical temperature profiles at different positions of the packed bed reactor as a function of time in the charging and discharging process, which provide information on the charging / discharging kinetics. Figure 2 shows a temperature lift of ~15oC in the discharging process with the outlet temperature suitable for underfloor heating applications. Our results also show an efficiency over 85% in the charging process, which is significantly higher than the discharging process efficiency of ~45% with the latter likely due to incomplete discharge and experimental system insulation. By using the charging and discharge efficiency data, one works out the overall system efficiency, which is often called the system- level coefficient of performance (COP) of 39%. Finally, a discussion on the enhancement of the system efficiency is made.

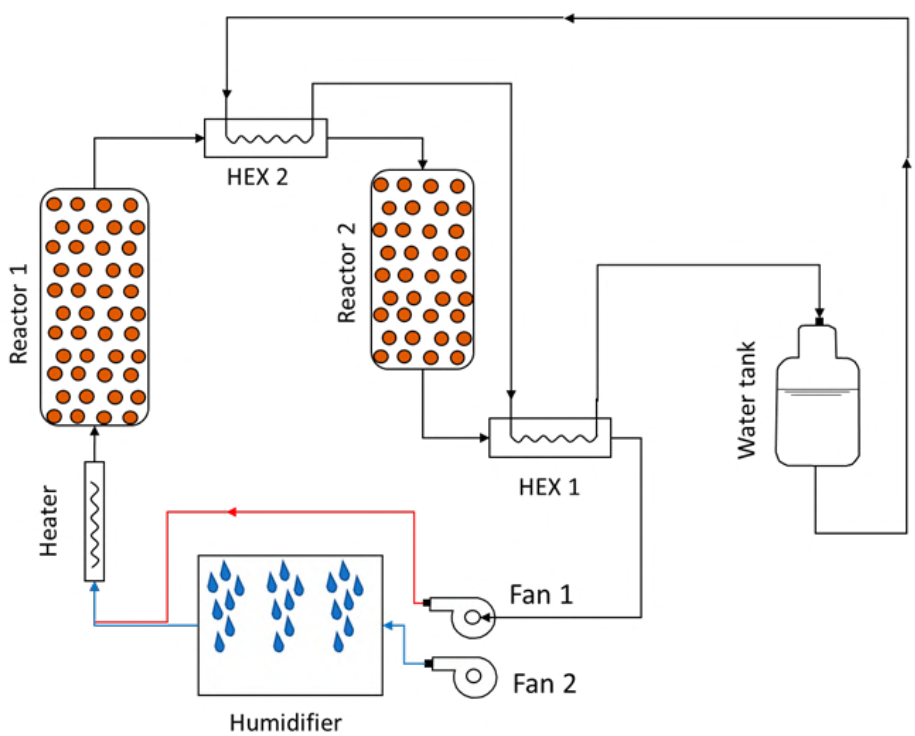


Fig. 1: Schematic diagram of gas-solid packed bed TCES system

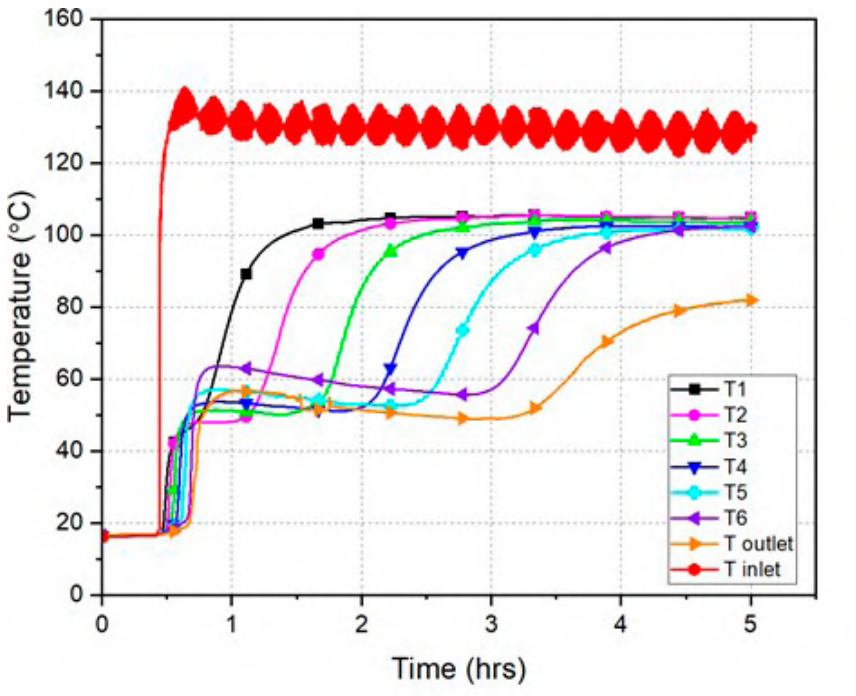


Fig. 2 (a): Experimental results for the TCES system during the charging process

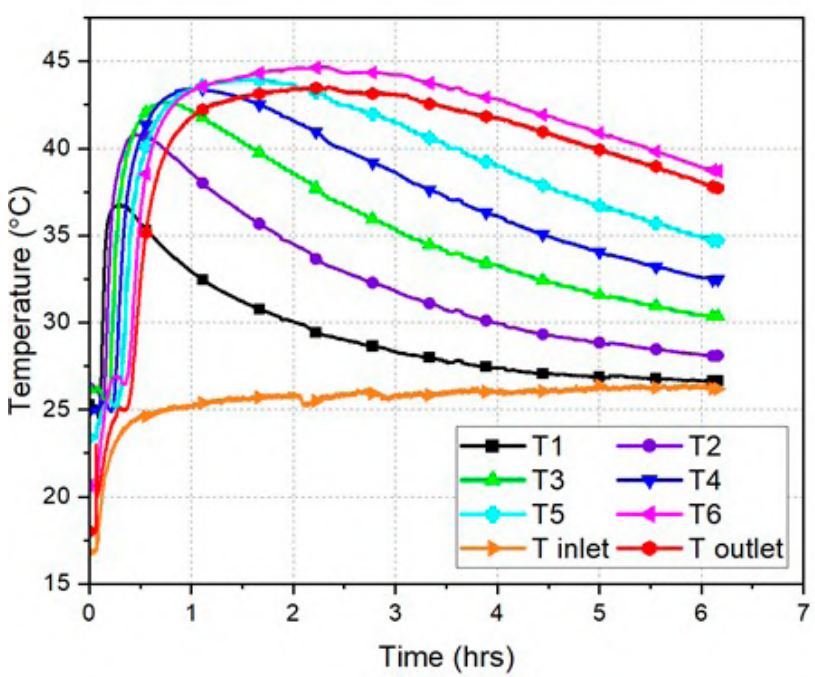


Fig. 2 (b): Experimental results for the TCES system during the discharging process

Solids Suspension and Mixing using a FUNDAMIX Vibromixer

Almasi, Sara (1); Ghobadian, Barat (2); Poux, Martine (3); Aubin, Joelle (3)
(1) INP Toulouse, (2) Tarbiat Modares University, (3) National Polytechnic Institute of Toulouse

Vibromixers are alternative mixing devices to conventional rotating impellers for a number of applications, both single and multiphase, in the chemical, biochemical, pharmaceutical and foods industries. They consist of a reciprocating plate (perforated or not), which oscillates in a vessel to pump the fluid and create flow (Wojtowicz & Paskowska 2015, Wojtowicz 2017). Vibromixers have shown to provide effective mixing in liquid reaction and shear-sensitive systems (Orlewski et al. 2018), as well as liquid-liquid dispersion (Lo et al. 1998, Kamienski & Wojtowicz 2003) and solid-liquid suspensions (Wojtowicz 2014). They have shown to ensure equivalent mixing intensity to stirred tanks, however generating significantly lower shear rates (Orlewski et al. 2018). Furthermore, it has been shown that the power consumption is up to 40% less than in stirred tanks (De Arcos Gonzalez-Turmo 2014, Wojtowicz 2014). Whilst, the concept of mixing with reciprocating baffles or plates has been around since the 1970s (De Arcos Gonzalez-Turmo 2014, Hafez & Prochazka 1974, Tojo et al. 1975 (see Lo)), there have been few systematic studies of multiphase mixing phenomena in vibromixed vessels.

In this work, the impact of oscillating conditions and plate geometry on the suspension of solids in viscous liquid and the dispersion of solid-liquid-liquid system are explored experimentally in view of improving the performance of a heterogeneously catalyzed mass transfer limited liquid-liquid reaction.

The FUNDAMIX vibromixer (DrM) is used in a 3L vessel. The impact of reciprocating disc size and position, as well as flow mode (up-pumping or down-pumping) is investigated to assess the mixing performance in solid-liquid, liquid-liquid and solid- liquid-liquid systems. Visualization experiments are conducted to assess the operating conditions required for suspension of the solids and how these affect the homogeneity of the mixture. The effectiveness of liquid-liquid and solid-liquid-liquid mixing is be assessed via drop size measurements. Separation efficiency post-mixing is also evaluated.

The results show that the FUNDAMIX is an effective mixers for multiphase systems, providing small drop size and homogeneous distribution of solids, which are important for reaction performance.

Liquid-solid phase change and heat transfer in a phase- change-material (PCM) based heat exchanger: A numerical study

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(1) Birmingham center for energy storage, University of Birmingham

Keywords | Liquid-solid heat transfer, phase change, heat exchanger; thermal energy storage; numerical modelling

This work is concerned with phase change and heat transfer behavior of a phase change material (PCM) based heat exchanger (HEX) integrated into an air-conditioning system (AC). Such an area has significance in heating and cooling decarbonization for the net-zero 2050 target. A mathematical model was built for the PCM integrated HEX for understanding the phase change and heat transfer behaviour, and for optimizing the HEX unit particularly in terms of matching the heat transfer rate between the AC and the HEX. A PCM-HEX-AC system was then designed and constructed to measure the performance of the PCM-HEX as well as the integrated PCM-HEX-AC system. The PCM integrated HEX used a plate-fin heat exchanger (PF-HEX), which was found to be efficient and easily fabricated. The model was solved numerically in an Ansys Fluent environment. The optimization considered fin density, fin height and fin arrangements. The modelling results show that the PF-HEX configuration can provide a 7 K ~ 8 K temperature difference between inlet and outlet during both discharging and charging process.es By improving the PF-HEX design, the discharging time and charging time could be reduced considerably compared with a HEX with no fins. The PF-HEX design could also effectively address the low heat transfer coefficient of PCM – a 4.5-time enhancement was observed compared with normal heat transfer coefficient. Finally, the PCM based PF-HEX was tested in a Telecommunication base station cooling system. The results showed a significantly reduced temperature variation by ~5 times.

Phase separation of phase change materials for cold energy storage

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(1) Birmingham Centre for Energy Storage, University of Birmingham, Birmingham, (2) Southern University of Science and Technology, Shenzhen

Keywords | Phase change materials, phase separation, cold energy storage

Phase change materials (PCMs) have attracted significant attention in recent decades due to their potential roles in and some salient and

desirable technical features for thermal energy storage. PCMs can be divided into 3 categories of organic, inorganic and eutectic PCMs. Of all categories, their stability during phase change is of fundamental importance, especially for industrial scale applications. However, for some PCMs, particularly inorganic PCMs with salt hydrates as the main ingredients, phase separation often exists and the formulated PCMs. This leads to heterogenous phases during thermal cycling particularly during freezing, creating a negative influence on the thermal-physical properties. This work aims to address this issue by developing various gelling and thickening agents for a salt hydrate based formulation with phase transition temperature of ~22 degC. We found that the addition of an appropriate proper agent can give no phase separation. We have also found that the formulated PCM had a significantly reduced supercooling while maintaining the high latent heat after long-term thermal cycling.

CPFD numerical simulations of a fluidized bed with an immersed tube bundle

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(1) Universidad Carlos III de Madrid, (2) Universidad de Castilla-La Mancha

Keywords | Fluidized bed, heat transfer, numerical simulation, tube bundle

Fluidized beds are considered a widely used technology to improve heat transfer in industrial processes, especially for thermochemical and concentrated solar energy applications. These devices can be combined with the use of surfaces immersed in the bed to extract heat from it for heat and power generation [Doherty et al., 1986; Wang et al., 2004]. In the present study, a numerical simulation is implemented in Barracuda Virtual Reactor (v. 21.0.1) to perform a hydrodynamic and heat transfer analysis of a fluidized bed heat exchanger.

The simulation was approximated to a 2-D numerical model, considering a scaled thickness (15 mm). Two uniform grid sizes, with approximately 90000 and 280000 real cells, and two-time steps (10-3 s and 10-4 s) have been studied. The numerical results of a 2-D model are compared and validated with those of the experimental study performed by Kim et al. (2003).

The case with coarser grid and 10-4 s time interval showed good approximation with the experimental results of Kim et al. (2003), which was selected to better explain the hydrodynamics and heat transfer in an immersed horizontal tube bundle, located according to the graphical abstract.

One tube was selected to study the local heat transfer coefficient (HTC) and particle volume fraction (PVF) distribution at different angular positions. At the top of tube (ω = 90°) there is no renovation of particles, which explains the maximum and constant PVF reached (0.6) during the simulation time, and with minimum particle velocity (close to 0). This fact is related to the defluidization phenomena occurring in this region, contributing to explain the low HTC values. Opposite tendency is shown at the bottom region, where particles are continuously in motion, resulting in high HTC and low PVF, showing a good heat transfer dissipation.

Experimental characterization of the behaviour of agglomerates in gas-solid fluidized-bed reactors

Errigo, Matteo (1); Materazzi, Massimiliano (1); Lettieri, Paola (1)
(1) University College London

Keywords | Fluidized-bed reactors, thermal hydraulics, thermal imaging, infrared, x-ray imaging, agglomerates, defluidization

Gas-solid fluidized-bed reactors have historically been widely implemented in several industries, among which the petrochemical, pharmaceutical and energetic ones. Recently, however, they have been applied to a range of emerging fields, such as waste management and CO2 capture. These reactors guarantee a high efficiency of chemical reactions and an easy temperature control thanks to their continuous and efficient mixing, allowing for excellent mass and heat transfer. However, bed material agglomerates tend to form in such reactors due to the extreme thermal conditions and the potential presence of moisture, even more likely if the fluidization is uneven and leads to the formation of hotspots. These agglomerates then tend, in turn, to impair even fluidization, establishing a positive feedback loop, which strongly affects the fluidized-bed reactor operation.

The scope of this study is exactly that of quantitatively characterizing the thermal-hydraulic characteristics of such agglomerates and to run parametric studies on the main factors affecting such properties. X-ray imaging allows for the visualisation of gas bubbles and agglomerates

within the bed thanks to their difference in density with respect to the emulsion phase as well as thanks to the dependence of the attenuation properties on the material. This technique will be used to follow the agglomerates within the reactor and to determine whether they tend to accumulate in any area, consequently causing local or global reactor defluidization.

On the other hand, the heat transfer between agglomerates and bed will be observed by the means of a shortwave infrared camera, after an appropriate calibration has been performed. The experimental setup consists of a flat pseudo-2D fluidized bed, allowing for the continuous observation of the agglomerate temperature evolution as they move within the bed. A few first preliminary results have been obtained by introducing hot agglomerates in a colder fluidized bed. The temperature evolution of the agglomerates decreases exponentially as they transfer heat to the bed, and, by reducing the size of the agglomerate, a steeper temperature evolution can be observed, as it would be expected.

Thermo-economic optimization of a novel confined thermal energy storage system based on granular material

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(1) Universidad Carlos III de Madrid

Keywords | Exergy; Granular material; Optimization; Thermocline; TES

Concentrated Solar Power (CSP) is a suitable technology for production of green electricity. However, to attain a uniform electricity production, CSP should be couple with large Thermal Energy Storage (TES) systems. Among the different technologies of TES systems, storage of sensible heat in granular material is widely used due to its simple operation. These TES systems store energy as an increase of temperature of a large mass of small solid particles, through which a fluid circulates exchanging heat. In this work, a novel confined bed is proposed to mechanically prevent the motion of the solid particles conforming the TES system even for high fluid velocities, to guarantee that the exhaust temperature of the fluid is maximum during a discharge process. In the confined bed, a thermocline evolves from bottom to top of the system, separating the low and high temperature of the bed during the discharge process. An analytical model was applied to describe the evolution of the thermocline and the effect of the different operating parameters on the thermocline thickness.

The effect of the thermocline thickness was combined with a thermo-economic analysis of a confined bed TES system proposed for a case of study. The confined bed proposed was optimized considering thermodynamics aspects, namely the fluid exergy increment in the bed, and economic factors, specifically the total investment cost of the TES system. The optimization resulted in low values of the fluid velocity, between 0.2 and 0.4 m/s, but still higher than the minimum fluidization velocity of sand particles of 750 mm, justifying the requirement of a confined bed, and low bed aspect ratios, between 0.25 and 0.9, to prevent excessively high fluid pressure drops. However, the bed aspect ratio increases significantly for higher granular material particle sizes, up to a ratio of bed height to diameter of 3 for a particle size of 1 mm and a TES demand time of 6 h.

5 PARTICLE FORMATION AND DESIGN

Interaction of nanofibrillated cellulose with papermaking additives

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(1) Chemical and Materials Engineering Department, Complutense University of Madrid, (2) Chenical and Materials Engineering Department, Complutense University of Madrid

Keywords | Nanocellulose hydrogels, papermaking, flocculation, microfibrillated cellulose, Nanofibrillated cellulose

Increasing attention is paid in the use of nanofibrillated cellulose nanofibers (NFC) as sustainable additive in papermaking to improve mechanical properties or barrier properties, for example. This could make paper to reach higher product quality overcoming its limitations. However, NFC can affect the production process as shown by the effect of NFC on retention and drainage processes (Merayo et al., 2016). Furthermore, the efficiency of NFC often depends on the dispersion grade (REF). Interactions of NFC with papermaking additives determines their effect on the process (Merayo et al., 2017). These interactions depend on the nature and dose of additive and on the morphology and surface of NFC and can aggregate or disperse NFC, which affects its efficiency. There are many studies on the effect of NFC on paper properties (Balea et al., 2020), but the interaction with process and product aids is less explored (Raj et al., 2016; Raj et al., 2017). Therefore, the aim of this research is to study the effect of the morphology of NFC on their interactions with different additives used in the papermaking process.

The interactions NFC-papermaking additives leading to aggregation or dispersion were studied by monitoring the chord length distribution of the NFC suspensions by means of a Focused Beam Reflectance Measurement (FBRM) probe and zeta potential measurements. Simplified gel point was also used to compare floc properties. Mechanical nanofibrillated nanocellulose (MNFC) and TNFC prepared by means of TEMPO-mediated oxidation, followed by high pressure homogenization, were used to study the effect of NFC size on the interactions.

Results showed that the use of TNFC on the process requires reoptimizing the dose of coagulant to avoid TNFC coagulation. However, MNFC had a lower affinity for coagulants than TNFC because of their larger size and lower anionic charge (Figure1).

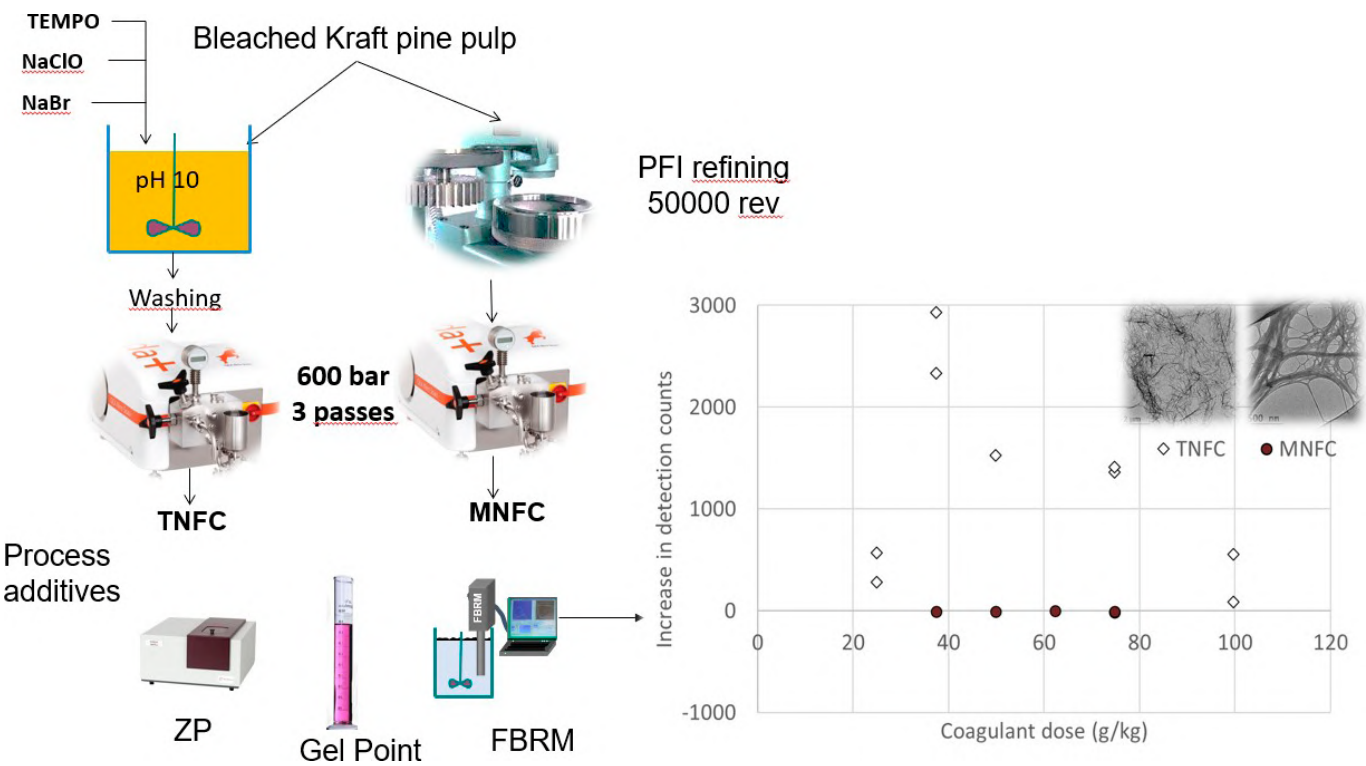


Figure 1. Experimental procedure with some results.

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Effect of color powder to mitigate photoinhibition of nitrifying bacteria under strong light irradiation

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Keywords | Wastewater treatment, Nitrifyfing bacteria, Light-shieldig powders, Photoinhibition

Recently, the nitrogen containing wastewater treatment using “microalgae-nitrifying bacteria consortium” has been attracting attention as an eco-friendly process. In the consortium, oxygen can be supplied by photosynthesis of microalgae, and nitrification by nitrifying bacteria is occurred. However, it is known that nitrifying bacteria suffer from photoinhibition by high light irradiation such as sunlight (Merbt et al., 2012). Therefore, Nishi et al. (2020) developed a “light- shielding hydrogel”, in which nitrifying bacteria is immobilized in hydrogel with carbon black particles to protect bacteria from light exposure. On the other hand, under strong light irradiation, “black color” light-shielding hydrogel may cause temperature to increase. Since high temperature could negatively affect nitrifying bacteria, not only to protect from light exposure but also to suppress temperature increase is needed.

This study investigates an alternative light-shielding material for carbon black suitable for preparing the light shielding hydrogel to fulfill the above requirement. To avoid severe temperature increase, we used powders with three colors (red, blue, yellow) other than black as light-shielding materials. First, by using each color powder dispersed water, temperature increase was measured under 1600 μmol photons m-2 s-1 of light irradiation. The results show the temperature increase was suppressed approximately 40% by using color powders other than black. Then, photoinhibition mitigation effect of each color powder was examined using nitrification experiments under 1600 μmol photons m-2 s-1 of light irradiation. In the experiment, nitrifying bacteria were immobilized in alginate hydrogel with each color powder (three colors and black for comparison). The results show that the nitrification efficiency using black powder was the highest of 69.1%. Yellow shows the second-highest nitrification efficiency at 11.4 %. It is expected that higher concentration of yellow powder added into light-shielding hydrogel could effectively improve the mitigation effect of photoinhibition.

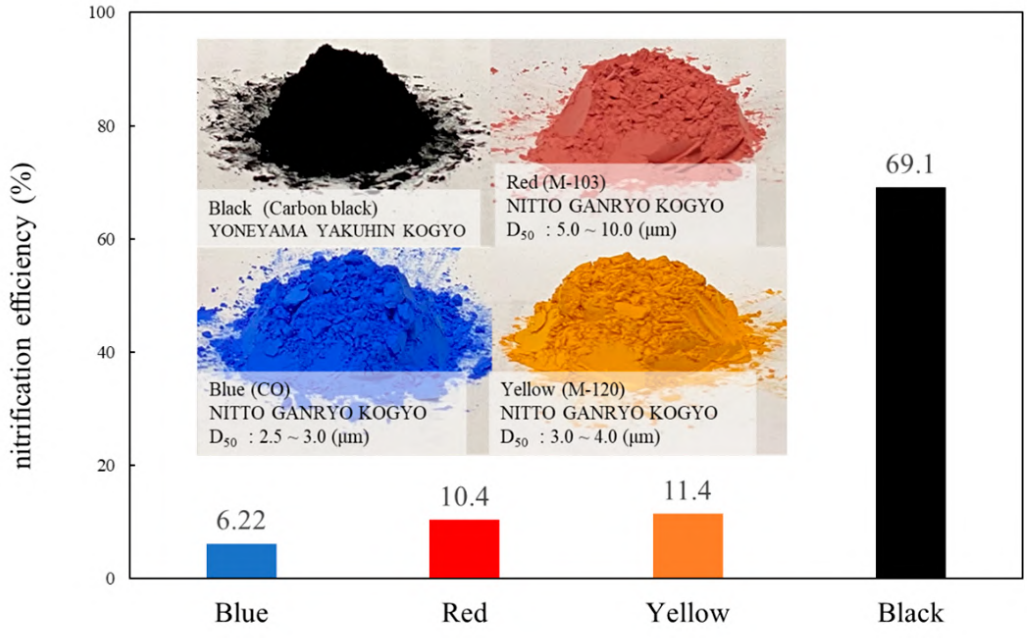


Fig1. Nitrification efficiency of light-shielding hydrogel using different color powders.

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Controlling particle properties by an integrated recycle-loop with a high shear environment during the anti-solvent crystallization of acetylsalicylic acid

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Keywords | Crystallization, Wet milling, High shear mill, Particle control, Process intensification, Recycle-loop

In the pharmaceutical industry, the particle properties obtained from the final crystallization step determine the processability and bio-availability of the active pharmaceutical ingredients. However, tuning particles into the desired properties without extra processing steps e.g., milling and granulating, is still challenging. Therefore, in this research the possibility to tune particles during the crystallization step using an inline high shear mill in an external recycle loop is investigated. An anti-solvent crystallization of acetylsalicylic acid was performed to proof the efficiency of particle tuning in this loop setup with respect to a traditional batch reactor. The high shear mill as well as the anti-solvent flow rate were investigated as potential particle tuning parameters. On the one hand, the loop setup resulted in smaller particles with a narrower particle size distribution relative to the particles obtained from the batch experiments. On the other hand, comparing repetitions of experiments in the loop setup resulted in more consistent particle properties. Both results were caused by the high shear environment in the mill which enhancing the breakage of the particles and increases the nucleation rate. Equipping a batch reactor with an inline high shear mill in an external recycle-loop is a promising method to obtain smaller particles with a narrower distribution.

6 PARTICLE SEPARATION

Adsorption of chlorobutanol from water by clay

Djebbar, Mustapha

Keywords | Adsorption, Freundlich, Langmuir, Clay, Toxic pollutants, beidellite, chemical compositions

objective of this study is to improve the physicochemical of matérial to remove toxic polluants present in water from natural clay. the clay used in this work is caracterized by UV-Visible, DRX, IR and déterminated the chemistry composition of clay. The effect of the experemental parametres of temperature, PH, contact time and cocentartion, retention of polluant by clay were studies. The Qmax of adsorption clay is 13 and 14 mg/g for the natural and treated clay and 19 for th beidellite.

Deflector wheel classifier for finest particles with integrated material sorting

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(1) Technische Universität Clausthal, (2) CUTEC Clausthaler Umwelttechnik Forschungszentrum

In various industries, the sorting and classification of fine particles is an essential step, e.g. in the metallurgical, pharmaceutical, food, coal, chemical, and cement industries. The deflector wheel classifier is considered the predominant separator in the industrial separation of fine particle fractions by sorting the particles according to their size through the flow forces acting on the particles. Using multiphase CFD simulations for the air and the particles in the classifier, the separation process is investigated and compared with measurements from experiments. In deflector wheel classifiers, flow vortices often occur between the blades, which impair the separation process. The aim is to improve the separation process by geometric changes to the classifying space or the blade geometries, such as the blade profile, the blade height, and the length of the blade. In the CFD simulations, particle tracking is implemented, the separation curves are calculated and compared, and validated with the existing separation curves from experiments for different flow parameters and different blade geometries. Furthermore, the influence of the changed geometries on the separation process will be investigated and the flow behavior in the classifier, as well as the particle movement behavior, will be explained. Based on the calculated trajectories, initial statements can be made about particle-wall collisions between the classifier blades and thus about triboelectric charging. Through these particle-wall collisions and also particle-particle collisions, the particles become triboelectrically charged. The triboelectric charge can then be used as a material sorting feature and particles from different materials can be sorted based on their charging behavior. The triboelectrically charged particles of different materials are fed as feed material into a sorting barrel after the wheel classifier, where the material sorting takes place based on the electrostatic separation concept. The first results of the electrostatic separation will be presented.

Physicochemical behavior of surface modified cellulose nanofiber sol by a time-dependent nuclear magnetic resonance (TD-NMR)

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Keywords | cellulose nanofiber, TD-NMR, hydrogen bond, adsorbed water, surface modification

【Problem statement】

CNF(Cellulose nanofiber) has a three-dimensional structure by forming hydrogen bonds among the fibers. It is an organic material that has functions such as gas barrier properties, high strength, and transparency. To maximize the CNFs' performance in the composite, surface modification is required to improve the wettability between the fiber surface and matrix. However, there is few reports regarding characterization of the surface modified CNFs' physicochemical behaviors.

【Objective】

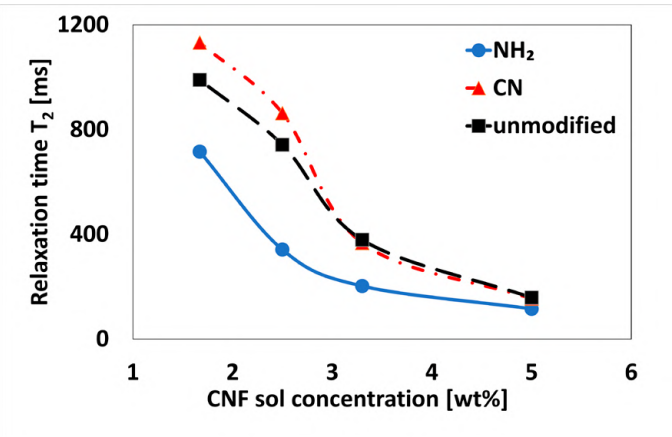
In this study, a time-dependent nuclear magnetic resonance (TD-NMR) was applied to investigate surface physical property of the modified CNFs. The relaxation time of the liquid molecules bound on the particle's surface is shorter than that of the free state liquid. That is, relaxation time of the modified CNFs will be affected by the modified groups. To deeply understand the relaxation time, rheological behavior and gas adsorption experiments were combined.

【Methods】

Commercial CNFs (Sugino Machine Limited) were chemically modified with aminopropyl silane and cyano propyl silane, which were denoted as NH2-modified CNF and CN-modified CNF, respectively. For the TD-NMR experiment, unmodified, NH2- and CN-modified CNF sols with different solid concentrations were used.

【Results】

Fig. 1 shows relaxation time (T2) for unmodified, NH2 and CN-modified CNF sols with different concentrations such as 5, 3.3, 2.?, 1.33, wt%. . With decrease in the concentration, T2 increased. By combination with the rheological behavior, the CN with highest T2 could have smallest interaction among fibers while the NH2 with lowest T2 could have the largest interaction among the fibers. The TD-NMR can be effective technique to characterize the surface-modified CNFs.



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Nanobubble-induced centrifugal field flotation of nanoparticles

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Keywords | Flotation, Separation at interfaces, Separation features, Multi-dimensional particle properties

Nanobubble-induced centrifugal field flotation of nanoparticles
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Classical froth flotation is a well-established process for the separation of product and gangue particles with a size above about 10 µm. For smaller particles, especially nanoparticles, their collision with macro-bubbles does not result in the efficient formation of stable bubble-particle-aggregates which are essential for froth flotation. Therefore, dissolved air flotation was applied to create nanobubbles directly on the surface of nanoparticles as a result of air oversaturation.

After a pressure cycle the liquid is separated from the froth and analyzed with a Nano-Particle-Tracking analyzer (NTA) providing information

on size, scattered light intensity and zeta-potential of single particles. Successful flotation could be deduced from, both, an increase of particle size and a reduction of particle number. In addition, the formation of free nanobubbles was observed. The relative importance of these effects depends on surface roughness.

Different kinds of chemical surface modification where applied (surfactants, collectors) and the pH value was shifted over the full range from acidic to alkaline. It could be shown that TiO2 nanoparticle flotation depends on, both, morphology and size. The flotation efficiency of smaller aggregates is lower than for aggregates in the size range above 200 nm.

The settling of nanoparticles and the rising of nanobubbles are both very slow processes, and therefore the observed formation of a particle-containing froth can only be explained by the (additional) interaction with macro bubbles. Therefore, against expectation, centrifugal separation was not required and further examinations at technically relevant concentration are carried out in a Hallimond tube.

After proving the working principle of nanoparticle flotation on, both, microscopic and macroscopic scale, its selectivity for different materials is addressed. Present work therefore focuses on the search of a reagent regime that stabilizes the particles against homo and hetero flocculation, but leaves their difference in hydrophobicity strong enough for selective growth of surface bubbles.

Quantifying the impact of the simplifying conventions within the ISO 16890 aerosol model

Redder, Christian (1)
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Keywords | Air filtration, ISO 16890, Modelling, Air Quality Monitoring

The ISO16890 series of standards on air filters for general ventilation has introduced a methodology based on the widely- used fractional aerosol classifications PM1, PM2.5 and PM10. Air filters are classified according to their efficiency values against these fractions with specific ePM1, ePM2.5 and ePM10 efficiency-values. For the determination of the efficiency values of ePM1 and ePM2.5 classified filters, the standard applies a weighted bi-modal lognormal distribution as model for the particle size/ weight distribution, which is parametrized by a parameter set referred to as “urban”. The model is well established (Hinds 1999), the parameters are based on worldwide measurements. The approach can be understood as a harmonization with air quality and environmental monitoring (AQM) data which commonly use PM2.5 and PM10 measurements. While this is certainly a big advantage for the practical use of the standard, it is important to be aware that the resulting filter classifications are based on simplifying conventions: **1.** The fraction particle size distribution range is limited by two sharp cuts (0.3µm and fraction size). The problem is illustrated in Figure 1. **2.** The efficiency values for ePM1 and ePM2.5 filters are based on only one particle size distribution (“urban”). The analysis of a 5year data set from North Rine Westphalia (Germany) indicates, that the variation of the particle size distribution, even within a limited area, is very extensive. **3.** The density of the aerosol is assumed to be constant over all particle sizes. Several investigations of the particle density suggest, that the density is dynamic and higher for the coarse mode (Zhao et al. 2017; Hu et al. 2012; Liu et al. 2015). The potential impact of these conventions on filter efficiencies and the comparability with standards using other definitions of the PM fractions should be quantified. Therefore extensive modeling using models of impactors, cell models of ePM1 50% and ePM1 90% air filters and systematically variation of the aerosol model parameters was carried out.

Conventions 1 and 2 were found to have notable impact. The Results are summarized in Figure 2.

Figure 1 – Illustration of different particulate matter fraction definitions

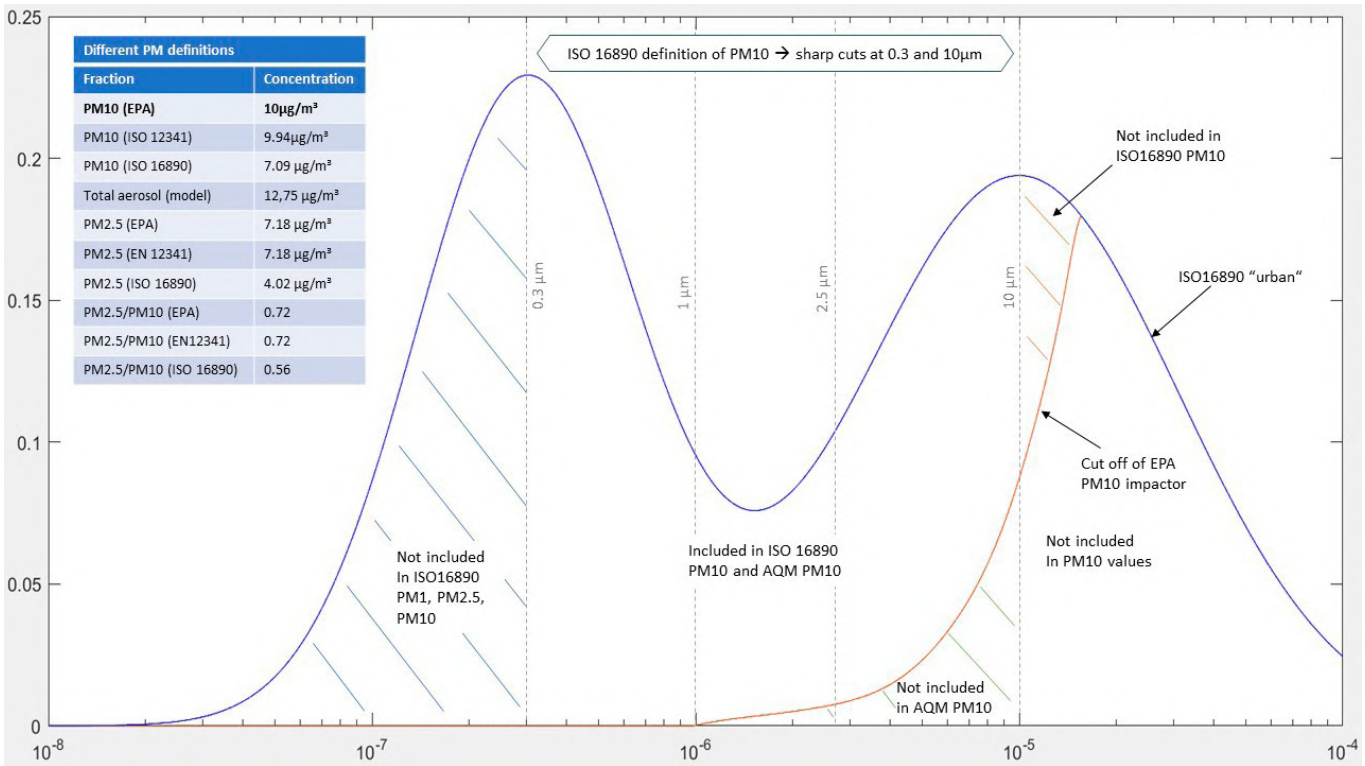


FIGURE 2 – Abstract/Results

Convention 1: The fraction range of the particle size distribution is limited by two sharp cuts (0.3µm and "naming" size)

Real world: A significant amount of aerosol mass is <0.3µm. The MPPS of most air filters is <0.3µm. AQM reference methods use impactors with characteristic curves to cut of the larger fractions.

Found impact: The integral mass of the ISO 16890 fraction definition is up to 44% smaller than with the AQM definitions. The weight between PM2.5 and PM10 is distorted. Filter efficiencies are overestimated up to 11.5% (ePM10) and 4.6% for the classifying fraction (ePM1). The impact is more significant for the ePM1 50% classified filter than for the ePM1 90%.

Conclusion: The upper cut-off is not critical. The 0.3µm cut-off leads to an overestimation of filter efficiencies, panders the impact of convention 2 significantly and reduces the comparability with AQM data. An extension to 0.1µm may reduce the impact.

Dependency

Convention 2: The efficiency values for ePM1 and ePM2.5 filters are based on one particle size distribution ("urban")

Real world: The particle size distribution is subject to strong variations. The comparison to the data set from NRW indicates, that the mean value corresponds satisfactory to the convention, but a very wide range of data do not.

Found impact: The ePM1 values of the ePM1 50% classified filter showed variation intervals up to 50.3% and 1.32% for ePM1 90%. The ePM1 values varied 20.2% (ePM1 50%) and 8.2% (ePM1 90%). The variation has the highest sensitivity towards the σ-value of the fine mode. The problem is pandered by the 0.3µm cut-off (Convention 1).

Conclusion: May be critical when data are used for exposure calculations.

Convention 3: The density of the aerosol is assumed to be constant over all particle sizes.

Real world: The density is subject to timely variations. The coarse mode has a tendence to higher densities.

Found impact: Depending on interpretation underestimation of ePM2.5 (2.5%) and ePM10 (3.8%) values of the ePM1 50% classified filter. All other values affected <1%.

Conclusion: Not very critical

7 AEROSOL PARTICLES

Size-dependent penetration of aerosol particles during simulated exhalation through the facemasks and respirators

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The usefulness of facemasks in reducing exhaled virus during COVID-19 pandemic has been questioned by showing a low correlation between new infections and mask mandate (Bundgaard et al., 2021). However, the complete understanding of the role of masks and respirators (MRs) requires a detailed insight into the physics of aerosol particles/droplets exhaled by infected individuals as a basic condition of aerosol spreading and later inhalation by bystanders (Dhand and Li, 2020; Sosnowski, 2021). Despite the good filtration efficiency of MRs in the laboratory tests during aerosol inhalation, MRs may be less efficient during exhalation due to improper MR fit to the face surface.

We show here the experiments done with the silicone phantom of the head and the system for the generation of exhalation flows of aerosol particles (0.5-20 /mu m), also during cough or sneeze. Particle size distribution (PSD) was measured with laser aerosol spectrometers to find the change of aerosol penetrating through a few types of MRs, including surgical and cloths masks and FFP2/FFP3 respirators. Tested MRs only partly eliminate large particles and allow the spreading of particles within the micrometer size-range. Particle removal from exhaled aerosol depends on the mask fit. Only some types of FMRs (mostly, the respirators) can effectively remove exhaled few-micrometer droplets as required to protect bystanders from inhalation of the infective aerosol. We concluded that non-valved respirators provide partial protection against SARS-CoV-2 spreading if they are properly worn to minimize the leaks.

Work supported by “IDUB against COVID” project granted by Warsaw University of Technology under the program Excellence Inititiave: Research University (IDUB).

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Developing affordable nano-biosensors for therapeutic drug monitoring of antibiotics

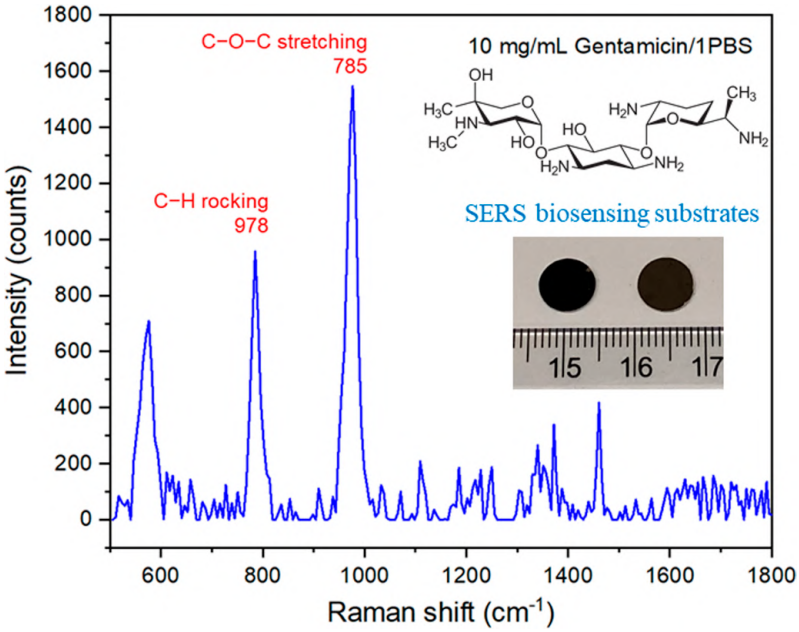
Li, Haipeng (1); Sotiriou, Georgios (1)
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Keywords | Flame aerosol technology, nano-biosensor fabrication, therapeutic drug monitoring, antibiotics

Antibiotics are intensively used in clinics to prevent and treat bacterial infections for maintain human health. Antibiotic overuse causes human health concerns because of antibiotic-resistant infections. For example, 2.8 million antibiotic- resistant infections including 35.000 deaths occur each year in US (Cristea 2017), and over 25.000 deaths are caused by antibiotic-resistant bacteria each year in EU (Düzgüneş 2021). Thus, therapeutic drug monitoring (TDM) of antibiotics to prevent side effects is essential for human health. Surface-enhanced Raman scattering (SERS) biosensors have promising application in the field of TDM of antibiotics, due to their advantages of label-free, high sensitivity, rapid diagnostic, simple operation, and point-of-care potential (Jaworska 2016). However, the practical application of SERS biosensors in clinical practice is still restricted by their sensing substrates, due to the challenges of poor-reproducibility and high-cost.

In this work, we used flame spray pyrolysis (FSP), an aerosol nanofabrication process famous for its scalability and reproducibility, to examine the massive and economic manufacturing of paper-based SERS biosensors. Totally around 900 SERS sensing substrates can be manufactured using flame nanoparticle deposition of 80 seconds and the fabrication cost is estimated as ~ 0.01 €/sample. The results have

demonstrated that the fast and quantitative Raman measurements of gentamicin in 1xPBS solutions can be achieved within 10 min using our SERS biosensors, offering a proof-of-concept for the TDM application in clinical practices.



Graphical abstract: Fast and label-free detection of gentamicin molecules using our SERS nano-biosensors. Flame aerosol fabricated SERS biosensing substrates.

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8 NANOPARTICLES: PRODUCTION, CHARACT. AND APPLICATIONS

Nanoscale zero valent iron, a useful tool for soil remediation

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(1) IMIDRA

Keywords | Soil pollution, metal(loid), availability, nanorremediation

Soil metal(loid) pollution is a worldwide issue due to its impact on ecosystems and human health. Thus, the development of effective soil remediation techniques should be prioritized. Recently, nanotechnology has enabled the generation of cost- effective and environmentally friendly remediation materials compared with some traditional technologies. Concretely, nanoscale zero valent iron (nZVI) can reduce metal(loid) bioavailability in soil (fraction which is available for (micro)organisms or plants). nZVI has a core-shell structure (core of Fe0 responsible for reduction processes; shell of iron oxides/hydroxides from spontaneous Fe0 corrosion, responsible for sorptions and/or complexations (Figure 1)). Research showed that the effectiveness of nZVI to immobilize metal(loid)s in soil strongly depends on the metal(loid) characteristics, soil properties, dose of nZVI, grade of pollution and presence of other pollutants. Assays carried out in a calcareous soil concluded that the effectiveness of immobilization was Cr>As>Pb>Zn>Cd, whereas Cd, Cu and Ni showed poor reduction of their availability, especially in acidic soils (Gil-Díaz et al., 2017, 2020). This strategy significantly reduced the toxicity of soil leachates as well as its phytotoxicity, allowing the growth of plants (Gil-Díaz et al., 2014, 2016a, 2016b). Monitoring of nZVI treatment at field scale in a brownfield highly contaminated with As and Hg for 32 months showed reductions of 70 and 80%, respectively (Gil-Díaz et al., 2019).

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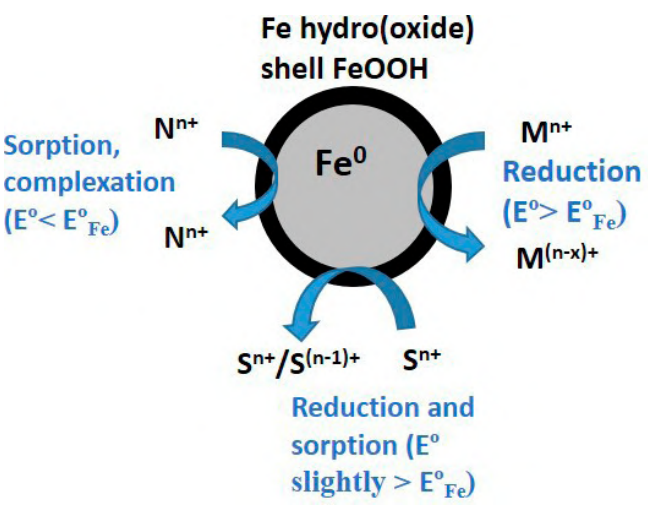


Figure 1. Conceptual model for metal(loid) removal with nZVI.

Iron nanoparticles for decontamination of Hg-polluted waters

Gil-Diaz, M. (1); Rodriguez-Alonso, J. (2); Maffiotte, C.A. (3); Millán, R. (3); Lobo, M.C. (1)
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Keywords | water, mercury, nanorremediation, nZVI,nFe3O4,nFeS

Mercury (Hg) is considered one of the most toxic pollutants to human health and ecosystems. The recent advances in nanotechnology have supposed the development of new material with exceptional small dimensions (< 100 nm) which confer them higher surface area, reactivity and different properties. In this regard, iron nanoparticles have been proposed as promising and even more efficient, durable, and cost-effective options for metal(loid) remediation compared to traditional technologies. The aim of the present study was to evaluate the capacity of three types of commercial iron nanoparticles (nanoscale zero valent iron (nZVI), nFe3O4 and nFeS) to remove Hg2+ from polluted water under a range of conditions. The three nanoparticles were characterized by Scanning Electron Microscopy (SEM), transmission electron microscopy (TEM), Fourier-transform infrared (FTIR), X-ray photoelectron spectrometry (XPS) and zeta potential). Mercury dissolutions were treated with different doses of nanoparticles (0.09-7.2 gFe/L), at different pH (3-9) and shaken for the selected time (0.5-48 h). Hg concentration was determined in the extract using thermal decomposition and atomic absorption spectroscopy with gold amalgamation. The three nanoparticles removed Hg2+ from aqueous solutions being the immobilization processes stable for at least 48 h. nZVI particles were faster and more efficient than nFe3O4 and nFeS (Figure 1). nZVI immobilized

94-98% of Hg2+ regardless of the pH. XPS revealed that the mechanism by which nZVI immobilizes Hg2+ depends on the dose and includes reduction to Hg0 and adsorption and/or complexation of Hg2+ to the nZVI shell. In contrast, adsorption was the main mechanism for nFeS and nFe3O4, which reached 85 and 80% of Hg2+ removal, respectively, depending on the pH and time (Gil-Díaz et al. 2021). The three iron nanoparticles emerge as potential candidates for the decontamination of Hg polluted waters and their selection depends on the efficacy required, the degree of pollution, the cost and length of the process.

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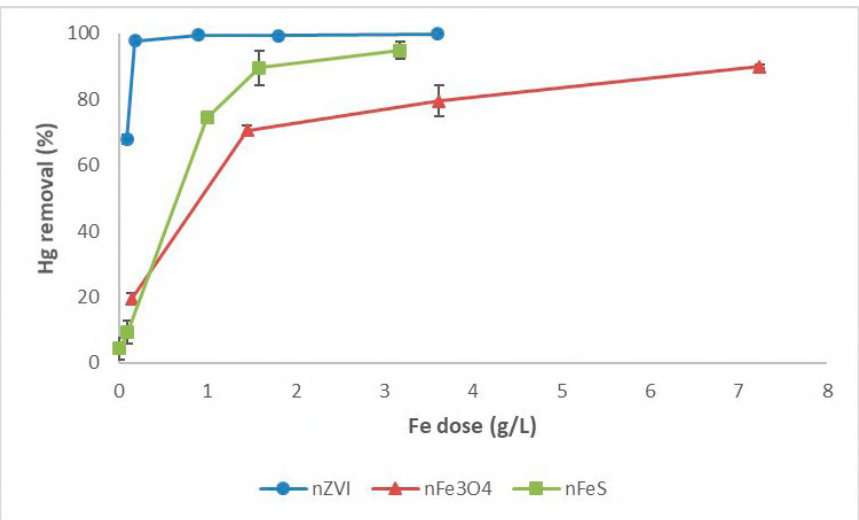


Figure 1. Percentage of Hg2+ removal from solutions after treatment with nZVI, nFe3O4 and nFeS at different dose.

Nanocelluloses as catalysts for the removal of recalcitrant contaminants from wastewaters

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Peracetic acid (PAA) is an alternative to H2O2 in advanced oxidation processes (AOPs), since it has a weaker O-OH bond, which means a greater potential to produce OH• radicals and thus to eliminate contaminants present in the wastewaters. Besides, PAA has less dependence on the effluent pH and produces non-recalcitrant and non-toxic by-products. However, it is necessary to use catalysts capable of activating PAA to produce OH• radicals. Transition metal ions were the first catalysts used for the activation of PAA. However, they are mostly toxic catalysts and difficult to reuse. Therefore, it is necessary to develop sustainable technologies capable of degrading recalcitrant contaminants using non-metallic catalysts, to minimize the environmental impact of the treatments. The common trend is the use of carbon materials, as activated carbon fibers, mainly due to their great activity, stability and reusability (Zhou et al. 2015).

In this context, cellulosic materials can be an interesting alternative to be used as catalyst for the activation of PAA. For instance, nanocelluloses (NC) show exceptional properties, such as a large specific surface that may provide the activation of the oxidizing agent. In the present work, three types of NC were used, cellulose nanofibers (CNF), cellulose nanocrystals (CNC) and bacterial cellulose (BC) for the PAA activation. The recalcitrant contaminant used to validate the process was Rhodamine B. The removal effect of both PAA and cellulose concentration (CNF, CB, CNC and cellulose fibers) was studied by response surface methodology (Figure 1).

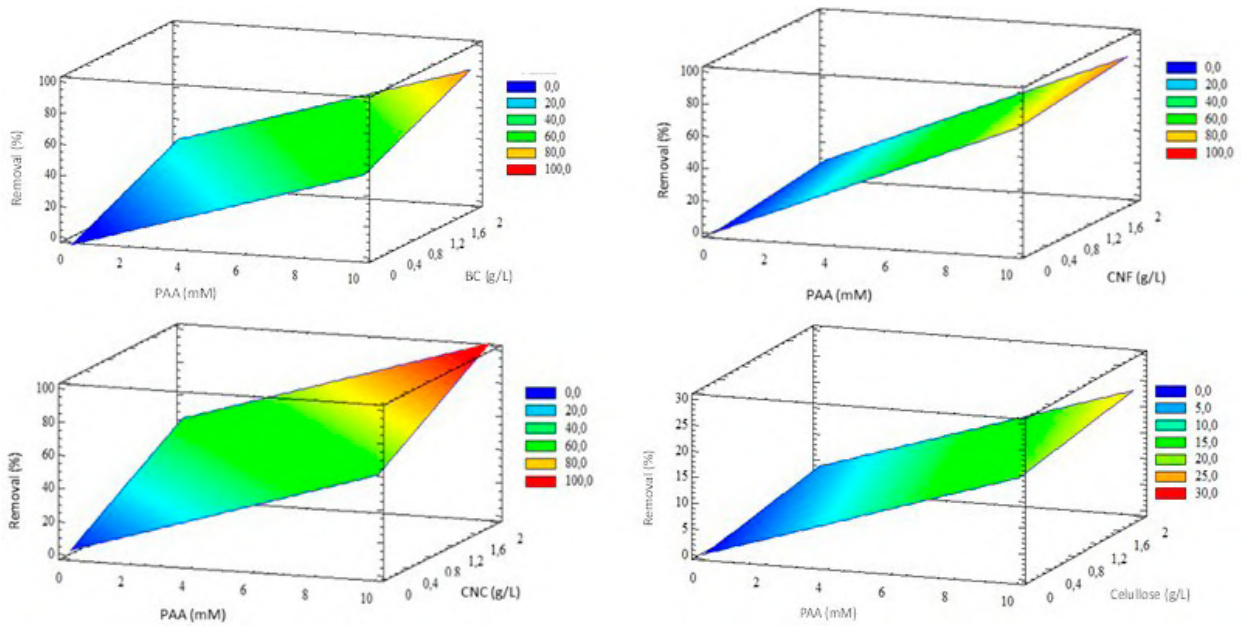


Figure 1. Response surface plot for removal of Rhodamine B

Results show that the oxidation of Rhodamine B, employing nanocellulose as a catalyst and PAA as oxidizer, is feasible (100% removal with CNC). It is concluded that CNC is a promising solution for removing recalcitrant pollutants from wastewater with minimum environmental impact.

Acknowledgements

Thanks to the project CTQ2017-85654-C2-2-R (MCINN).

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Sawdust as a raw material for nanocellulose production by means of bleaching

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Keywords | nanocellulose; cellulose nanofibers; pine sawdust; bleaching pretreatment; refining; high pressure homogenization; nanofibrillation yield; polymerization degree

The furniture industry generates around 14·106 m3 of sawdust in the UE [1]. There are different uses for sawdust, such as absorbents and ruminants feed production. However, part of the sawdust is still disposed in dumps or burned. Sawdust contains around 60-70% of cellulose [2] and it is in form of fine particles, which makes cellulose recovering easier and faster than that from wood chips.

The objective of this work is the recovery of pine sawdust as a raw material for the production of CNF [3] through bleaching and mechanical pretreatments prior to mechanical defibrillation. PFI refining and different bleaching pretreatments were tried: (i) bleaching with NaClO under three different conditions and (ii) bleaching with NaClO2. The five pretreatments were followed by high pressure homogenization [4].

Chemical composition and Kappa number of the pulps obtained after each pretreatment were analyzed and the CNF obtained from the homogenization of each pretreated pulp were morphologically characterized, by transmission electron microscopy. Degree of polymerization (DP), zeta potential (ZP), nanofibrillation yield and transmittance, at 800 nm, of the CNF gel were also determined (Table 1). Results showed that sawdust was a suitable raw material to produce CNF using bleaching as chemical pretreatment. Furthermore, the nanofibrillation yield increased with the bleaching intensity, giving rise to gels with a higher percentage of nanofibrils. Bleaching with NaClO2 decrease chemical degradation of cellulose and allowed keeping a large part of the hemicellulose and maintaining the high degree of polymerization of the raw cellulose while obtaining higher nanofibrillation yield than those for the strongest NaClO bleaching pretreatment tried, leading to similar kappa number.

Table 1. Characterization of the CNF suspensions

Pulp pretreatments:		PFI refining	Bleaching with NaClO			Bleaching with $NaClO_2$
Conditions:		20.000 rpm	4 wt,% NaClO, 10 min	4 wt,% NaClO, 60 min	8 wt,% NaClO, 180 min	8 wt,% $NaClO_2$, 180 min
CNF properties	Consistency, %	0.8 ± 0.04	0.9 ± 0.07	1.0 ± 0.02	0.9 ± 0.02	0.9 ± 0.01
	DP	1924 ± 6	1814 ± 7	1228 ± 5	498 ± 4	1930 ± 7
	ZP, mV	-15 ± 2	-23 ± 2	-27 ± 2	-25 ± 4	-21 ± 2
	Nanofibrillation yield, %	3.3 ± 0.1	10.8 ± 0.4	15.5 ± 0.1	16.4 ± 0.6	19.3 ± 1.3
	Transmittance, at 800 nm	14.7±1.1	35.1±5.0	30.2 ± 1.4	24.8 ± 3.6	30.3 ± 0.01

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Assessment of the rheological behaviour of cationic micro/nanofibrillated cellulose obtained by two distinct cationization methods

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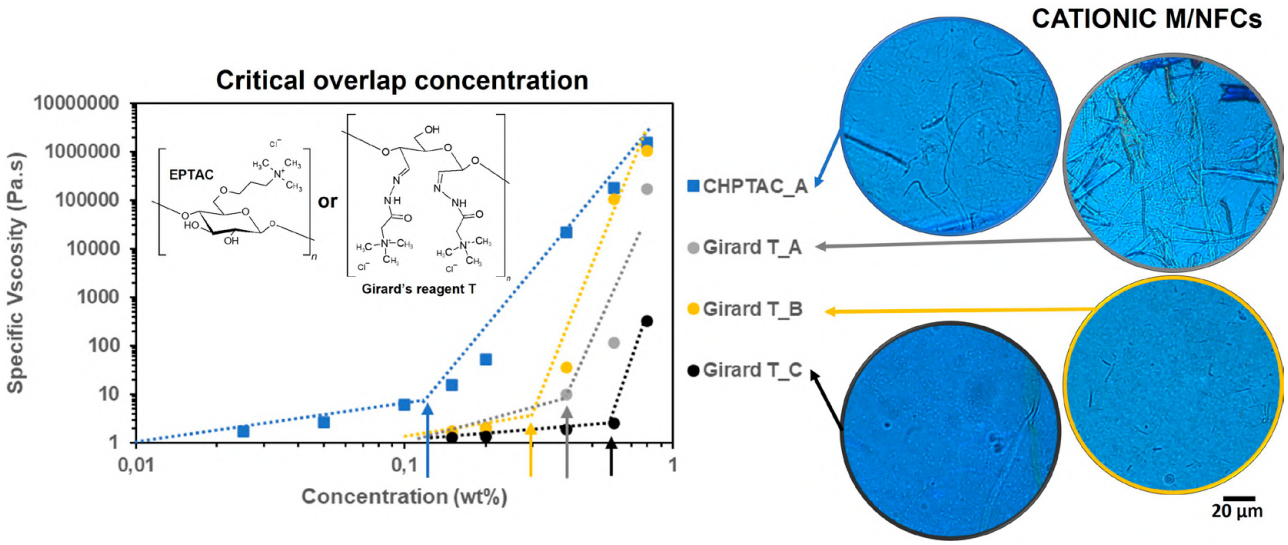
Keywords | Cationic Cellulose, Nanocellulose, Rheology

Cellulose in the form of micro/nanofibrillated celluloses (MNFCs), have been known to present the ability to form 3D networks by entanglement or alignment of the fibrils, leading to a complex rheological behavior. Owing to their chemistry and morphology (high aspect ratio), these materials can form high viscous aqueous suspensions, even at concentrations as low as 1 wt%. Such property enables MNFCs to serve as an effective rheological modifier in a wide range of fluid-based applications.

In the present work, cationic MNFCs were produced by quaternization of bleached Eucalyptus kraft pulp (BEKP) through a direct reaction with 2,3-epoxypropyltrimethylammonium chloride (EPTAC) in an alkaline medium or a dual step reaction (sodium periodate oxidation + cationization with Girard's reagent T) followed by mechanical treatment in a high-pressure homogenizer (Pedrosa et al. 2022).

The obtained MNFCs suspensions were submitted to rheological tests, with flow curves being obtained for distinct concentrations (0.03 – 0.80 wt%) in controlled stress mode with shear stresses ranging between 0.2 and 50.0 Pa. It was concluded that the cationization treatment plays a big role on the rheological behavior of the MNFCs suspensions. The fibers treated with EPTAC resulted in suspensions with higher viscosity compared to those obtained from fibers treated with Girard's reagent T.

This higher viscosity results from the strong 3D fibril network, which is related to a good fibrillation of the material and to the long and thin fibrils that can overlap. The minimum concentration from which the fibrils can more easily touch the neighboring fibrils and form a network (overlap concentration - c*) was also determined. The results confirm the higher tendency for the fibers treated with EPTAC to form a 3D network, with this sample presenting the lowest c* (Fig. 1.).



Comparison of photocatalytic activity of ZnO particles with different morphologies for persistent organic pollutants (POPs) degradation.

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Keywords | Persistent organic pollutants, Photocatalyst, Wastewater treatment, Zinc oxide

In recent years, pollution by persistent organic pollutants (POPs) is becoming one of the severe environmental issues which cause damage to human health. Photocatalysts have been attracting attention as one of the innovative POPs removal methods. The photocatalyst is a semiconductor that triggers an oxidation-reduction reaction by light exposure on the particle surface and can achieve mineralization of pollutants. ZnO is a typical photocatalyst and often synthesized and characterized with various particle shapes such as flowerlike (Qu et al., 2020), multi-shell (Chen et al., 2018) and rodlike (Wang et al., 2011). However, direct comparison of their photocatalytic activity under the same experimental condition is very limited.

Therefore, in this study, we synthesized three different ZnO particles with the shapes of flowerlike, multi-shell and rodlike, and compared their particle properties and photocatalytic activities for the same degradation target compound. The photocatalytic activity under UV light irradiation was evaluated by the degradation of 10 ppm 2,4-dinitrophenol (DNP) as model POPs.

Based on the FE-SEM observation and X-ray diffraction (XRD) measurement, it was confirmed that flowerlike, multi-shell and rodlike shape ZnO were obtained as expected. The DNP degradation rates were in the following order: rodlike < multi-shell < flowerlike (**Fig.1**). This order is same as the order of the BET specific surface area and opposite to that of the crystallinity. These results suggest that highest photocatalytic activity for DNP degradation in the flowerlike shape ZnO is mainly contributed by a very high specific surface area and not by crystallinity of ZnO.

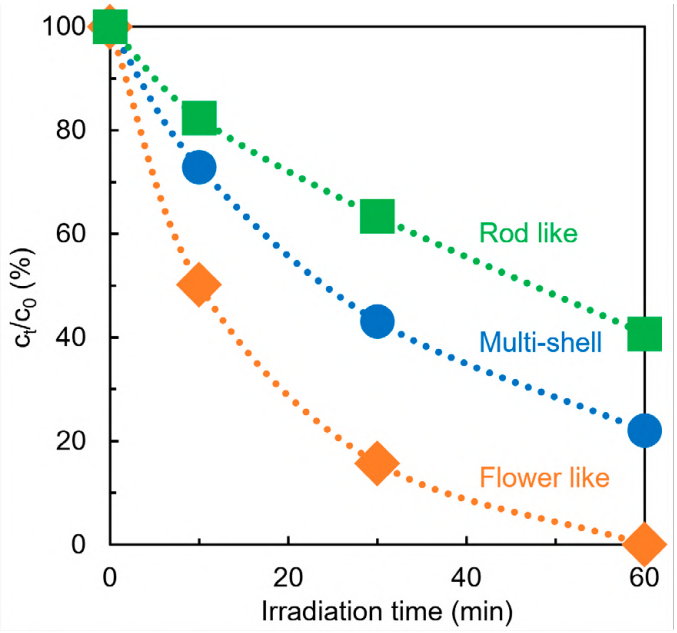


Figure 1. Photocatalytic activity of flowerlike, multi-shell, rodlike shape ZnO for photodegradation of DNP.

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Activated carbon/Fe3O4@CaO as a novel nano-catalyst to produce biodiesel from sunflower oil

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As of now, a major fraction of worldwide energy carriers is produced from fossil fuels. Given the diminishing non-renewable fossil sources and associated environmental impacts, biodiesel is an excellent alternative to fossil fuel (Seffati, Esmaeili et al. 2020).

Biodiesel is generally produced via a transesterification reaction between an oily biomass and methanol in the presence of either a heterogeneous or homogeneous alkaline catalyst (Borges, Díaz et al. 2012, Keihani, Esmaeili et al. 2018). In recent years, the use of heterogeneous alkaline catalysts in biodiesel production processes has been considered because of inherent advantages such as non-toxicity, reusability as well as ease of separation from the reaction mixture (Vujicic, Comic et al. 2010, Nisar, Razaq et al. 2017). Despite their these advantages, most of the available heterogeneous catalysts lack high catalytic activity and stability compared with homogeneous catalysts due to the lack of contact between the reaction medium and the catalyst (Ye, Zhukhovitskiy et al. 2017, Bharti, Chalia et al. 2021). One of the promising ways to overcome this limitation is to support these catalytically active centers on porous materials with large surface area such as activated carbon (AC) (Julkapli and Bagheri 2015, Narowska, Kułażyński et al. 2019).

In this study a novel, efficient and recyclable mesoporous activated carbon/Fe3O4@CaO solid alkaline nano-catalyst was synthesized. For this purpose, AC powder was prepared from almond shell and it was then impregnated with Fe3O4 nanoparticles to prepare AC/Fe3O4. The AC/Fe3O4 nanoparticles were then encapsulated with CaO, which was produced from domestic waste eggshell. The synthesized nano-catalyst was characterized using FTIR, BET, XRD, and FTIR analysis. The catalytic performance of AC/Fe3O4@CaO on the production of biodiesel via

the transesterification of sunflower oil has been studied. The maximum yield of biodiesel (95.58%) was predicted by the Response surface methodology-central composite design (RSM-CCD) at 65 0C, 4 wt% catalyst content, 120 min duration, and 9: 1 methanol to oil ratio. Finally, the physical and chemical properties of biodiesel were measured and the results were compared to ASTM D6751 and EN14214 standards. The synthesized biodiesel satisfied ASTM and EN standards.

Graphene enhanced composite phase change materials for high performance EV battery thermal management

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Keywords | Composite phase change material, formulation, property enhancement, thermal management, EV batteries

We report our recent work on the use of composite phase change materials (CPCM) for electric vehicle (EV) battery thermal management. A CPCM typically consists of a phase change material (PCM) for thermal energy storage, a structural material (SM) for shape stabilisation, and a highly thermally conductive material (TCM) for heat transfer enhancement.

This work uses decanoic acid as the PCM; the reason for the selection of decanoic acid as the PCM is mainly due to its phase transition temperature of 31.5oC, which is close to the middle of lithium-ion battery optimal operating temperature range (between ~15 and ~50oC). Olefin block copolymer is selected as the SM due to flexibility and durability for accommodating both thermal and mechanical stresses due to repeated volume change induced by heating-cooling cycles. Graphene is used as the TCM due to high thermal conductivity and hence a quick response to alleviate temperature rise of batteries. Several techniques are used to characterise the thermal properties of the CPCMs including thermal conductivity, phase transition temperature, specific heat capacity, and mechanical properties such as deformation resistance, tensile stress and compressive stress. Our optimised CPCM contains ~70% of PCM with significantly enhanced thermal and mechanical properties.

The formulated and characterised CPCMs are then integrated to a LiFePO4 based battery pack for repeated charging/discharging experiments. The results demonstrate that the use of CPCM could reduce the battery surface temperature rise significantly from ~19oC to ~3oC.

Facile synthesis of poly acrylic acid (PAA) modified inorganic nanoparticles (INPs) using in situ method

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(1) Soka University, Japan, (2) Kanagawa

Keywords | copper ferrite, inorganic nanoparticle, in situ method, polyacrylic acid, zinc oxide

Inorganic nanoparticles (INPs) have attracted much attention due to their unique properties. In addition, to suppress aggregation and/or to further extend their application, surface modification by polymer materials have been tried. Recently, a “in situ” method, in which INPs synthesis and polymer immobilization can be achieved simultaneously, have been developed (Tural et al., 2009). The method is much simpler and facile as compared to the conventional methods such as “grafting-to” methods. In our previous work (Hayashi et al., 2022), we successfully synthesized various polymers (e.g., polyacrylic acid (PAA)) immobilized magnetite nanoparticles. However, target INP tried so far was only magnetite, and synthesis temperature was limited to under 100 °C. In this study, synthesis of PAA-immobilized copper ferrite nanoparticles (PAA-CuFe2O4) and PAA-immobilized ZnO nanoparticles (PAA-ZnO) at high temperature using the in situ method was tried to evaluate the versatility of the method. PAA-INPs samples were synthesized by changing the input ratio of PAA/INPs from 0 to 0.1 [mol-PAA/mol-INPs] based on the theoretical yield of INPs. The X-ray diffraction (XRD) result shows both CuFe2O4 and ZnO were successfully synthesized for the synthesis temperature conditions above 100°C. Figure 1 shows the effect of PAA input ratio on INPs yield and immobilized PAA amount. The results suggest that although the CuFe2O4 yield was essentially constant, ZnO yield became highest at the PAA input ratio of 0.25. Regarding immobilized PAA amount, although the amount increased with the increase of the PAA input ratio for both cases, it reached maximum at the ratio of 0.5 for the PAA- CuFe2O4, whilst it kept increased up to 1.0 for the PAA-ZnO. These results show that the in situ method is highly versatile for immobilizing PAA on magnetic and non-magnetic nanoparticles.

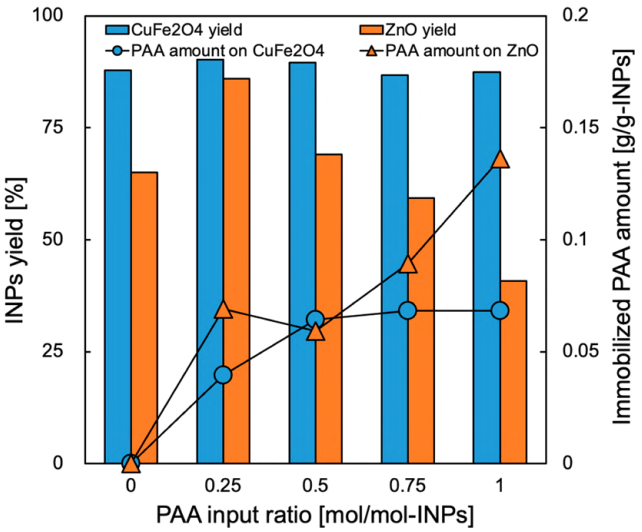


Figure 1. The INPs yield and the PAA immobilized amount

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Magnetic delivery and controlled release by magnetoliposomes

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Keywords | magnetoliposomes, magnetic nanoparticles, targeted drug delivery, controlled release, radiofrequency heating, liposomes, antibiotics, cancer

Magnetic drug delivery allows for a very precise and localized application of active substances. The commonly studied magnetite nanoparticles possess excellent responsivity to the magnetic field and moreover, allow for local heating when exposed to the external radiofrequency magnetic field. However, their capacity for cargo is very limited.

Liposomes, phospholipidic nano-sized vesicles are capable of encapsulation both hydrophobic and hydrophilic substances. In pharmaceutical applications, the liposomes are usually employed as drug carriers thanks to their ability to passively accumulate in tumor tissue in concentrations higher than plasmatic and hence increase the efficiency of the anti-cancer treatment. However, the release of the encapsulated drug is usually based on simple diffusion through the liposomal membrane and such a release mechanism is applicable only for substances with appropriate diffusion properties.

The release from the liposomes can be affected by temperature. When exceeding the phase transition temperature of the membrane, its fluidity increases and this facilitates the release of the encapsulated drug. The necessary temperature change can be achieved by combining the liposomes with magnetic nanoparticles.

The presented work describes the preparation, characterization, and utilization of magnetoliposomes composed of phospholipid-stabilized iron oxide nanoparticles bound to the membranes of liposomes carrying cargo. Such magnetoliposomes can be translocated in a stationary magnetic field and when reaching the final destination, the drug can be released by applying the radiofrequency pulse. The described system can be utilized for various medicinal applications. For example, magnetoliposomes may allow for high precision cancer therapy when injected into an arteriole supplying the tumor accessible to a magnetic field. Another possible application is the delivery of highly potent but toxic antibiotics to a spatially delimited infection site, which might be particularly useful for the treatment of pathologies caused

by multidrug- resistant bacteria.

Production of lignin nanoparticles from of kraft black liquor by solvent exchange process

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Keywords | Kraft lignin, black liquor, lignin nanoparticles, solvent exchange process.

Lignin is considered a high potential biopolymer and widely available as a kraft lignin in black liquor generating from the kraft pulping process. Kraft lignin accounts for almost 85% of total industrial lignin, which mainly undergoes burning to produce steam or electricity. However, it can be applied to produce high value and wide application products (e.g., lignin nanoparticles).

Lignin nanoparticles have been identified to use as UV absorbent, drug delivery, antioxidant, hybrid nanocomposite, and reinforcing agent. These nanoparticles can be produced by several processes like solvent exchange, self-assembly, polymerization, ultrasonication and acidification. Among these methods, the solvent exchange is a simple and efficient method that does not require any chemical modification. The schematic process is shown in Fig. 1.

In this work, lignin nanoparticles were produced using residual black liquor obtained from the hemicellulose separation process. For the hemicellulose recovery process, the black liquor was filtered using ultrafiltration membranes to concentrate hemicellulose. However, along with hemicellulose, some high molecular weight lignin was also retained during ultrafiltration, which was used for the preparation of nanoparticles. First, the hemicellulose was precipitated using acetic acid while lignin was kept dissolved using dioxane (Lisboa et al., 2005).

Further, the dissolved lignin, after separation of hemicellulose, was used to prepare the nanoparticles by solvent exchange process using dialysis membranes of 12-14 kDa molecular weight cut-off. The formation of nanoparticles, their size and morphology were confirmed using laser diffraction size analyser and transmission electron microscopy. The size of nanoparticles (d 0.9) was below 1 micron; however, the studies illustrate that the size of nanoparticles can be controlled by changing the concentration of lignin in the lignin solution. The nanoparticles preparation in this work, along with lignin valorisation, offered a solution to manage the residue in efficient way.

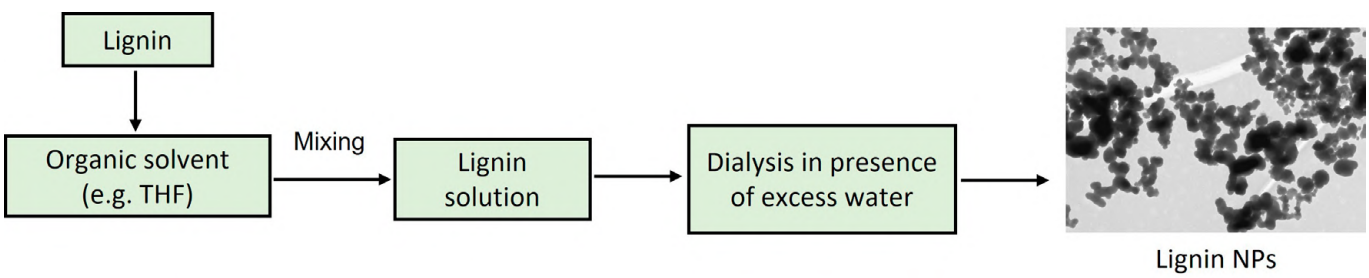


Fig. 1 Schematic representation of process for preparation of lignin nanoparticle by solvent exchange.

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9 MODELLING AND SIMULATION

An open-source framework for dynamic flowsheet simulation of solids

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Keywords | Flowsheet simulation; Process modeling; Solids handling; Open source software

In solids processing technology, it is very common to combine multiple processing stages with different types of apparatuses to obtain final products with desired particle properties. Therefore, the investigation and optimization of such processes is a complex and expensive task.

To address this challenge, a flowsheet simulation framework Dyssol (Skorych et al., 2017) is being developed. This software is capable of simulating the transient behavior of complex process structures in the area of solids process engineering. It applies the waveform relaxation approach, supplemented by convergence and extrapolation methods for rapid calculations of dynamic processes. The used transformation matrix technique (Skorych et al., 2019) allows proper calculation of multidimensional interdependent distributed parameters of solids. Dynamic and steady-state models are supported and can be used together in the same flowsheet. The available unit library can be extended to suit any specific application.

The system has a graphical user interface that allows to create and configure flowsheets and analyze simulation results. Dyssol comes with comprehensive online documentation, which includes a description of the interface, models documentation, explanations of the algorithms and methods, etc. Available simulation examples and tutorials make it easy to get started with the program and to develop new models.

Dyssol is written in the C++ language following the principles of object-oriented programming, which makes it easy to extend the system. The source code and executables are released under the BSD license, allowing distribution, modification, and use for any purpose (Skorych et al., 2020). The program is available under Linux and Windows, and its source code is accessible on GitHub (https://github.com/FlowsheetSimulation/Dyssol-open).

Acknowledgment

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Skorych, V., Dosta, M., and Heinrich, S., 2020. Dyssol – An open-source flowsheet simulation framework for particulate materials, SoftwareX, 12: 100572.

Discrete Element Modelling for Dosing Operation - Model Calibration and Validation

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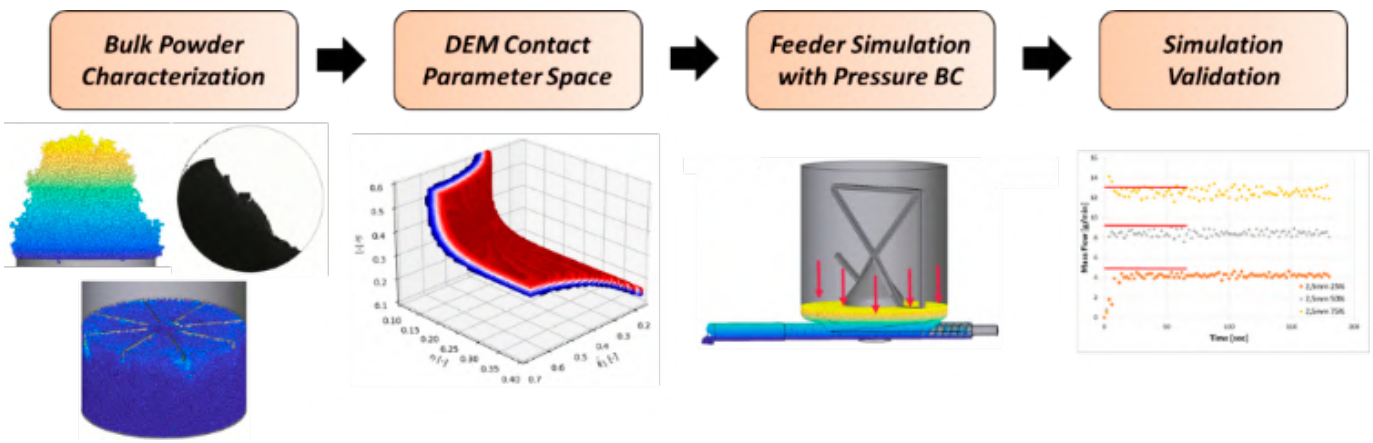
Keywords | DEM; cohesive powder; calibration; dosing; feeding; validation

In order to speed up pharmaceutical development and increase the availability of high-quality medicines, high-fidelity simulations based on physical principles have proven to be extremely valuable. The Discrete Element Method (DEM) is one such simulation technique that

has found widespread use in predicting the performance of pharmaceutical manufacturing processes (Bhalode and Ierapetritou, 2020). In such a simulation model, several particle-level contact parameters have to be correctly specified, or calibrated, in order to accurately mimic the flow behaviour of the bulk powder.

Until now, no efficient and generally applicable workflow for an accurate determination of contact parameters for cohesive powders has been established (Coetzee 2017). In this work, we develop a novel workflow for the calibration of DEM particle- particle contact parameters for cohesive powders (Forgber et al., 2022) that is applicable to any number of simulated characterization tests. Using latest machine learning and regression techniques, the workflow is demonstrated by matching the powder bulk behaviour under shear, compression and static and dynamic angle of repose for a model cohesive powder.

The contact parameters identified are further used in the simulation of a low-throughput dosing device (Three-Tec ZD 5 FB). In this simulation, we drastically reduce the number of particles in the simulation and, hence, the computational cost, by applying of a novel pressure boundary condition. Using this approach, we obtain good agreement with the experimentally measured mass flow. We further demonstrate the predictability of the model for various screw speeds and screws. The workflow and major findings which will be addressed in the presentation are summarized in the below Figure.



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Forgber, T., Khinast, J.G., Fink, E., 2022. A Hybrid Workflow for Investigating Wide DEM Parameter Spaces. Powder Technol. submitted.

Computational fluid dynamics simulation of a continuous spin freezing process in single vial unit

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(1) Ghent University

Keywords | Continuous manufacturing (CM), single vial unit (SVU) freeze dryer, computational fluid dynamics (CFD), spin freezing, good manufacturing practice (GMP)

Freeze-drying is a common method for increasing heat-sensitive and water-labile pharmaceuticals' stability and shelf life. The pharmaceutical sector is currently transitioning from batch manufacturing to continuous production. According to the recent FDA guideline regarding (Q13) continuous manufacturing (CM) of drug substances and drug products, maintaining the level of control in CM necessitates understanding of process dynamics. To understand the dynamics during the spin freezing step as part of a recently developed continuous freeze-drying process, a detailed model based on computational fluid dynamics (CFD) is developed for the single vial unit (SVU) freeze dryer. The CFD simulation provides local flow pattern inside the cooling chamber of the investigated system. The cooling gas flow pattern in the chamber is critical in the process of meeting certain good manufacturing practice (GMP) criteria in the pharmaceutical industry.

During the spin freezing step, glass vials are rapidly spun along their longitudinal axis. Cooling and freezing of the product is done by a sterile cold gas flow. The rotational speed and the velocity of the gas depend on the cooling rate of the product varies from 2000 rpm to 5000 rpm

and 2 to 20 m/s respectively. Three different modeling approaches were used to simulate the revolving vial: implementing rotational speed as a boundary condition, multi-reference frame (MRF) in which the rotational speed act as a source term in the Navier stokes equation, and moving mesh approach that mesh is actually rotating. Further, the CFD simulation results are compared to experimental data.

Using detailed mechanistic modeling improves understanding of process dynamics and aids in the optimization of existing designs. Here, for the first time, CFD simulation is implemented to understand the SVU spin freezing process dynamics. The optimal design will be proposed in future research based on the generated CFD simulation employing various optimization algorithms.

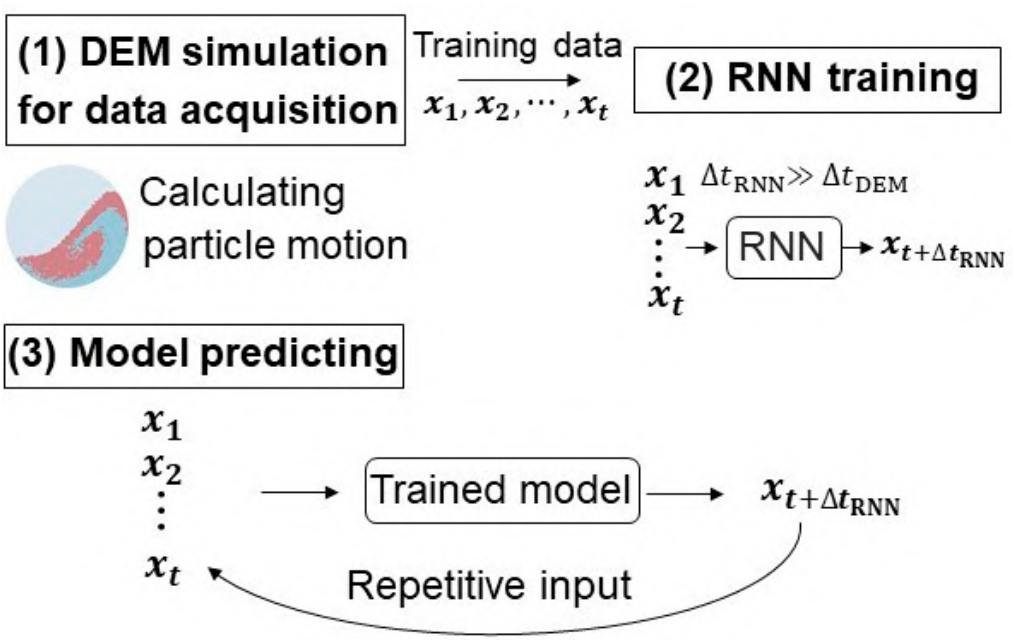
Development of ultra-fast computing method for powder mixing process

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(1) Department of Chemical Engineering, Osaka Metropolitan University, (2) Osaka Metropolitan University

Keywords | DEM, Machine Leaning, RNN, High-speed calculation

Powder mixing is ubiquitous in a wide variety of manufacturing sectors including chemical industries, and it plays an important role. The degree of mixing of powders (a measure of uniformity) has a significant effect on the quality of the final product obtained by processing the powders, and thus methods to predict the degree of mixing are required. Computer simulation using the Discrete Element Method (DEM) is a powerful tool for the analysis of powder mixing process. In a DEM, dynamics (position and velocity) of an individual particles are numerically solved. These calculations are performed at extremely small time-step (micro seconds order). Therefore, a large number of iterations are required to calculate the entire powder mixing process, and it is difficult to predict a practical mixing time.

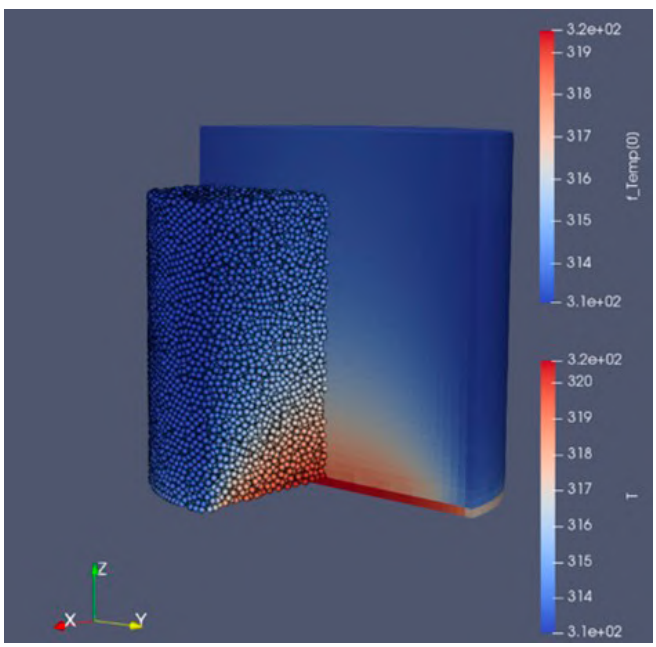
Recently, some studies on the combination of DEM and machine learning (ML) have been conducted. In these studies, macroscopic properties (angle of repose, kinetic energy, mixing index etc.) were predicted. However, there is no study on prediction method of an individual particle flow and mixing behavior. In this study, we developed a method for fast calculation of particle motions with large time steps by means of a machine learning (Fig. 1). Proposed model, which used recurrent neural network (RNN), was comprised of these steps. First, the quasi-steady state particle behavior was calculated by DEM (Fig. 1 (1)). Next, RNN was trained by the obtained particle behavior. At this time, the time step in proposed model was sufficiently larger than that in DEM (Fig. 1 (2)). Finally, the particle behavior was predicted using the trained model (Fig. 1 (3)). Proposed model was applied to a drum mixer and its performance was evaluated in terms of particle velocities, mixing behavior, and granular temperature.



Calibration of CFD-DEM Heat Transfer Model using Packed Bed Experiments

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(1) Johnson Matthey, (2) DCS Computing GmbH, (3) Newcastle University

Thermal processing is a necessary step in many granular materials manufacturing processes and may be used to affect a physical, chemical or phase transformation. CFD-DEM method is now widely used to model heat transfer in multiphase systems. For simulating processes involving powders, coarse graining is usually used to limit the computational effort due to the large number of particles involved however there is a lack of clear methodology to derive heat transfer parameters for these systems. The aim of this research is to provide a reliable methodology for calibrating coarse-grained heat transfer models. Transient heat transfer experiments are performed on packed powder beds with airflow, with three different materials. Unresolved CFD-DEM simulations are performed using LIGGGHTS-CFDEMcoupling. A process scaling methodology similar to Hou et al. [1] has been applied to scale the data from a few seconds of simulations to longer experimental timescales in order of hours. A sensitivity analysis for the effect of coarse graining factor, the gas flow rate and the applied stress on the bed, on the heat transfer in the packed bed is performed. This information is then used to calibrate the heat transfer model parameters using experimental data.



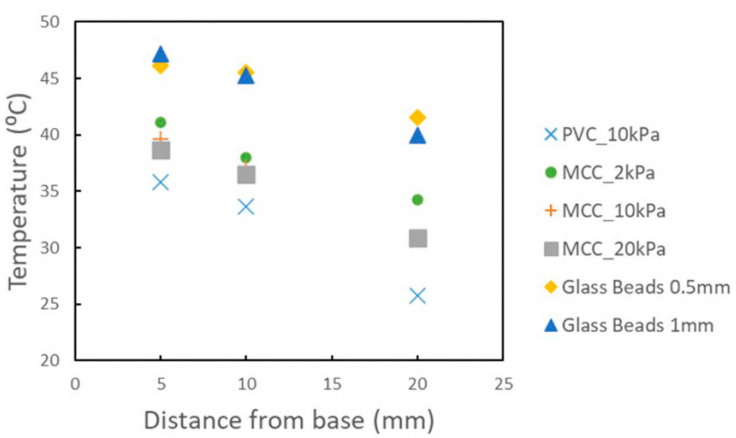


Figure. Experimental Results (a). CFD-DEM Simulation of a packed bed with gas flow through, (b) Bed Temperature profile for various materials at gas inlet set temperature 90 oC and gas flow rate of 6 lpm

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Development of a Hybrid Model using Evolutionary Approach in Particle Technology

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Keywords | Symbolic Regression, Generic Programming, Predictive Modelling.

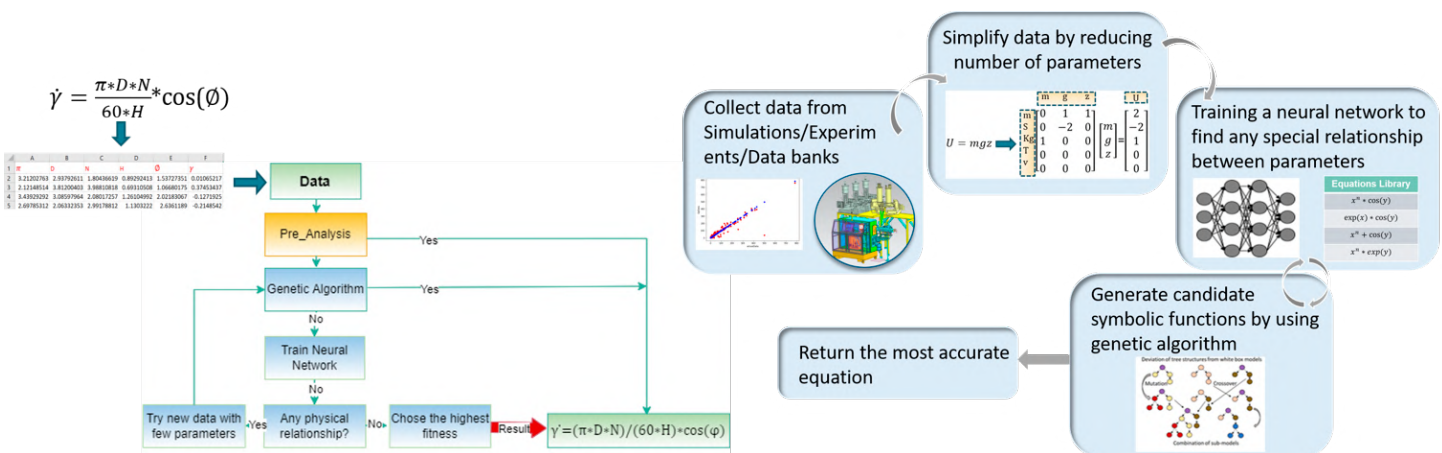
Governing equations are foundational in the process engineering field. Accurate models enable the understanding of physical processes, which in turn create an infrastructure for technology development. The traditional derivation of governing equations is based on first principles such as conservation laws, or universal laws such as gravity. Due to the limitation in knowing equations for many processes, reliance on first-principles derivations is rendered unreasonable. One of the most challenging tasks for both physics and artificial intelligence (AI) is finding a symbolic expression that fits the data of an unknown function (symbolic regression). Modern machine learning methods allow data-driven models to be obtained in a variety of ways. However, the more complex the model, the more difficult it is to interpret.

Functions that exist in many scientific processes often have some common properties. Discovering and exploiting these properties, existing physical or mechanistic models can help us to facilitate the modeling effort.

More precisely, in this work, a table of data whose rows are of the form {x1, ..., xn, y}, where y = f (x1, ..., xn) is prepared, and the goal is to determine the correct symbolic expression for the unknown function f by using a genetic algorithm.

Firstly, simplifying the data was attempted by applying a pre-analysis and finding the dimensionless parameters. Secondly, an attempt was made to find any existing physical properties among the data by taking advantage of a neural network.

Finally, by using a genetic algorithm, different symbolic functions were generated and the most accurate one was returned as the final expression. Next, the obtained function was applied as a white-box model which predicted the physics of our system.



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DEM modelling of swelling of grains

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(1) University of Surrey, (2) Newcastle University

Keywords | Granular materials, swelling, DEM

Many granular materials are hydrophilic absorbents, which change their volume as they absorb fluids. This phenomenon is called swelling and usually involves a large quantity of grains. To investigate the bulk behaviour of swelling granular systems, a DEM model that can predict the swelling of single grains with the deformation of the granular system is needed. In this work, a first order kinetics model was employed to describe the swelling rate of a single grain of super absorbent polymer (SAP). The model was implemented into the DEM code LIGGGHTS and successfully validated by using experimental data on the swelling of a SAP granular bed. The simulated particle profiles at different swelling time are shown in Figure 1a.

Using the validated DEM model, a numerical analysis of the effect of material properties on the swelling behaviour of granular media was performed. Experimental data of swelling of single particles were used to calibrate the swelling kinetics and capacity of a single grain of three different materials: super absorbent polymer (SAP), rice and microcrystalline cellulose (MCC Avicel PH102). The information was then employed to simulate the granular beds' expansion until the saturation state was reached. As expected, the swelling behaviour depends significantly on material properties; the fastest to reach its maximum expansion is the granular bed made of PH102, followed by SAP and rice (Figure 1b). However, as shown in the insertion in Figure 1b, the final bed expansion is greatest for SAP, followed by Avicel PH102 and rice.

The developed model can be employed to analyse consequences of volume changes in granular materials, such as segregation and heat generation

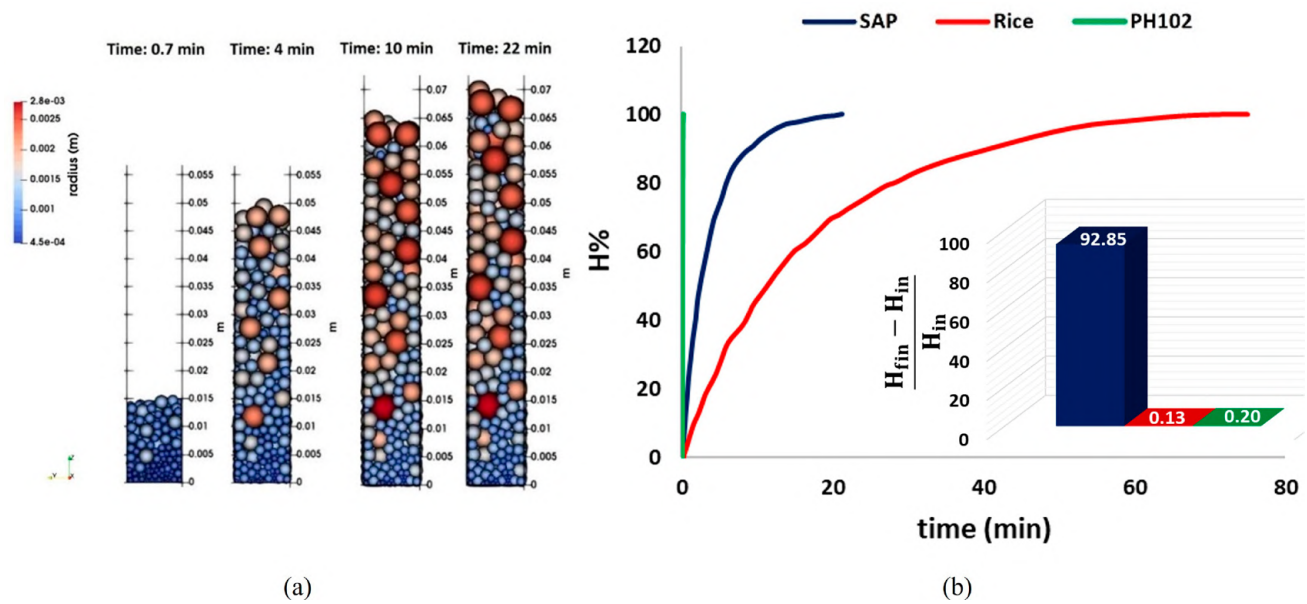


Figure 1 (a) Particle profiles at different swelling time of the granular bed made of super absorbent polymer (b) time evolution of the height (H%) of the granular beds made of SAP, rice and PH102. The insertion shows the swelling capacity of the granular beds.

DEM analysis of mixing performance of cohesive powders in a high shear mixer

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(1) University of Leicester

Keywords | DEM, cohesive powder, high shear mixer, subdomain mixing index

Cohesiveness is often ignored in Discrete Element Modelling (DEM) of mixing of different powder materials. However, cohesiveness is a primary characteristic that causes the formation of agglomerates during flow and mixing of complex powder formulations. Inadequate mixing can have significant practical implications, therefore it is essential to understand the dynamics of the mixing process in the presence of cohesion. This study employs DEM to investigate the effect of cohesiveness on the mixing performance of a high-shear mixer, showing that cohesive particles in large quantity can significantly hinder the mixing process.

The adhesive contact law of Johnson-Kendall-Roberts (JKR) [1] and the Hertz model are employed to compare the flow of cohesive and non-cohesive particles. The quality of mixing is quantified using a non-sampling mixing index, Subdomain Mixing Index (SMI) [2]. The mixing quality is analysed considering Active Pharmaceutical Ingredient (API) particles with properties within the representative range for pharmaceutical

powders. The study examines the mixing performance observed from the SMI data obtained for both cohesive and non-cohesive particles. The flow regimes are also characterized for different powder fill ratios to determine an optimum loading.

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Dispersing Carbon Black in Cathode Slurries – a Numerical Approach

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(1) Institute for Particle Technology

Keywords | Battery, Carbon Black, Dispersion, Rheology, PBM

Batteries play a vital role in the energy transformation from fossil fuels towards more sustainable electrochemical storage systems. Due to the increasing demand and funding over the recent years, great progress has been made in the area of cell capacity, durability and production efficiency. In the future, the cell performance increases constantly and new material compounds need to be available.

In the area of battery research, the electrode slurry rheology is an important property. Not only for the applicability in the production steps e.g. the coating, but also for the electrochemical performance of the cell. Cathode slurries consist of the active material (e.g. NMC) and the solvent (e.g. N-Methylpyrrolidon (NMP)). A binder (e.g. polyvinylidene fluoride (PVDF)) is used to increase the mechanical stability of the layer and the adhesion to the aluminum current collector. Carbon Black (CB) particles form a matrix with binder and act as a conductive agent. During the dispersion step of the electrode slurries, the fractal CB is further disagglomerated and dispersed and into smaller particle sizes and it has a significant impact on the viscosity.

The poster shows a numerical approach based on the population balance method (PBM) for a dispersion process in a planetary mixer. It addresses the effect of dispersion process on CB size distribution according to a state-of the art NMC-622 cathode slurry. Therefore, an existing model for high viscosity and highly filled nanoparticle suspensions is adapted for the used planetary mixer. The research demonstrates the required physical properties for the simulation of the dispersion process. Furthermore, the poster dedicates the difficulties and challenges in obtaining data. First results show a good agreement between the experiments and the PBM.

High-fidelity CFD-DEM simulation of a capsule-based dry powder inhaler

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(1) RCPE GmbH, (2) IPPT, Graz University of Technology

Keywords | High-fidelity CFD-DEM simulation of a capsule-based dry powder inhaler

The performance of dry powder inhalers depends on the interplay of powder properties, device characteristics and patient spirometric capacity. Data on the air flow and powder behavior in the device are difficult to obtain experimentally due to the small spatial and temporal scale of the system. Instead, they can be estimated in computational studies of the inhaler device.

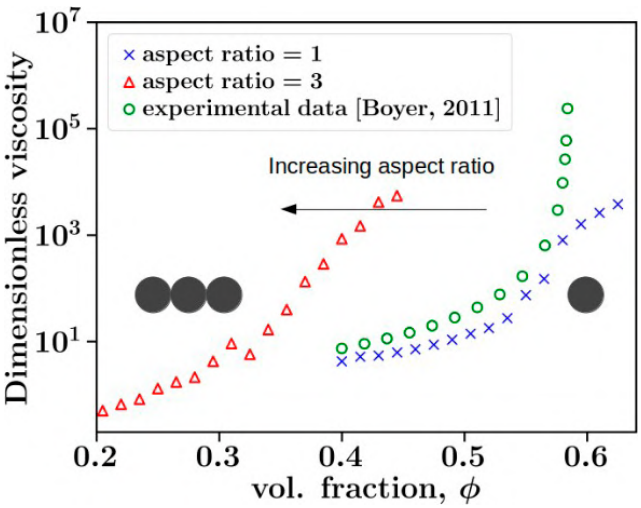
A CFD-DEM study of a lactose carrier powder in the capsule-based Aerolizer® inhaler is presented. Two-way coupling between CFD and DEM is necessary as the airborne particles alter the air flow field in the device. It is shown that the powder flow through the pierced capsule holes depends on the orientation of the capsule holes regarding the air inlets. The extent of agglomeration of the powder leaving the capsule is higher in a simulation at an approximated patient inhalation profile than in a simulation at a constant flow rate, similar to what is typically used in impactor testing.

The presented fully coupled computational fluid dynamics – discrete element method (CFD-DEM) simulations can provide insight into the phenomena governing inhaler performance. The results demonstrate that realistic inhalation profiles affect the performance of the inhaler and cannot indiscriminately be substituted by constant flow rates in device studies.

Dense suspension rheology: From spheres to rods

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In nature and in industrial applications, most particles are non-spherical, yet current research into dense suspensions has focused on spherical particles. The geometry of a rod-like particle will give more varied frictional contact physics between the particles (i.e. rolling and sliding friction) and there is a possibility for orientational order or disorder [Trulsson, 2018]. Both effects are likely to influence the rheology [Wyart and Cates, 2014, Rathee et al., 2020]. For instance, experiments show that an increase in aspect ratio (the ratio of the length to diameter of a particle) may lead to jamming at lower volume fractions [James et al., 2019]. My aim is to establish what new physics and rheological behaviour arise when considering rod- like particles compared to spheres. I will ultimately develop a particle-based simulation model for ellipsoids with variable aspect ratio. The protocode, for aspect ratio 1, produces viscosity against volume fraction data that compare qualitatively with expeiments (figure) [Boyer et al., 2011]. The next steps are the implementation of a rotational velocity verlet scheme [Rozmanov and Kusalik, 2010], contact detection between ellipsoidal particles and implementing corrections to the stokes drag equations. Meanwhile, a basic model with spheres glued together to replicate rods demonstrates a clear shift in the jamming volume fraction with an increase in aspect ratio (figure). However, this model likely misses out some physics due to the incomplete shape created by rods made from spheres.



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Wyart, M. and Cates, M. (2014). Discontinuous shear thickening without inertia in dense non-brownian suspensions. Phys.Rev.Lett., 112:098302.

Model of particles interaction in rotary kiln

Mulenko, Martin (1); Skocilas, Jan (1); Ayas, Mehmet (1)
(1) CTU in Prague

Keywords | DEM, Rotary Kiln, Particles, Keramzite

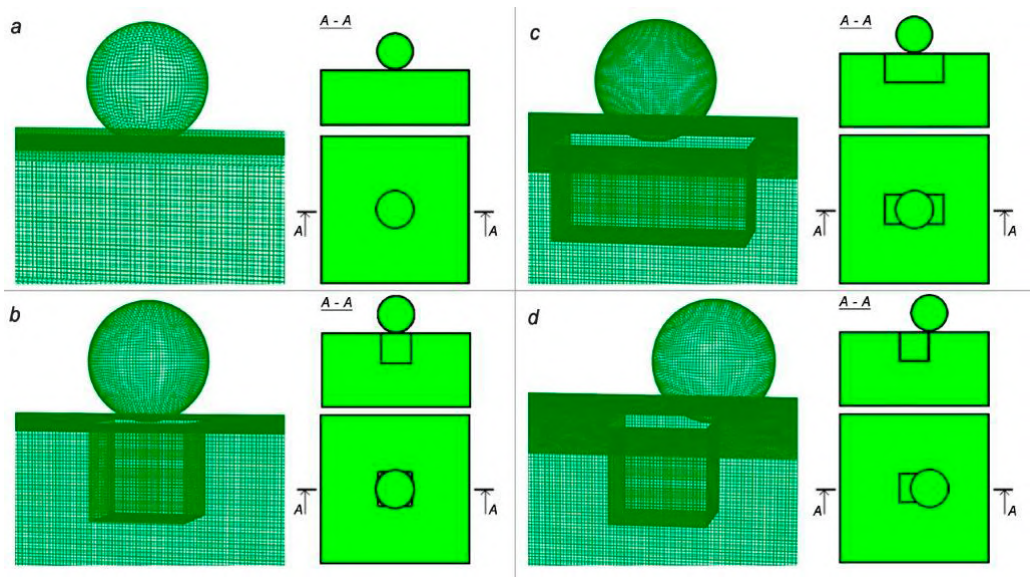
Investigation of collision mechanisms of fine particles with porous surfaces

during cold spraying

Aleksieieva, Olha (1); Toporov, Andriy (2); Tretiakov, Pavlo (2); Bozoglu, Mustafa (1); Antonuk, Sergiy (1)
(1) Institute of Particle Process Engineering, Technische Universität Kaiserslautern, (2) Chair of Chemical Process Engineering and Apparatus Design, Donetsk National Technical University

Keywords | finite element method, impact, thermal spraying method, microdefect, protective particulate coating, damaged surfaces

The component surfaces of industrial equipment are exposed to permanent erosion, wear and corrosion processes due to mechanical and thermal stresses, interactions with solid particles or chemical reactions with an aggressive environment. The restoration of component proper state can be achieved by applying a protective particulate coating locally onto the damaged surfaces using a thermal spraying method. Thermal spraying is a deposition process to create homogenous or heterogeneous coatings on the surface of components with various sizes and forms (Nikolaus, M et al., 2017). This study focuses on the cold gas spraying process, in which the necessary binding energy of particles to surface is mainly achieved by the high kinetic energy during the impact on the surface (Breuninger et al., 2019). The impact of the single microparticle on a microdefect in the damaged surface was studied by numerical simulations with finite element method. Different rectangular microdefect slot forms and contact scenarios were analyzed (Figure). The Johnson-Cook model was applied to consider the plastic deformation of contact partners occurring during high-velocity collision at different velocities (500 - 900 m/s).



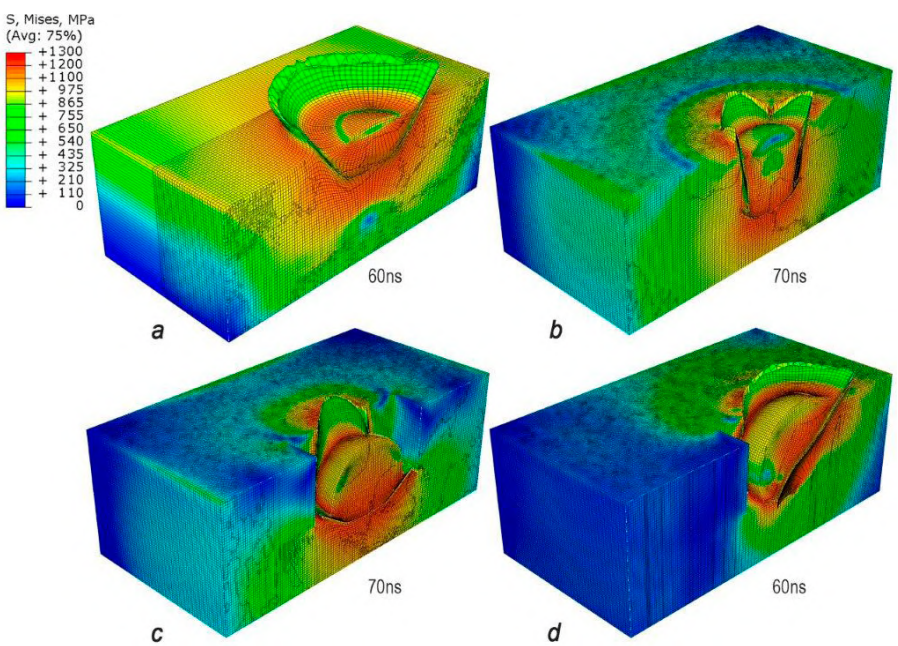


Figure. (left) 3D FEM models and (right) the von Mises stress distributions of simulated collision cases of particle with the substrate: a) without microdefect; b) with cube-shaped microdefect; c) with rectangular-shaped microdefect; d) with the particle collision on the edge of microdefect.

The different impact scenarios result in different temperature and stress distributions in the contact zone, penetration depths, and deformations during the collision. The obtained results are compared with the previously performed experiments (Breuninger et al., 2017) and used for the determination of the optimal process parameters to reach a strong bonding of particles with the damaged surfaces.

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Nicolaus, M., Möhwald, K.; Maier, H.J.: A combined brazing and aluminizing process for repairing turbine blades by thermal spraying using the coating system NiCrSi/NiCoCrAlY/Al. J. Therm. Spray Technol. 26 (2017) 1659-1668.

Second-order moment method of kinetic theory of granular flow for particle simulations in the Eulerian framework

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(1) National Institute of Clean-and-Low-Carbon Energy

Keywords | SOM-KTGF; fluid-particle; second-order moment; dense concentration, median concentration

The numerical method, second-order moment method of kinetic theory of granular flow (SOM-KTGF) is proposed to demonstrate the capability of this model on the simulation of the particle flow in the Eulerian framework. The model SOM- KTGF is capable to simulate the particle flows not only in a dense concentration by considering the inter-particle collisions, but also in a median concentration where the motion of the suspended dilute particles cannot satisfy the Boussinesq approximation owing to the non-equilibrium condition of the fluctuation.

In this poster, we present the studies of various fluid-particle flows simulated using SOM-KTGF. For example, the simulation of the fluid-particle flow in the spouted fluidized bed applied the method in a high concentration of particles where the friction stress existed. The prediction of particle-laden flows in the horizontal channel presents the capability of SOM-KTGF predciting particle flows in the median concentration where the particle suspended in the region. Furthermore, the clusters and fluid-particle fluid dynamics in fluidized riser can demonstrate that the SOM-KTGF model is able to simulate the complex particle flows, where co-exist the suspended particles (in median concentration) and clusters (particles' assembly in dense concentration) in the same fluidized riser.

The research demonstrate the capability of SOM-KTGF in the prediction of particles from the dense to median concentration in a higher fidelity.

10 STED* IN PARTICLE-BASED MATERIALS AND PRODUCTS

Thermal-mechanical energy harvesting through liquid-solid nanotriboelectrification: shock absorber in EV

Zhao, Weiwei (1)
(1) University of Birmingham

Keywords | Nanotriboelectrification; Shock absorber; Energy Harvest and Conversion: EVs

When aqueous solution is forced to into a cycle of entering and exiting from a hydrophobic nanoporous material, the liquid and solid acquire opposite electric charges because of nanotriboelectrification. These charges can be collected and generate current. In this work, the energy driving the liquids entry to and exit from the nanopores is the vibrational energy, i.e. cyclic displacement inside the shock absorber when driving. This process also passively harvests ambient heat when the fluid is pushed into the nanopores. Thus, mechanical and thermal energy, in terms of vibrational displacement and ambient heat energy, have been harvested and converted into electric charges. The nominal conversion efficiency of electric power is approximately 25% when compared to the vibrational power absorbed. This work will contribute to increasing the energy efficiency of a broad range of commercial apparatuses including EVs, reduce their energy consumption, and contribute to the overall goal of the world towards a carbon dioxide neutral society.

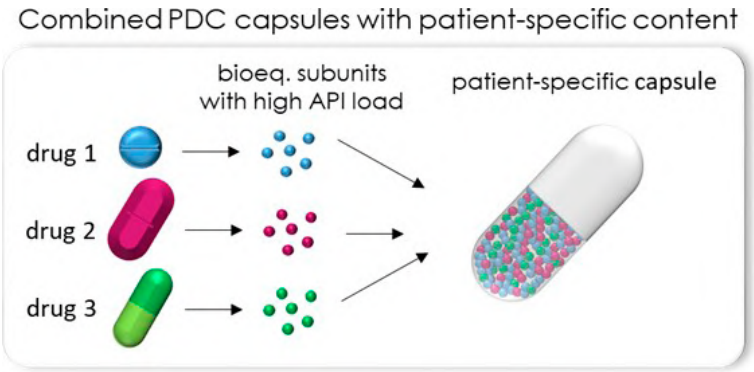
Personalized Dose Combinations: New Flexible Alternative Dosage Form to Fixed-Dose Combinations

Sonntag, Erik (1); Mutylo, Elizaveta (1); Štěpánek, František (1)
(1) UCT Prague

Keywords | Personalized medicine, MUDS, Polymorbidity, Fixed-dose combinations

Personalization is experiencing a rapidly growing trend in various industries. Mainly owing to the advent of Industry 4.0, it is possible to create products with a tremendous level of customization while maintaining a large product throughput. With a few clicks in the application, one can easily specify the desired features of a new car, eyewear, or breakfast cereals. This trend is also gradually entering the pharmaceutical industry environment; however, the implementation here is slightly slower due to significant legislative restrictions. Nevertheless, it is in the pharmaceutical industry where personalization makes enormous sense.

This work is devoted to introducing a new dosage form called “personalized-dose combination” (PDC). Such a form represents a formulation in which multiple drugs are combined based on a prescription of the individual patient. Combining several drugs into just one formulation has been used in pharmacy for a long time, as it leads to a reduction in the number of dosage forms used by polymorbid patients. Several systematic reviews then show that every reduction in the dosage forms that a patient must take leads to a significant increase in adherence. Currently, fixed-dose combinations (FDCs) are available on the market, representing multi-drug formulations. However, FDCs have a defined composition and dose of individual drugs that cannot be changed. PDCs then represent a flexible alternative to FDCs, as their composition can be variably adjusted according to each patient. The PDC is a MUDS (multiple-unit dosage system) formulation in which various types of particles such as minitablets or pellets are mixed into one capsule. Combining several different particles makes the desired formulation flexible regarding individual drugs’ identity, potency, and release profiles. With a suitably chosen composition and type of the particles, it is possible to prepare almost any combination of drugs “tailored” for a particular patient while maintaining the benefits of industrial production (speed, quality, traceability). The content of the presented research is the introduction of the three developed prototypes of minitablets, the properties of which have been modified to create a PDC formulation. Furthermore, the device developed for continuous production of these combined MUDS capsules is demonstrated.

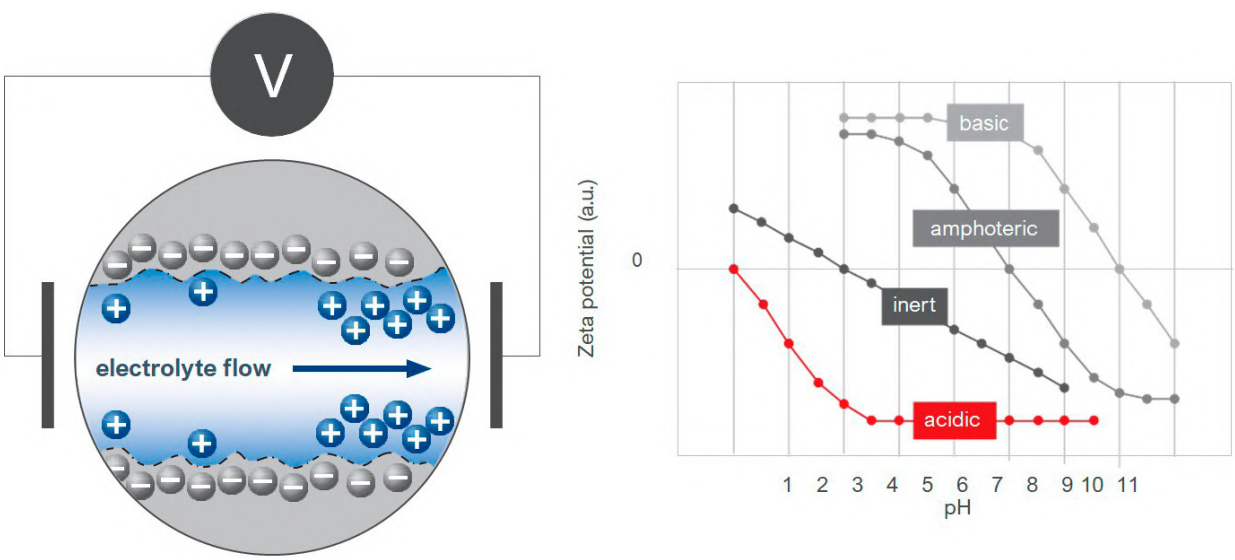


From coarse particles to nanoparticles-functionalized surfaces: how the zeta potential can unravel new applications

Brasil, Henrique (1); Luxbacher, Thomas (1)
(1) Anton Paar GmbH

Keywords | zeta potential, streaming potential, surface chemistry, functionalized materials

Detailed knowledge of surface properties can often promote the development of new materials in chemical and technological applications, biology, or medicine. Surface chemistry is a common indicator if a product is suitable for an intended application. A time-efficient and sensitive technique to evaluate surface properties is the zeta potential, which represents the surface charge that occurs in an aqueous solution when reactive (functional) groups dissociate on hydrophilic surfaces or water ions adsorb onto hydrophobic surfaces. Varying the pH value of the aqueous phase influences the equilibrium between dissociation and adsorption processes, giving insights into the chemical behavior of the surface. Here we show three examples of how the zeta potential measured by streaming potential or streaming current can be used to characterize coarse particles and functionalized surfaces. First, we show how the zeta potential can be used to track clay stabilization for its application in oil and gas recovery. Then, we demonstrate how the zeta potential of nanoparticles and textiles allows for setting optimized conditions for the particles' adsorption. Finally, the zeta potential was used to evaluate the adsorption of antimicrobial chitosan nanoparticles to functionalize foils for packaging application.



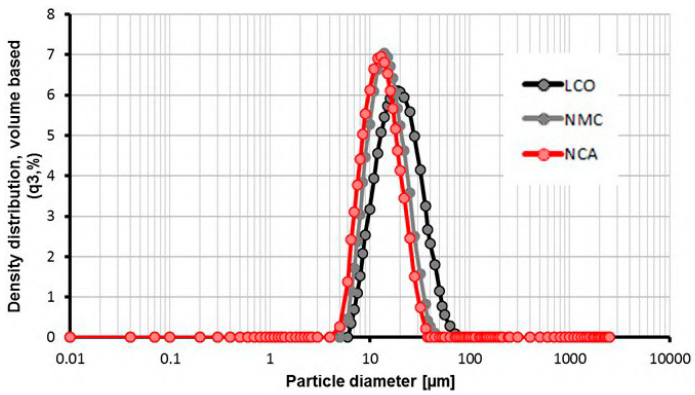
A pH scan of the zeta potential describes the chemical behavior of surface functional groups and determines the isoelectric point (IEP). The IEP is defined as the pH where the zeta potential assumes 0 mV.

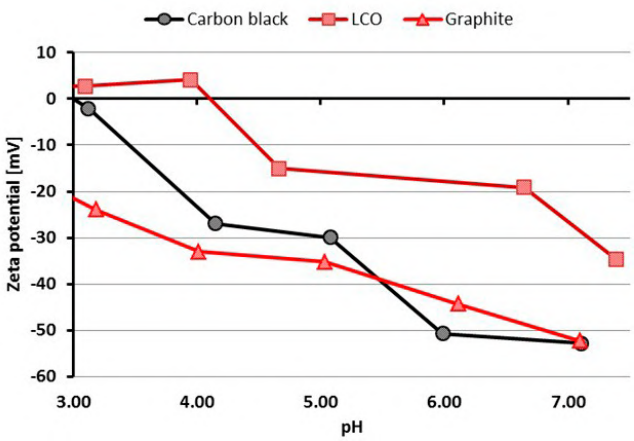
Particle size and zeta potential of electrode materials: better characterization, better performance

Brasil, Henrique (1); De Matteis, Giorgia (1); Luxbacher, Thomas (1)
(1) Anton Paar GmbH

Keywords | electrodes, Li-ion batteries, particle size, zeta potential

The power efficiency of many portable devices such as mobile phones, tablets, or speakers is extremely related to the use of rechargeable lithium-ion batteries, which have a high working voltage and a high energy density. Recently, their application has expanded to various fields such as electric bicycles, electric vehicles, and hybrid vehicles. For this reason, the particle characterization of electrode materials and possible additives is crucial for quality control and for the optimization of manufacturing processes to produce safe and long-lasting batteries. Here, the particle size distributions of three cathode materials and two anode materials were investigated using the particle size analyzer by laser diffraction. The zeta potential measurements of three aqueous suspensions of carbon black (0.05 %), graphite (0.05 %), and lithium cobalt oxide (LCO) (0.1 %) were conducted via electrophoretic light scattering (ELS) using a dosing unit accessory for automatic pH-dependent titration. The optimal mixing between different components in the final electrode slurry strictly depends on electrostatic interactions. Therefore, the zeta potential measurements are necessary to obtain a uniform coating with greater resistance to fracture.





JE. CHALLENGES OF MICROPLASTICS ANALYSIS AND CONTROL

From microplastics to garbage islands: challenges for the life cycle of fisheries

Ceballos Santos, Sandra (1); Ruiz-Salmón, Israel (2); Laso, Jara (2); Fernández-Ríos, Ana (2); Campos, Cristina (2); Margallo, María (2); Aldaco, Rubén (2)

(1) University of Cantabria, (2) Universidad de Cantabria

Keywords | fisheries, microplastics, life cycle analysis, marine debris, environmental impact

From microplastics to garbage islands: challenges for the life cycle of fisheries

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Department of Chemical and Biomolecular Engineering, University of Cantabria, Avda. De los Castros, s.n., 39005 Santander, Spain

The abundance of plastics in oceans is a remarkable concern. Ultraviolet light and movements produce the breakage of the plastics into smaller pieces that are mistaken for food by wild marine life. Their ingestion is a potential toxic and death variable for biodiversity. Fisheries must face this issue because fleets are also responsible of microplastics from marine coatings applied to boats that can leak during fishing activities and macroplastics from abandoned, lost or discarded fishing gears during fishing activities (Loubet et al., 2022).

The adaptation of fisheries could be possible, for instance, by replacing nylon nets to biodegradable ones, but may have a lower catch efficiency. Besides, the scientific community must contribute with advances in more effective environmental impact analysis tools. Current Life Cycle Impact Assessment methods do not consider certain impacts, such as biotic depletion or the degradation of the marine environment due to plastics accumulation in the ocean or damage to seafloor (Avadí et al., 2019). Furthermore, inventory flows linked to the presence of plastics (from nano to macro) are non-significant and the environmental consequences of the release of plastic debris to water bodies is not included in current metrics yet (Saling et al., 2020). Consequently, fisheries and researchers must collaborate to consolidate a sustainable pathway of production and consumption.

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JE. III ANQUE-DECHEMA LEADING EDGE CONFERENCE "PARTICLE TECHNOLOGY. SHAPING THE FUTURE"

Effect of the conductive carbon black and polymeric binder interactions on the particulate structure of lithium-ion-battery cathodes

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Keywords | particulate structure, slurry drying, lithium-ion-battery, cathode, polymeric binder, carbon black

During the production of Lithium-ion battery electrodes the various components are dissolved in a solvent, coated onto a current collector and dried subsequently. During the drying of the electrode slurries, a network of polymeric binder (PB) and conductive carbon black (CB) forms which is determinant for the mechanical properties and strongly influences the electrochemical performance of LIB-electrodes. Eminent for an increase of the energy density of LIBs in industry scale production is a reduction of these inactive components, which can lead to low amounts of PB and low ratios of PB to CB in the cathode.

Therefore, in the present work a comparison of cathodes using two different PB/CB-ratios is carried out. The slurries are composed of NCM622 as active material and polyvinylidene difluoride (PVDF) as binder to ensure a mechanical stability of the particulate coating. To increase the electrical conductivity, conductive graphite and carbon black are used. The slurries are produced in a planetary mixer using industry relevant parameters. To improve the robustness of the data, three industry relevant drying profiles are investigated, using a pilot scale continuous convective dryer. The cathodes are analyzed regarding adhesion strength, electrical resistance, as well as porosity and tortuosity, as a measure of ionic conductivity.

SEM-images are taken to evaluate the interaction of PB and CB in the cathode structure.

The analyses reveal expected influences of differing contents of PB and CB on adhesion strength and electric conductivity. Additionally, lower PB/CB-ratios lead to higher porosity, which is an indicator for high ionic conductivity and potentially offers a higher fast charge capability. Especially, in the area of low pore sizes, which is dominated by effects of the nanometer scaled CB-particles, a higher porosity can be observed for the low PB/CB-ratio. Furthermore, SEM-imaging reveals that in cathodes using the low PB/CB-ratio the PVDF clumps together with the CB preventing the characteristic crosslinks between the active material particles and between the particulate layer and the current collector, which can explain the exceptionally low adhesion strength of the cathodes.

JE. MULTIDIMENSIONAL PARTICLE PROPERTIES: CHARACTERIZATION, SEPARATION, APPLICATION

Multidimensional Separation by Magnetic Seeded Filtration

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Keywords | separation, multidimensional, magnetic, selectivity, agglomeration, surface properties

The demands on separation technology have steadily increased over recent years. Highly specific, selective separation techniques that are also suitable for fine particles in dilute suspensions, are needed more and more. In this work, magnetic seeded filtration (MSF) is presented as a separation concept that is capable of handling these challenges. Magnetic seed particles are added to a suspension and after agglomeration with the target particles, the agglomerates are removed by magnetic separation.

MSF is capable of achieving high separation efficiencies of up to 95% on a broad parameter scale, even in dilute suspensions. Selective hetero-agglomeration based on surface charge is performed and its limitations in terms of selectivity are discussed. Additionally, separation based on hydrophobic interactions is investigated and shows promising results. This is followed by consideration of the second separation criterion: particle size. It is shown that in the parameter range considered, the separation efficiency increases with increasing particle size. This influence requires a detailed discussion due to many, partially opposing effects during hetero-agglomeration. Subsequently, both dependencies are combined in a single experiment and a multidimensional separation is realized, i.e. a multi-component suspension is divided into defined substance and size classes.

The necessary addition of magnetic material is economically and ecologically questionable and the main point of criticism against the process. Therefore, this work investigates three different agglomerate processing and magnetic seed particle recycling strategies: Separated hetero-agglomerates are processed thermally, chemically and mechanically in order to recycle the magnetic fraction and eventually also recover the separated non-magnetic particles. To disclose eventual long- term effects, all approaches were investigated in a cyclical manner, where magnetic seed particles are recycled and re-used over multiple experiments. Again, consistently high recycling rates and thus also high separation efficiencies of »95% were measured throughout the experimental cycles. Furthermore, where applicable, the non-magnetic fraction was recovered with similarly high recovery rates.

In conclusion, this work presents MSF as a holistic separation concept that is both capable of performing on high levels while also being economically and ecologically viable.

Modeling of Nanoparticle Fractionation in Tubular Centrifuges Involving Arbitrarily Shaped Particles

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Keywords | Centrifugation, Fractionation, Separation of non-spherical Nanoparticles, Dynamic Process Simulation

Classification is an established process in solids processing technology to divide particulate products into defined fractions based on their size. However, complex particle systems with strict specifications regarding product-relevant properties often require a different approach. Thus, multi-dimensional fractionation is needed in which both geometric (particle size and shape) and material (density, interfacial properties) separation characteristics are considered. If a task involves the efficient separation of nanoparticles, high centrifugal forces are required. Due to their advantageous throughput rates and extreme operating conditions, tubular centrifuges are well suited for this application. During separation, a low concentrated suspension is fed into the tube centrifuge rotor with a constant volumetric flow rate. The fine fraction

escapes at the overflow of the centrifuge, while the coarse fraction accumulates on the inner rotor wall. To gain process knowledge and estimate the separation result for a well characterized product, dynamic process simulation is an effective tool. Previous studies focused on the one-dimensional classification of fines, whereby only one separation characteristic, the particle size, and its distribution, was considered in the calculation. A new approach strives to further extend well established one- dimensional model equations by additional separation criteria, i.e. the elongation, flatness and material density of individual particles. The numerical calculation of the particle transport behavior in tubular centrifuges shall thus be given a broader range of applications and help to identify advantageous operating parameters and apparatus specifications.

The presented work includes the generation of multi-dimensional particle trait distributions that define a property range of non-spherical particles as a function of at least two geometric features. For each combination of these discrete properties and a defined material density, the sedimentation coefficient in infinite dilution is derived. A theoretical background is provided by analytical and empirical model equations which express particle drag in creeping flow as a function of the single particle geometry. After calculation, the collected information is compiled into a database and transferred to the process simulation tool. Finally, the algorithm allows the prediction of a separation efficiency for the multi-dimensional fractionation of spherical, plate-shaped, and needle-shaped nanoparticles in tubular centrifuges.

SPP2045 – A9 – Fractionation of Nanoparticles by Preparative Gel Electrophoresis

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Keywords | Separation, Nanoparticles, Synthesis, Functionalization, Agarose gel

Monodisperse nanoparticles (NPs) with uniform properties regarding size and morphology are essential already today e.g. for optical (plasmonic) applications. In future, such particles will be necessary for many further areas, as in the field of medicine or for self-assembly processes, where such NPs promise structures with highest precision. Since the required narrow particle size distribution often is not reached after the synthesis especially at larger scale, post-synthetic purification is needed.

In agarose gel electrophoresis, NPs migrate through a gel induced by an external direct current electric field. This migration in an electric field is expressed by the electrophoretic mobility μE, which depends on a variety of parameters (particle size, surface chemistry, shape etc.). A difference in μE allows a spatial separation of particles with different properties within the gel. We observed that the key parameter influencing the migration is the ratio between the particle size and the mesh size of the three-dimensional gel network, leading to two types of mechanisms (Barasinski et al. 2020): If the mesh size is similar to the particle size, then the NPs move via the restricted migration mechanism, where their movement is slowed down significantly or the NPs are even entrapped within the mesh network. In contrast, NPs significantly smaller than the mesh size move show unrestricted migration, which is influenced mainly by their surface charge. In this case, the gel mainly serves to prevent particle diffusion. Figure 1 (left) shows the separation of binary mixtures of NPs under both migration mechanisms and according to the particle size, the surface charge and the morphology. The used spherical SiO2 NPs are visible as bright bands within the gel, whilst Au NPs show a red (spherical NPs) and a violet (rod-shaped NPs) band depending on their morphology. As a second step, the multidimensional separation is targeted, where a sample is separated into several fractions according to their particle size as well as net charge via 2D gel electrophoresis using gradient gels with mesh sizes that decrease from top to bottom, as illustrated in Figure 1 (right).

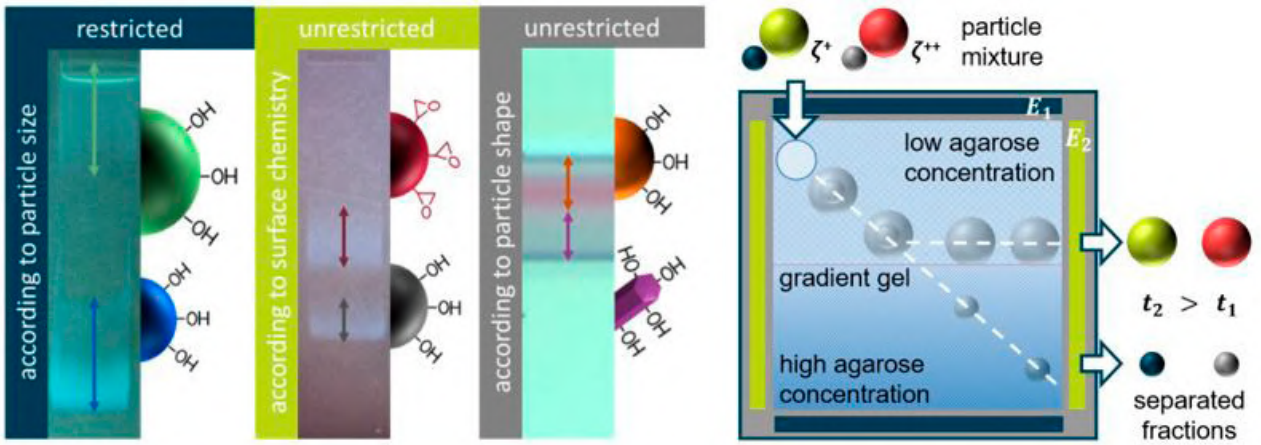


Figure 1: Separation of nanoparticles according to size, surface chemistry or shape (left, Barasinski et al. 2020 and 2022, modified) and a schematic illustration of a multidimensional separation of particulate samples by 2D electrophoresis in a gradient gel (right).

Barasinski, M., Garnweitner, G., Restricted and Unrestricted Migration Mechanisms of Silica Nanoparticles in Agarose Gels and Their Utilization for the Separation of Binary Mixtures. *J. Phys. Chem. C* **2020**, 124, 5157–5166

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Designing classifying aerodynamic lenses for multi- dimensional fractionization

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Keywords | aerodynamic lense, material separation, aerosol mixtures, classification, multi-dimensional characterization, differential aerodynamic particle sizer

A classifying aerodynamic lens (CAL), which fractionizes aerosols by size and material, was designed using analytical and stochastic models. The separation principle is based on differences in particle relaxation time. Separation by multiple properties is a challenging task, especially when particles of different size and material may behave equally in an aerodynamic separation process. Thus, it is essential to gather intelligence about the properties of the involved particle species, such as the relaxation time distribution. In this work aerosol (mixtures) are characterized, via a Differential Aerodynamic Particle Sizer (Babick et al., 2018), Scanning mobility Particle Sizer, and Aerodynamic Particle Spectrometry.

For the separation via CAL, operation parameters are selected, based on a prediction model, fed with the gathered characterization data. Separation efficiency is measured off-line, using gravimetry, scanning electron microscopy and energy-dispersive X-ray spectroscopy. The separation of a copper and silicon aerosol mixture achieved enrichment for silicon, as well as a narrowing in the size distribution.

Our study indicates that classifying aerodynamic lenses could be applicable in the recycling of composite-materials.

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Acknowledgement

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Multidimensional and correlative characterization of nanoparticles

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Keywords | multidimensional characterization, semi-automatic segmentation, TEM, XRD, gold nanoparticles, multi-core iron oxide nanoparticles

In recent decades, the research on nanoparticles (NPs) has strongly been intensified owing to their unique properties and novel technological applications. Various physical and chemical properties of NPs are determined by their morphological characteristics, including their size, shape as well as by potential microstructure defects. Therefore, characterization methods are required that are capable of determining these NP properties simultaneously in a sufficiently detailed and statistically relevant manner.

In the present study, different kinds of NPs were investigated by a combination of high-resolution transmission electron microscopy (HRTEM) complemented by local fast Fourier transformation (FFT) of the HRTEM images, high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) and X-ray diffraction (XRD). Low-magnification HAADF- STEM images were subjected to a semi-automatic segmentation routine (Neumann et al., 2020) enabling a statistically relevant ensemble characterization of the NPs.

In the first part of our contribution, the correlative determination of the size of complex NPs is demonstrated on the example of multicore iron oxide NPs. A combination of TEM and XRD revealed the hierarchical structure of the NPs. The influence of the coherence of the individual cores and crystallites within the NPs on their magnetic properties probed by alternating gradient magnetometry is discussed.

In the second part, the statistical determination of atomic-scale characteristics of NPs is demonstrated on the example of Au NPs with comparable size but different defect structures. Firstly, two distinctive types of Au NPs, multiple-twinned and plate- shaped particles, were identified by FFT/HRTEM. Secondly, the amount of the respective NP type was determined statistically using the semi-automatic segmentation of HAADF-STEM images. Both distinctive types of Au NPs were identified unambiguously from low-magnification HAADF-STEM images by using the correlation between the size of the NPs and the HAADF-STEM intensity variation. In plate-shaped NPs, moreover, the defect density was estimated qualitatively by considering the correlation between their defect density and morphology.

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Comparative study on the deterministic-hydrodynamic size, shape and density fractionation of polydisperse fine particle systems

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Keywords | multidimensional fractionation, Serpentine microchannel, Discrete Element Method, Lattice Boltzmann Method, Astigmatism Particle Tracking Velocimetry (APTV)

The effects leading to fractionation in microfluidic separators are often not fully understood. We therefore study several microfluidic fractionation methods (DLD-, MOFF, and Serpentine-channel), aiming at evaluating the mechanisms behind fractionation and their capability of fractionation by size, shape and density.

We combine numerical and experimental studies. This way we are able to make use of the validation capacity of the experiments and of the flexibility in particle properties and channel geometries that the numerical evaluation allows for.

On the experimental side, we use µPIV measurements to measure the flow in the channels and additionally fluorescence imaging and APTV measurements, to evaluate the particle positions over the channel width and height.

On the numerical side, we use a combination of the Lattice Boltzmann Method (LBM) and the Discrete Element Method (DEM). The LBM is used to calculate the fluid flow and the fluidic forces and torques, which are exerted onto the particles. The DEM, is used to evaluate

particle motion and contacts.

Through those methods, we were able to show for the first time, that particles in serpentine channels assume equilibrium positions not only over channel width, but also over channel height (Blahout et al. 2020), Fig. 1.

In DLD channels, we were able to show the dependency of the critical diameter on particle density and Reynolds number for pressure driven DLD channels (Reinecke et al. 2021), which allows for the design of DLD channels for combined size and density fractionation.

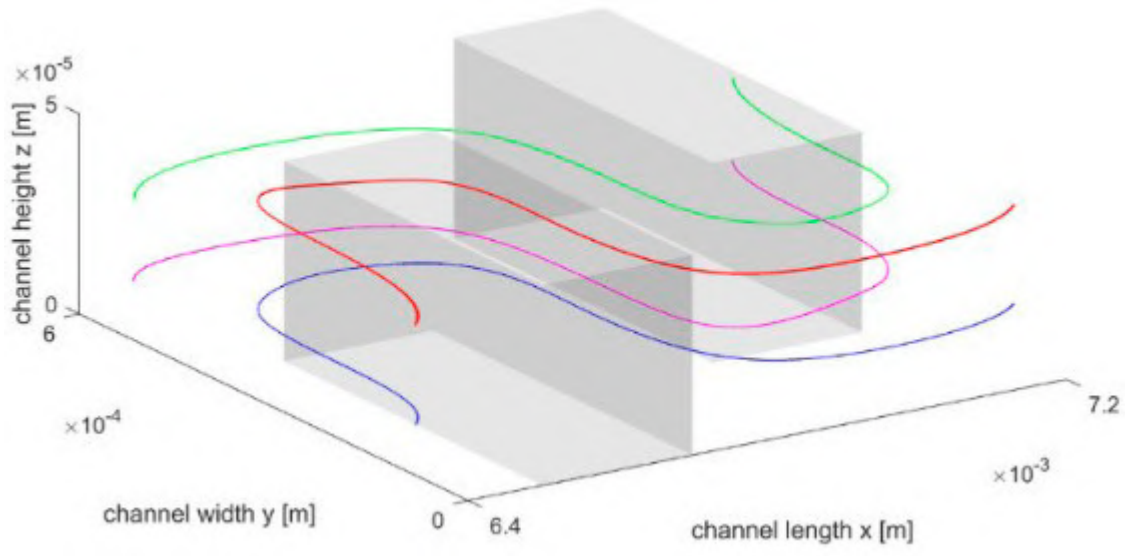


Fig. 1: Exemplary presentation of equilibrium trajectories of 15 particles inside a serpentine channel.

We thank the German Research Foundation DFG (KR3446/14-2; HU2264/3-2) for the financial support through the priority program SPP 2045.

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Continuous particle fractination using crossflow with a superimposed electrical field

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Keywords | Classification, Fine Particle, Suspension, Crossflow filtration, CFD-Simulation

In the present work a crossflow filtration technique is presented, which is a promising method for the highly specific separation of suspensions for particles < 10 µm.

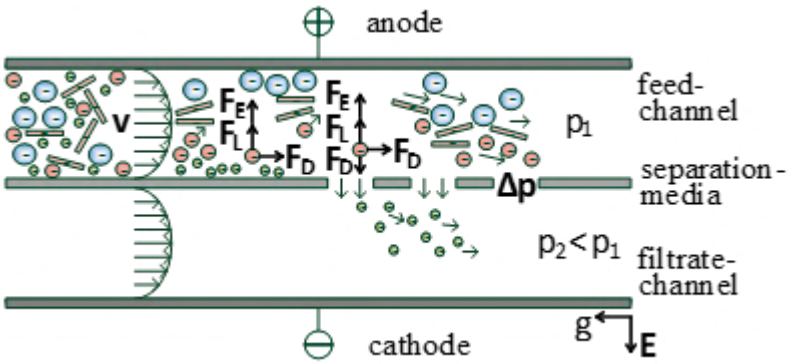


FIGURE 1: Scheme of the separation process.

In the process two main flows, both in laminar regime, are realised in parallel channels: one containing a feed suspension (feed channel) and the other a particle-free liquid (filtrate channel). Based on the size-dependent hydrodynamic forces acting in a laminar flow on the particles, bigger particles are transported into the core flow faster than smaller particles. The smaller particles remain longer in the wall boundary layer. To separate them, a separation medium with pores bigger than the particle size is adjusted between the two channels. A very small differential pressure between the two channels allows a suction of a small volume of the suspension flow containing only small particles in the filtrate channel. By adding an electrical field, which is acting orthogonally to the main flow, the particles are subjected to an additional electrophoretic force. The magnitude and direction of this force are determined by the particle properties, in particular the electrophoretic mobility and the electrical field strength. The multidimensional fractionation of a suspension according to the separation characteristics, such as particle size, shape and electrophoretic mobility, can be achieved by the combination of the lift force, drag force and electrophoretic force (FIG. 1).

A test setup is developed and used for experimental study of the separation efficiency. Additionally, a numerical study with computational fluid dynamics (CFD) is performed. Based on CFD, the optimal geometry of the channels and the separation medium are chosen, as well as the operation parameters for a hydrodynamic classification of particles < 10 µm and the combined multidimensional fractionation are determined. The separation behaviour of different materials with different particle size distributions, shape and electrical charge is investigated. The effect of influencing parameters such as particle concentration, pH value or additives are determined using various methods. The results of CFD simulations and experiments are used for the optimization of the fractionation process to increase the separation efficiency and perform the scale-up.

MultiDimFlot - Multidimensional separation of ultrafine particles using a mechanical flotation cell combined with froth fractionation

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Keywords | Ultrafine Particles, multidimensional separation, flotation, partition curves

Froth flotation is one of the most important techniques in the mining industry for the efficient separation of particles with sizes between 10 µm and 200 µm. The separation process is based on the difference in particle wettabilities, as hydrophobic particles attach to a gas bubble and are recovered in a froth, whereas hydrophilic particles tend to stay in the pulp. Although, the wettability is the most prominent separation feature, the micro processes that occur in the pulp and in the froth zone include complex interactions between the particles and the bubbles and also other particle properties, such as size, morphology, surface energy or the dispersion state have an impact on the separation.

Low ore grades and very fine composite particles in electronic devices are forcing the industry to adapt and improve existing flotation techniques to the processing of ultrafine particles (< 10 µm), as the material needs to be milled down to finer size fractions to obtain sufficient liberation of the valuable minerals. For that reason, the project “MultiDimFlot”, which is part of the German research foundation priority programme DFG-SPP 2045 “MehrDimPart”, aims at developing a method for the separation of ultrafine particles (< 10 µm) based on multiple particle properties.

A novel separation apparatus is used that combines the advantages of a mechanical flotation cell that comes with a high particle-bubble

collision rate (thus a high recovery) with those from a flotation column with a fractionating effect due to its deep froth (thus a high grade). An academic particle system, consisting of glass spheres, glass fragments and glass fibres as the valuable material and magnetite as the gangue material is used, whose particle properties are studied in depth. These investigations will help to further understand the behaviour of ultrafine particles during flotation and how certain particle properties affect the separation process. Furthermore, the possibilities and limitations of different analysis techniques, e.g. coupled SEM-EDX, flow cytometry or inverse gas chromatography are investigated for their use in ultrafine particle characterization.

Prediction of mineralogical particle composition using CT data and R-vine copulas

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Keywords | Mineralogy and ore characterization, multidimensional particle property, multivariate probability distribution, copula, image segmentation, machine learning,

Computed tomography (CT) can capture volumes large enough to measure a statistical meaningful number of micron-sized particles with a sufficiently good resolution to allow for the analysis of individual particles. However, the development of methods to efficiently investigate such image data and interpretably model the observed particle features is still an active field of research. When image data of particles exhibiting a wide range of shapes and sizes is considered, traditional image segmentation methods, such as the watershed algorithm, struggle to recognize particles with satisfying accuracy. Thus, more advanced methods of machine learning must be utilized for image segmentation to improve the validity of subsequent analyzes. Moreover, CT data does not include information about the mineralogical composition of particles and, therefore, additional SEM-EDS image data must be acquired. Here, micro-CT data of a particle system mostly composed of zinnwaldite- quartz-topaz-muscuvite composites is considered (Leißner et al., 2016). First, an image segmentation method is applied which uses deep convolutional neural networks (CNN), namely a U-net (Çiçek et al., 2016). This has the advantage of requiring less hand-labeling than other machine learning methods, while also being more flexible with the possibility of transfer learning. Then, parameterized models based on vine copulas (Joe, 2014) are designed to determine multivariate probability distributions of descriptor vectors for the size, shape, texture and composition of particles. For model fitting, the segmented three-dimensional CT data and co-registered two-dimensional SEM-EDS data are used. The models are applied to predict the mineralogical composition of particles, solely based on particle descriptors observed in CT data. The power of the introduced prediction models for estimating the mineralogical composition of particles by means of CT-based descriptor vectors are evaluated quantitatively. Results obtained for the goodness of fit and the predictive power are quantitatively assessed, see Figure 1.

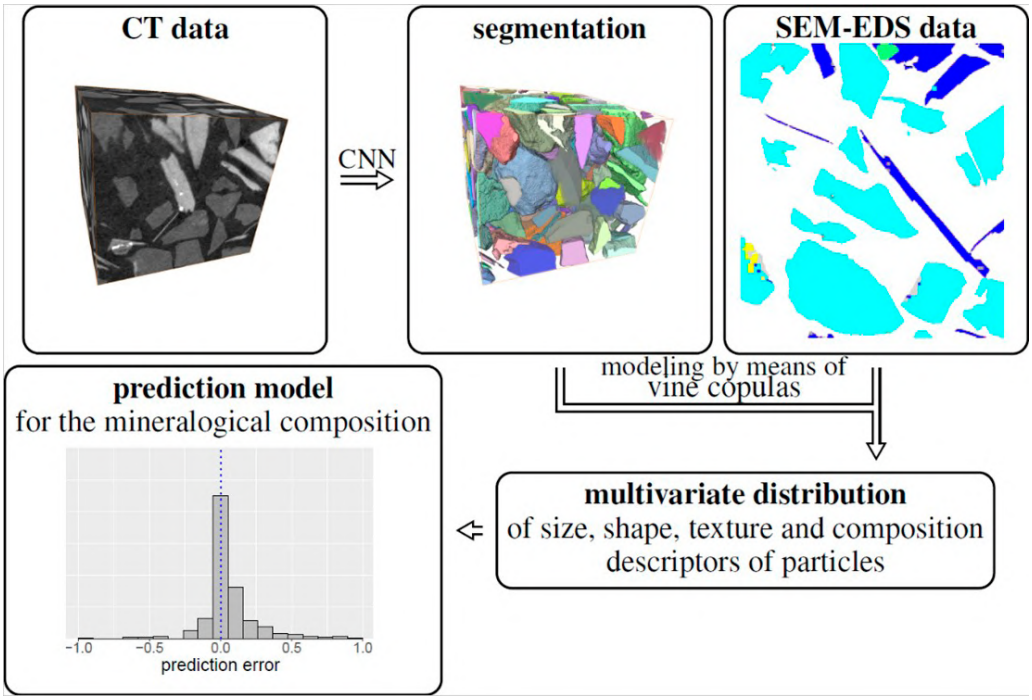


FIGURE 1: Calibration of the prediction model for estimating the mineralogical composition of particles from CT data.

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3D analysis of equally X-ray attenuating mineralogical phases utilizing a correlative tomographic workflow across multiple length scales

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Keywords | X-ray tomography, X-ray microscopy, correlative analysis, PP2045

The non-destructive microstructural analysis of materials, using X-ray computed tomography (CT) has emerged as standard analysis method in engineering sciences. Here, X-rays are attenuated material-specifically on their paths through a rotating sample, then converted into grey scale detector signals, which are finally reconstructed as a 3D volume. However, a direct derivation of the chemical composition from the determined grey values of the 3D volume is not possible without special equipment, like spectral detectors (Godinho et al., 2021), or the correlation with additional measurement methods, like energy dispersive X-ray spectroscopy (EDX) (Furat et al., 2018).

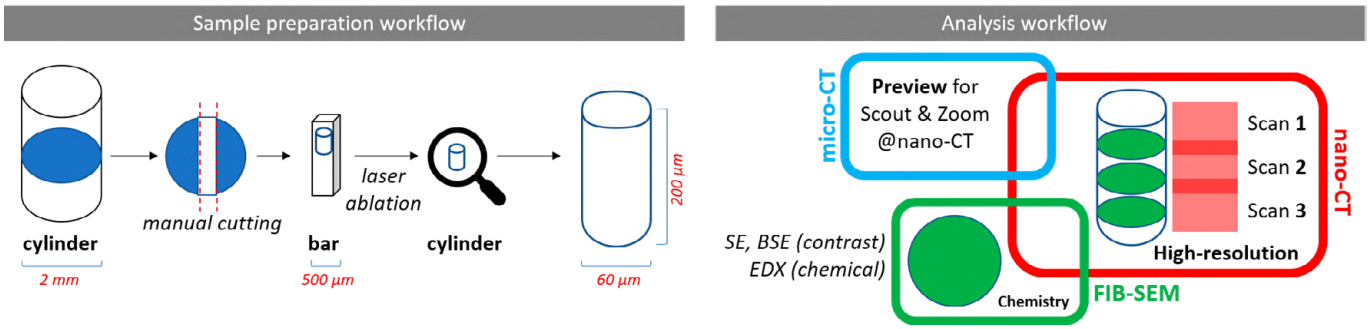


FIGURE 1: Sample preparation and analysis workflow for correlative analysis over multiple scales.

Within the framework of the Priority Program 2045, which is funded by the German Research Foundation (DFG) and deals with highly specific multidimensional fractionation of technically relevant fine particle systems, we present a 3D characterization workflow by correlating micro-CT, nano-CT and analytical scanning electron microscopy combined with EDX measurements over multiple length scales (Englisch et al., 2021). This multidimensional microscopy approach enables the distinction between the chemical components of micron-sized particle systems with similar morphologies and particle size distributions. The workflow is applied to a mixture of two minerals, namely saxolite (carbonate) and talcum (mixture of dolomite, magnesite and talcum), which have similar X-ray attenuating properties and consist of more than one phase.

Several mixtures of both particle systems were generated to have a defined starting point for method development and validation. An adapted sample preparation strategy, utilizing a resin nano particle mixture (Ditscherlein et al., 2022), is used to optimize the image processing workflow to generate particle-discrete data sets. On the other hand, EDX measurements enable the segmentation of the individual mineral phases. The combination of particle-discrete and phase-discrete data enables a quantitative characterization of the considered particle system by computing practically relevant parameters, such as the degree of mineral liberation, without stereological bias and in three dimensions.

The workflow is implemented utilizing existing and established laboratory measurement methods. Based on the acquired particle-discrete information, the method provides access to multidimensional particle property distributions enabling a detailed understanding of complex processes.

Selective particle separation at liquid-liquid interfaces

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Keywords | PP 2045, multidimensional separation, size specific separation, material specific separation

The PP 2045 deals with the multidimensional separation of fine particles smaller than 10 µm. The aim of subproject A3 is to investigate the material- and size-specific separation of particles smaller than 1 µm. Important for the multidimensional separation in subproject A3 is the wettability and size of the particles, as this determines the access into the organic phase [Leistner, 2019].

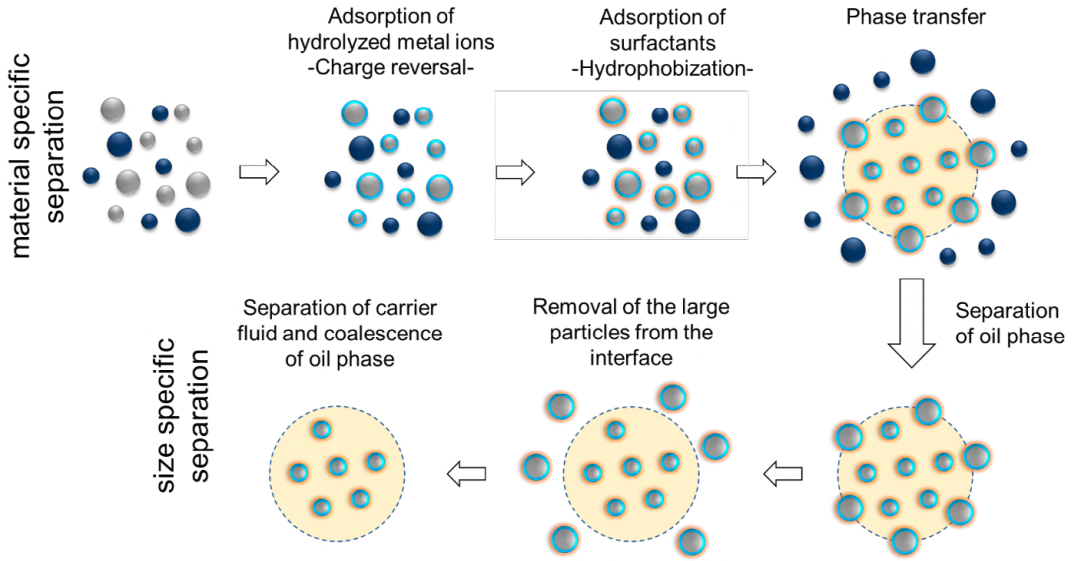


Figure 1: Schematic representation of material and size specific separation [similar to Peuker, 2019].

The used materials are naturally hydrophilic, but they have to be hydrophobized for the phase transfer with the help of surfactants. In addition, in a particle mixture the adsorption of surfactants should only address the target particle system. In this manner, a material specific separation feature, a selective adsorption of surfactants, is generated. This can be achieved with the help of hydrolyzed cations, which act as a bridge between the particle and the surfactant. Applicable hydrolyzed cations can be determined using ChemEQL [Müller, 1996]. The in this way hydrophobized particles can be transferred from the aqueous phase to the organic phase. The interface between aqueous and organic phase is used for material specific separation and will also give the opportunity for the size specific separation (Pickering emulsion). Pickering emulsions are characterized by larger particles that stay in the interface while smaller particles are dispersed in the oil phase, because larger particles are more stably fixed to an interface for a given contact angle and interfacial tension [Leistner, 2019]. Two different approaches can be used for the following size-specific separation. First, a pH change offers the possibility of reversibility of the hydrophobization and phase transfer. Because hydrolyzed cations are formed only in a certain pH range, the hydrophobization is only active in a certain pH window. The second possibility is the selective removal of the particles with the help of a temperature increase. Both possibilities will be investigated in this study.

Frequency-modulated dielectrophoretic particle chromatography

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Keywords | dielectrophoresis (DEP); microparticles; polystyrene; chromatography; interdigitated electrodes; microfluidic; separation

Separating microparticles (< 10 µm) according to properties such as size, shape, or material is a field of recent research, since conventional separation techniques do not cover this size range. However, many separation tasks for biological applications or material recycling include particles below 10 µm. Therefore, novel separation techniques are required that are capable of addressing (multiple) properties of the particles. Dielectrophoresis is one candidate for these separations.

Dielectrophoresis is the movement of polarizable particles in inhomogeneous electric field. The dielectrophoretic force depends on several particle properties such as size, shape and material. Additionally, it is influenced by process parameters as the composition of the medium in which the targeted particles are suspended (water, air or oil) or the frequency and voltage of the applied electric field. Generally, particles will move towards field maxima (positive DEP) or away from them (negative DEP), depending on their relative polarizability.

In a recent publication we introduced frequency-modulated dielectrophoretic particle chromatography (DPC) (Giesler et al. 2020). Here, a particle suspension is injected into a carrier flow in a microfluidic device flowing over an electrode array, which is connected to a voltage source. The electrodes generate an inhomogeneous electric field. In the approach, we modulate the field frequency by a (triangular) function which leads to a periodic change of the frequency between two values. Since strength and direction (positive or negative DEP) of

the dielectrophoretic motion are frequency dependent for a variety of microparticles, this leads to different particle trajectories inside the channel. Similar to the well-studied field-flow fractionation, this approach, in combination with the laminar carrier flow profile, leads to characteristic retention times for each particle type, thus allowing particle separation. In our lab we could achieve a separation of particles (size 2 to 6 µm) with respect to size and surface properties (Giesler et al. 2021).

References

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Giesler, Jasper, et al. "Separating microparticles by material and size using dielectrophoretic chromatography with frequency modulation." Scientific Reports 11.1 (2021): 1-12.

Two-Dimensional Particle Separation in Suspensions with High Solid Fraction Using High-Throughput Microsystems - Experiments and Simulations

Wullenweber, Maike Sophie (1); Kottmeier, Jonathan (1); Kampen, Ingo (1); Dietzel, Andreas (1); Kwade, Arno (1)
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Keywords | microsystems, deterministic lateral displacement, dielectrophoresis, high throughput, high solids concentration

Particles below 10 µm with narrow size distributions and defined material properties are increasingly needed for industrial applications, such as the production of active pharmaceutical ingredients, ceramics and particle-laden inks.

In our research, which is part of the DFG priority program 2045 "MehrDimPart", we investigate the deterministic lateral displacement (DLD) method, which allows size separation of particles by the flow around a large number of regularly arranged microposts. For a multidimensional fractionation, this method will be combined with dielectrophoresis (DEP) to allow fractionation based on electrical permittivity.

For industrial use, high throughput and high solids concentration are of particular interest. However, the DLD process has so far mainly been used on a microliter scale. Clogging and particle interactions limit the throughput and solids concentration of the feed suspensions (Wullenweber et al., 2022).

Parameter studies using coupled CFD-DEM simulations and experiments were carried out, in which the structure of the microsystems including spacing and shape of the microposts, the throughput and the solids concentration were varied. This allowed an improved understanding of the separation process that will contribute to the design of an efficient microsystem that allows for increased throughput, reduced blocking, and enables increased solids concentrations while maintaining a good separation efficiency.

Microsystems were fabricated using high aspect ratio plasma etching of silicon. Microposts with a height of 120 µm spaced by 20 µm were generated. Furthermore, these arrays were coated with a thin layer of SiO2 to reduce agglomeration of hydrophobic particles around the posts. In these microsystems, fractionation using both, a binary mixture of monodisperse PS particles and a polydisperse mixture of SiO2 particles, was investigated. Due to the height of the microposts, we could increase the flow rate to > 0.5 l/h to achieve a successful high-throughput fractionation by a single microsystem. In a further step, the outlet fractions should be further fractionated based on electrical permittivity using dielectrophoresis.

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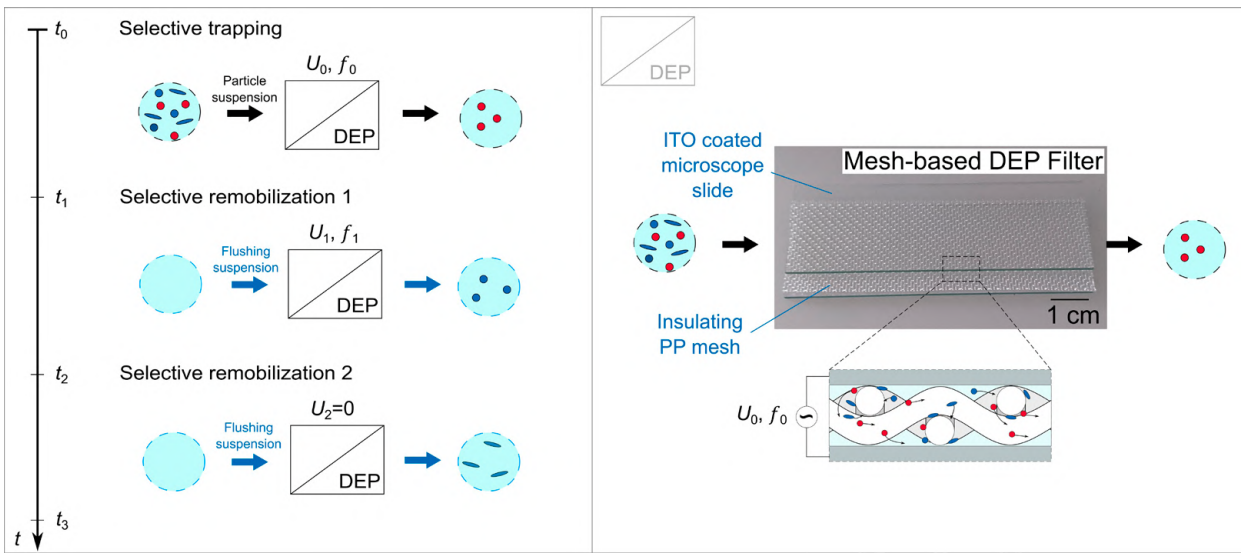
Multidimensional sorting of mixed microparticles in a mesh- based dielectrophoretic filter

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(1) University of Bremen

Keywords | dielectrophoresis; multidimensional sorting; selective remobilization; high-throughput; microparticles

The simultaneous separation of suspended particles with different properties in the lower micrometre range is becoming increasingly relevant. In synthesis of particles for instance, the result often shows variations in size and shape, and the subsequent classification according to shape in the microscale is challenging. In numerous studies, dielectrophoresis (DEP) has proven a high selectivity and versatility, which make it a promising separation technique in a variety of fields. DEP is the movement of polarizable particles in an inhomogeneous electric field. The direction and strength of the DEP force is not only dependent on particle and medium properties, but can be controlled electrically by the voltage (*U*) and frequency (*f*) of the applied field. Nonetheless, DEP is mainly used to separate or enrich particles based on a single property.

We present a novel DEP process in which particles are selectively trapped and subsequently selectively remobilized. Regular filter structures (meshes) with pores several orders of magnitude larger than the target particles are used as a filter medium and particles are trapped exclusively due to the action of dielectrophoresis. Multidimensional separation can be achieved by adjusting the field frequency and field strength considering both the deposition and the subsequent remobilization of the particles. Selective trapping has been demonstrated based on material and size and selective remobilization based on different surface conductivities and shapes of polystyrene particles. In combination, these results pave the way for high- throughput multitarget separations of particles in the lower micrometre scale in a single and scalable device.



Aggregation controlling factors in a binary colloidal dispersion of nanoparticles for 2-dimensional separation by selective agglomeration

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Keywords | Selective agglomeration, 2D-separation, surface interaction, binary colloid

To improve the product performance in synthesis/recycling of nanoparticles like quantum confined semiconductors (quantum dots, QDs) and noble metal nanoparticles a narrow particle size distribution is usually one important factor. Apart from size as the simplest dimension, a minimized dispersity in composition and surface chemistry is also essential to achieve well-defined nanoparticulate products e.g., during recycling. In this regard, selective agglomeration has already been proven as a promising method for the size classification of small semiconductor nanoparticles [Segets et al., 2015, Menter et al., 2019]. It is a purely interface-controlled method that is based on the aggregation of one colloidal component induced by titration of the dispersion by a second liquid. Developing a multidimensional separation of nanoparticles by selective aggregation requires a detailed understanding of surface chemistry and molecular interactions in multicomponent colloidal dispersion of nanoparticles. In this study, we tailor the stability/aggregation of a binary colloidal dispersion of ZnS QDs (< 10 nm) and Au nanoparticles (< 20 nm) in water as a continuous phase. Different physicochemical factors including the type of surface ligand, liquid phase composition, concentration ratios, and presence of unwashed ions in dispersion were systematically investigated to understand the aggregation behaviour of the binary colloid in liquid mixtures. A combination of UV-Vis spectroscopy, dynamic light scattering, transmission electron microscopy, nuclear magnetic resonance spectroscopy, and fourier transform infrared spectroscopy was used to analyze the

colloidal system. Then a stability/aggregation map was established based on the key affecting factors that are essential to approaching a suitable model system and efficient multidimensional separation process. In conclusion, our study provides a fundamental understanding of colloidal interfaces and complex interactions in binary colloids in solvent mixtures. This is the first step towards knowledge-based multidimensional separation of nanoparticles.

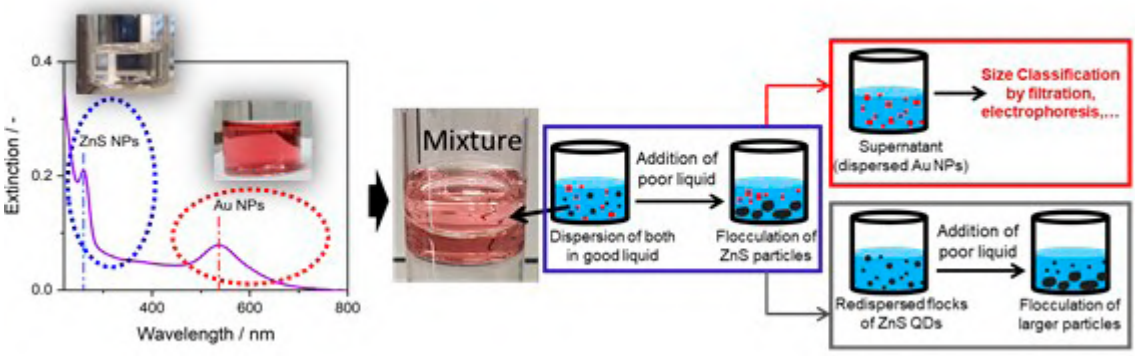


Figure 1. Multidimensional classification by selective agglomeration

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Selective particle fractionation in superimposed acoustic and electric fields

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Keywords | Aerosol classification, Finest particles, Particle separation in gases, new seperation approach

Selective particle fractionation in superimposed acoustic and electric fields

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The growing demand for fine particle systems with narrow specifications require new approaches in mechanical process engineering. Especially in the particle size range below 10 µm the mechanisms of action in established separation process reach their limits.

In this Joint Venture Poster Session, a new approach for a separation and fractionation process is presented, which is applied for particles in the gas phase and bases on a superposition of an electric and an acoustic resonant field. Because of this superposition of fields, the processes mechanism of action is capable of address even particles in the 0.5 to 10-micron particle range with high selectivity. It is also possible to address the gas-borne particles for different particle properties related to the particle inertia and material.

The following figure 1 sketches the process diagram.

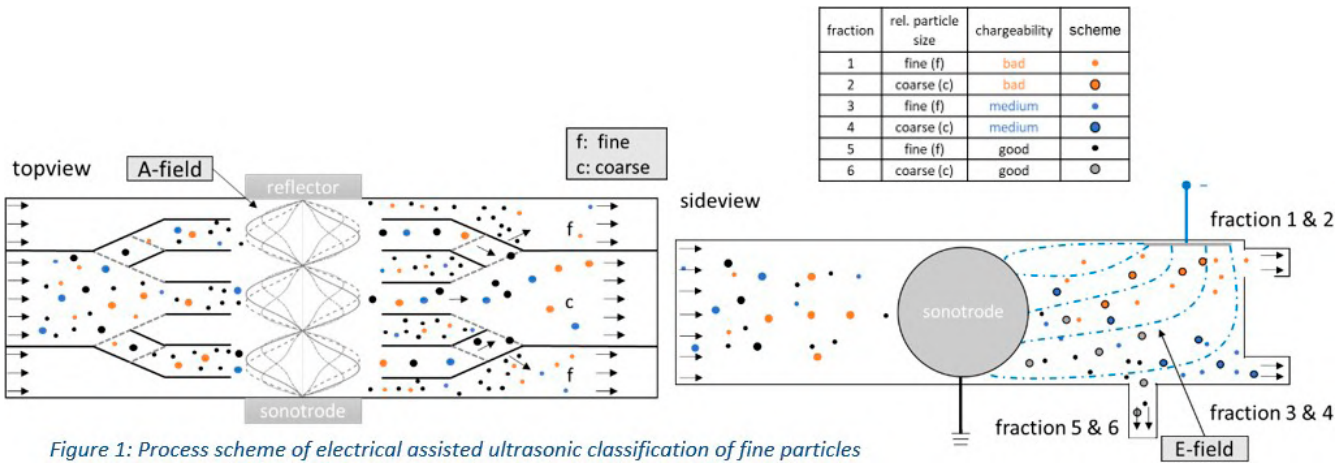


Figure 1: Process scheme of electrical assisted ultrasonic classification of fine particles

Mathematical modelling, experimental investigation as well as numerical simulations (CFD) are utilized to describe the separation process. From mathematical modelling of the particle movement in the reasonant sound field it can be shown, that for the parameters and particle sizes considered, there is a high selectivity with regard to the particle inertia (Sandmann et al., 2020). Furthermore, numerical simulations of the flow conditions in the intensive standing ultrasonic field were carried out, which, in conjunction with experimental investigations, enable a more precise process analysis.

In the numerical simulations side effects in high amplitude resonant sound fields are analysed with respect to acoustic streaming effects. Suitable measures for a drastic reduction acoustic stream will be discussed and evaluated.

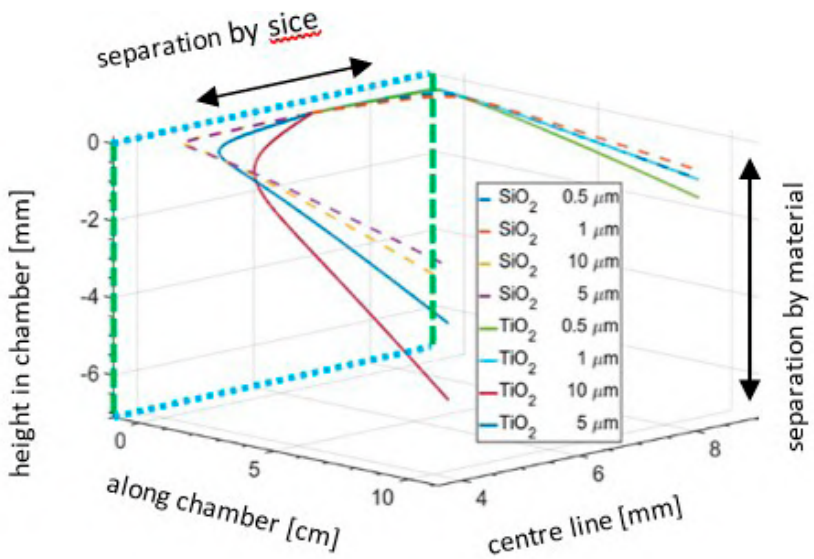


Figure 2: Mathematical modelling of particle trajectories in process cell

(Sandmann et al., 2020) Sandmann, K. and Fritsching, U. (2020), Selective Particle Classification in Ultrasonically Excited Aerosols. Chemie Ingenieur Technik, 92: 635-642

The Centrifugal Differential Mobility Analyzer (CDMA): A new Device for Multidimensional Particle Characterization

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(1) Particle Technology Group, Paderborn University

Keywords | multiple properties, hydrodynamic size, aerodynamic size, dynamic shape factor, multidimensional characterization

In order to characterize complex shaped, non-spherical particles, it is necessary to measure different particle properties simultaneously in order to get a multidimensional distribution of particle characteristics. For aerosols a two-dimensional characterization can be achieved via tandem arrangements of different instruments (e.g. DMA & CPMA). Our newly developed instrument provides the means to measure the two-dimensional distribution of mobility and aerodynamic diameters in one single compact device. The CDMA basically combines the methods of a DMA and an AAC and can be considered as a rotating DMA. It consists of two coaxial cylinders, which can rotate and an electrical voltage can be applied across the resulting gap. Electrical and centrifugal forces acting on the particles lead to their migration across the gap through particle free sheath air. The device can be operated at varying rotational speeds and applied voltages. For each set of parameters a fraction of particles with a specified drift velocity is selected at the exit of the outer cylinder. This drift velocity is determined by the electric field via the hydrodynamic diameter, while it is determined by the centrifugal field via the Stokes diameter. To measure the full two-dimensional distribution first the two marginal distributions can be determined by operating the device with an electric field only (i.e. using classical DMA theory to obtain the mobility distribution) and rotation only (i.e. using classical AAC theory to obtain the distribution of the Stokes diameter), respectively. Subsequently a two-dimensional scan of voltage and rotational speed is performed in order to obtain the full 2D-distribution.

A second prototype of the CDMA is currently manufactured. We will present the CDMA theory and will show results on data inversion in order to obtain 2D property distributions from nested scans of voltage and rotation. Achievable resolution and accuracy are discussed in detail. Furthermore, first experimental results of 2D distributions obtained with the prototype will be shown.

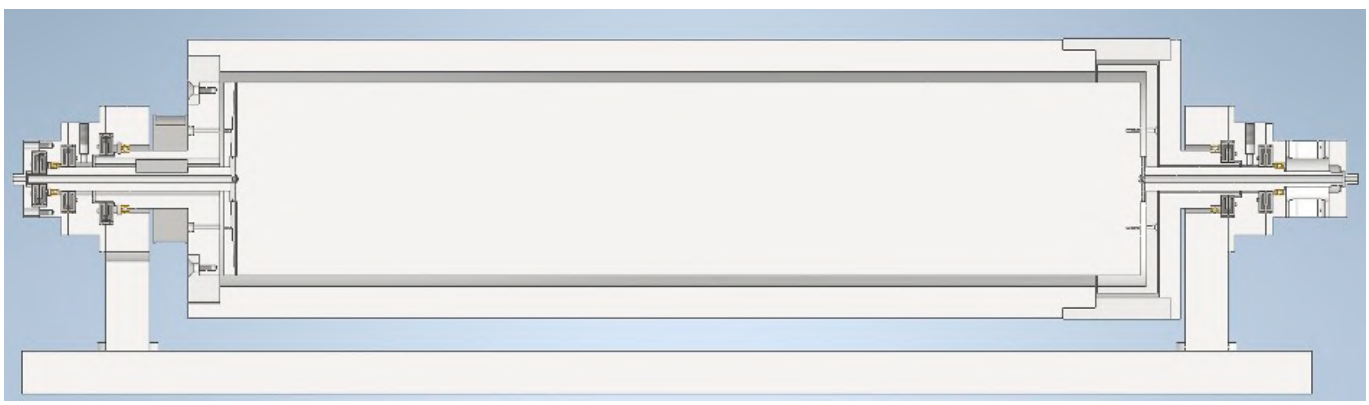
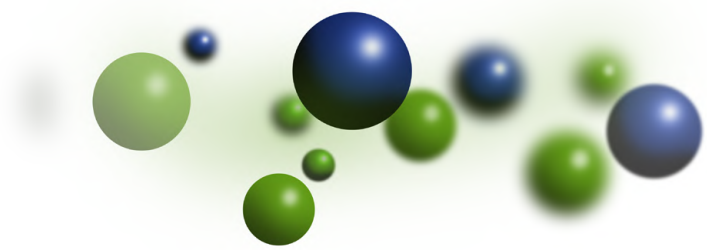


Figure 1: Cross-section of the CDMA device.

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