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Determining Reactivity Level of Granite Aggregate for Concrete

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***Abstract* – Granite rubble is one of the most frequently used aggregates in concrete manufacturing. Granite rubble is considered to be a non-reactive aggregate, however, depending on the quarry, granite may have various impurities including harmful minerals containing active SiO_2. The alkali – silica reaction is among the chemical reactions that have a significant effect on the durability of concrete. During the alkali – silica reaction certain forms of silica present in concrete aggregates react with high alkali content leading to formation of hygroscopic gel that expands in humid environment and slowly, although strongly enough degrades concrete structures. Concrete rubble of 3 different fractions was used for the test: 2/8 (Mix D); 11/16 (Mix E); 5/11 (Mix G). The tests revealed that granite rubble used for the tests contained few reactive rocks containing amorphous silica because after 14 days the expansion did not exceed 0.1 % ((D Mix expanded by 0.059 %, E Mix expanded by 0.066 %, G Mix expanded by 0.079 %) according to RILEM AAR-2 test method. After 56 days of testing none of the test specimens demonstrated significant micro-cracking and scaling specific to alkali corrosion; only gel deposits on the surface were observed.**

***Keywords* – Aggregates, alkali induced corrosion, concrete, expansion, granite rubble, reactivity.**

I. Introduction

Concrete is made of coarse and fine aggregates, water and binding material. Over time, investigations revealed that some aggregates used in concrete manufacturing cause reactions that have an adverse effect on concrete structure [1], [2]. A lot of natural aggregates are contaminated with amorphous silica and carbonates that both react with alkali (Na and K). Alkali enter the cement mix from cementitious materials. This reaction is commonly known as alkali induced corrosion reaction [3], [4].

Alkali induced corrosion of concrete is the chemical reaction of alkali and aggregates. This reaction occurs in concrete or mortar when reactive rocks from the aggregates combine with sodium and potassium hydroxides emitted mainly from hardening cement binder. The reaction product is hygroscopic gel, which through water absorption and expansion causes internal stress that sometimes significantly exceeds the bearing capacity of concrete or mortar. Internal stresses caused by reaction products result in microcracking of concrete or mortar [5].

Finnish researchers have found that the alkali-silica reaction occurs in the presence of aggregates with reactive silica, high alkali pH and sufficient moisture [6].

Over a number of years, detailed investigations into alkali induced corrosion have led to the conclusion that the process involves 3 components: amorphous hydrous and cryptocrystalline SiO_2 (opoka and flint), sodium and potassium hydroxides and calcium hydroxide. These components react in two stages [7]:

- firstly, sodium and potassium hydroxides (ROH) react with amorphous SiO_2 producing alkaline silicate with a popular name of soluble glass: $m SiO_2 + 2ROH + nH_2O = R_2O \cdot mSiO_2 \cdot n1H_2O$; m may vary from 1 to 4. Soluble glass does not have any destructive effect on concrete other than staining;
- secondly, alite ($3CaO \cdot SiO_2$), and afterwards belite ($2CaO \cdot SiO_2$), hydrolyse producing portlandite (calcium hydroxide $Ca(OH)_2$), which reacts with alkaline hydrosilicate producing water insoluble colloid substance $2ROH \cdot mSiO_2 \cdot nH_2O + Ca(OH)_2 = CaO$.

$mSiO_2 \cdot n1H_2O + 2ROH$ again releases alkali and forms a semi-permeable membrane around the reactive particle of the aggregate. The membrane allows the circulation of substances only towards the reactive particle in the presence of high osmotic pressure that expands and degrades reactive particles of the aggregate.

First of all, potentially reactive rocks shall be evaluated by means of petrographic analysis [8], [9]. The description of reactive rock from different countries is presented in RILEM AAR-1 [10]. According to RILEM AAR-1 rocks are classified into three classes: Class I includes very unlikely to be alkali-reactive aggregates, Class II includes alkali reactivity uncertain aggregates, and Class III includes very likely to be alkali-reactive aggregates [11].

Rock formation history in the world regions is different and the reactivity of aggregates also differs. In some countries some types of rock are reactive, whereas in other countries other types of rock are reactive [12].

The reactive silica content in rocks also varies. According to laboratory test results aggregates can be classified by alkaline reactivity from fast to normal (5–20 years), slow (5–20 years) and non-reactive [13].

There are coarse opoka rocks with carbonate inclusions and coarse pieces of porous flint among the carbonate and silicate rocks in all quarries in Lithuania. Thus, all fine and coarse aggregates obtained from Lithuania's quarries are contaminated with opoka and flint that actively react with sodium and potassium hydroxides present in the cement [14]. Opoka is twice more reactive than flint. The solubility of opoka in sodium hydroxide is 630 mmol/l and the solubility of flint is 330 mmol/l. It should be noted that reactive rocks are distributed unevenly and sampling variation exceeds 60 % [15]. Mineral composition is not the single factor that influences the reactivity of rocks. Alkaline

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induced corrosions is increased by the porosity of rocks. Liquids permeate better in porous rocks [16].

The aim of the paper is to find the granite rubble reactivity level according to RILEM AAR-2 test method.

II. Materials and Methods

Concrete mixes of 3 different fractions were used for the test: 2/8 (Mix D); 11/16 (Mix E); 5/11 (Mix G). Their chemical and petrographic compositions are presented in Tables I and II. Physical properties of granite rubble are presented in Table III.

Portland cement CEM I 52.5 R was used for the test. Cement specifications are presented in Tables IV and V.

A very high quality superplasticizer MC-PowerFlow 3100 was added in minute doses to produce the cement paste of the required plasticity. This superplasticizer accelerates the initial setting time and creates high early strength of the paste. The superplasticizer is based on polycarboxylate ether.

The paste was mixed mechanically and compacted in forms by a shaker. The specimens were kept in the moulds in humid conditions for 24 hours and afterwards they were demoulded and hardened in water until the deformation testing.

After 28 days of hardening in water mortar bars were bent and compressed by a press. The flexural and compressive strength were determined according to LST EN 1015-11:2004 [17].

TABLE I

Chemical Composition of Granite Rubble

Chemical composition	Indicator value, %
Water soluble chlorides	0.001
Total sulphur	1.000
Acid soluble sulphate	0.200
Organic impurities (humus)	negative
Light pollutants content	0.000

TABLE II

Petrographic Composition of Granite Rubble

Petrographic composition	Indicator value, %
Granite	95
Diorite	5

TABLE III

Physical Properties of Granite Rubble

Granite rubble fraction	Bulk density, kg/m^3	Particle density, kg/m^3
2/8	1480	2630
5/11	1530	2630
11/16	1450	2630

TABLE IV

Mechanical and Physical Characteristics of Cement CEM I 52.5 R

Fineness	Compressive strength, MPa		Densities, kg/m^3	
Fineness, Blaine tester, m^2/kg	7 days	28 days	Specific	Bulk
520	52	64	3125	1250

The reactivity of aggregates was tested according to RILEM RECOMMENDED TEST METHOD: AAR-2 (the ultra-accelerated mortar-bar test). This method is used to assess the level of reactivity of the aggregates. According to AAR-2 method procedure mortar bars (40 mm × 40 mm × 160 mm) hardened in water of 80 °C are kept in 1 M NaOH solution for 14 days at 80 °C and their expansion is measured. When the aggregates contain slowly reacting rocks, the hardening time should be longer (21 or 28 days).

The ultra-accelerated AAR-2 can be used for testing aggregate mixes, however, the aggregate particle size shall be from 125 μm to 4 mm. Coarse aggregate is crushed by jaw crusher according to the specific requirements presented in Table VI and washed.

Washed samples are dried at 100–110 °C for 16 hours. The mortar was made of 1 part cement and 2.25 parts aggregate. 600 g of cement and 1350 g of dry aggregate is required for three prisms (40 mm × 40 mm × 160 mm). 6 prisms with each aggregate were moulded. The water/cement ratio was 0.47. The mortar flow diameter must be from 205 to 220 mm. Polycarboxylate-based superplasticizer was used to obtain the required mortar flow. Superplasticizer was added at 0.2 % to the mortar containing granite rubble fraction 2/8 (Mix D) and the flow diameter 215 mm was achieved. Superplasticizer was added at 0.2 % to the mortar containing granite rubble fraction 11/16 (Mix E) and the flow diameter 216 mm was achieved. Superplasticizer was added at 0.2 % to the mortar containing granite rubble fraction 5/11 (Mix G) and the flow diameter 223 mm was achieved. Mix compositions per one test specimen are presented in Table VII. The mortar was made in accordance with cement class requirements as per EN 196-1 [18].

TABLE V

Chemical Composition of Cement CEM I 52.5 R

Loss on ignition, %	Insoluble residue, %	MgO, %	SO_3, %	Cl, %
0.7–1.7	0.0–0.5	2.3–2.5	3.1–3.8	0.02–0.07

TABLE VI

Aggregate Sieving Requirements

Sieve size		Mass fraction, %
Sieve size when the aggregate fraction goes through	Sieve size when the aggregate fraction remains on the sieve	
4 mm	2 mm	10
2 mm	1 mm	25
1 mm	500 μm	25
500 μm	250 μm	25
250 μm	125 μm	15

TABLE VII

Amount of Materials per Specimen

Mix	Cement, g	Aggregate, g	Water, ml	Pasticizer, g	W/C	Flow diameter, mm
D	200	450	94	2.4	0.47	215
E	200	450	94	2.4	0.47	216
G	200	450	94	2.4	0.47	223

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Fig. 1. Digital dial indicator and frame.

Fig. 2. Stainless steel container.

Fig. 3. Climatic chamber.

The elongation of specimens was measured by a digital dial indicator with the scale interval of 0.001 mm. The indicator is fixed in the frame (Fig. 1) where the prisms are placed. The frame and indicator axles are inserted into the moulded holes at the ends of the specimen.

Four prisms with each aggregate were selected for the test. The length of the moulded prisms is measured and the prisms are immersed into water at 80 °C. After 24 hours the prism lengths are measured again (zero reading) and the specimens are placed into stainless steel containers (Fig. 2) with 1 M NaOH alkali solution at 80 °C.

Containers with the specimens are placed into the climatic chamber (Fig. 3) where (80 ± 2) °C temperature is maintained. The elongation of specimens is measured regularly at the same hour of the day. When the specimens are removed after 24 ± 2 hardening in water at 80 °C temperature, the length, i.e. the zero reading (L_0) is recorded; this is a reference reading for further measuring of specimen expansion. After the specimens are placed into 1 M NaOH alkaline solution at 80 °C, the specimen dimensions (L_n) are recorded after 1, 3, 6, 9, 12, 14, 21, 28, 35, 42, 49, 56 days ± 2 hours.

The linear expansion of each specimen is obtained by calculating the difference between the length of the specimen at each period of measurement (L_n) and zero measurement (L_0), to the nearest 0.001 % of the effective length, as follows:

$$\text{Expansion} = \frac{100 \cdot (L_n - L_0)}{l}, \%, \qquad (1)$$

where: L_0 – measurement of the specimen just before immersion into sodium hydroxide solution (zero reading);

L_n – a reading taken at each period of soaking in sodium hydroxide solution, n being the number of days counted from the zero reading;

l – distance between the inner ends of the metal pins, measured to the nearest of 0.1 mm.

III. Results

The mortar density, compressive and flexural strength were measured in the tests. Portland cement characteristics are presented in Table VIII.

TABLE VIII

Physical and Mechanical Characteristics of the Specimens after 28 Days of Hardening

Specimen name	Density, kg/m^3	Flexural strength, MPa	Compressive strength, MPa
D (fr. 2/8)	2324	10.8	64.7
E (fr. 11/16)	2314	11.6	65.2
G (fr. 5/11)	2321	10.1	62.6

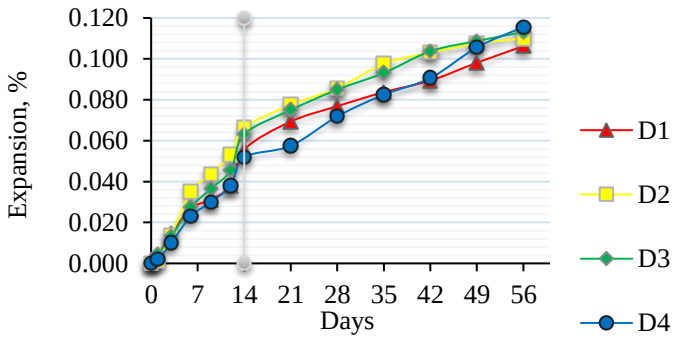

Fig. 4. Expansion of specimens with granite rubble fr.2/8 (Mix D) after 56 days.

The test of Specimen D mix with granite rubble fr. 2/8 after 56 testing days gave the results presented in Figure 4. The average elongation after 14 testing days was 0.059 % and after 56 testing days the average elongation of the prisms was 0.111 %. The expansion of the prisms occurs when the specimens are cured in 1 M NaOH alkaline solution at 80 °C. The expansion is caused by the alkaline silica gel that develops in the specimen (Fig. 5). After the testing gel deposits were observed on the specimen surface evidencing alkali induced corrosion. The deposits result from the washing of alkali and silica gel or calcium hydroxide from the deeper layers of the specimen. After 56 days of testing gel deposits on the surface of the rods was the only evidence of alkali induced corrosion; no surface cracking typical of alkaline induced corrosion was observed. The test revealed that granite rubble (fr. 2/8) was not heavily contaminated with reactive rock and can be classified as Class I – very unlikely to be alkali-reactive (expansion after 14 days according to AAR-2 < 0.10 %) because no surface damage was observed in visual examination of the specimens after the test.

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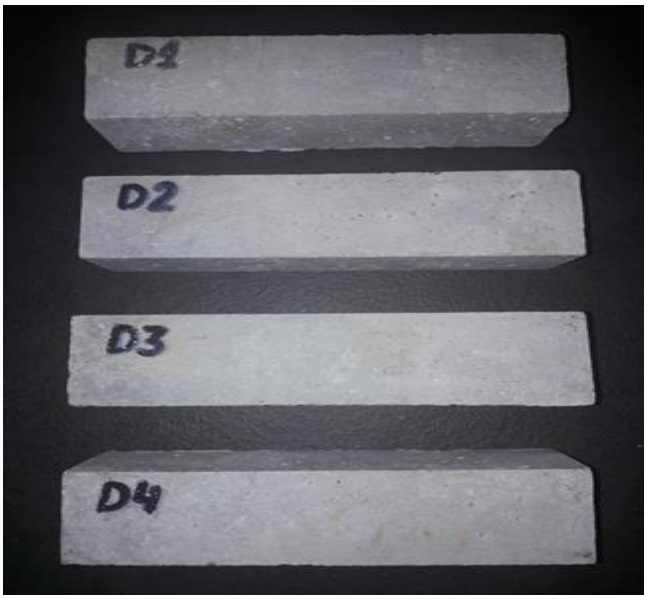

Fig. 5. Photos of specimens with granite rubble fr. 2/8 (Mix D) after 56 days in 1 M NaOH solution.

Fig. 7. Photos of specimens with granite rubble fr. 11/16 (Mix E) after 56 days soaking in 1 M NaOH solution.

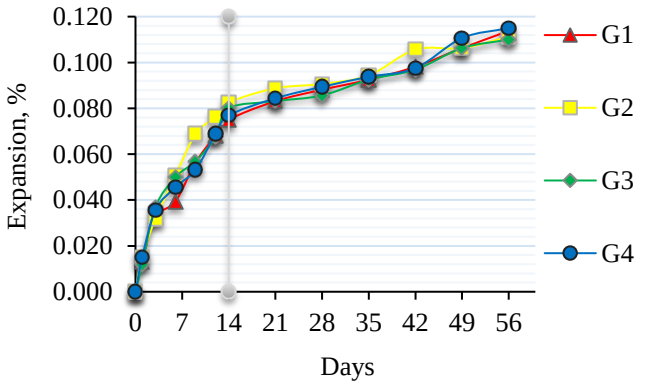

Fig. 6. Expansion of specimens with granite rubble fr. 11/16 (Mix E) after 56 days.

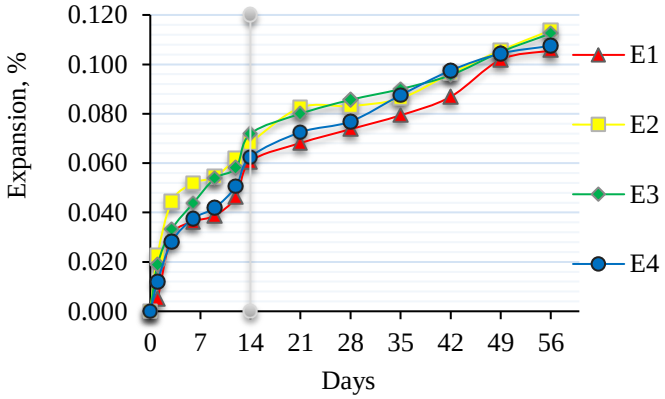

Fig. 8. Expansion of specimens with granite rubble fr. 5/11 (Mix G) after 56 days.

The results of tests of specimens with granite rubble fr. 11/16 (Mix E) are presented in Figure 6. The average elongation after 14 testing days was 0.066 % and after 56 testing days the average elongation of the prisms was 0.110 %. The expansion of the prisms occurs when specimens are immersed into 1 M NaOH solution at 80 °C. The expansion is caused by the alkaline silica gel that develops in the specimen. Gel deposits on the surface were also observed on the specimens of Mix E as on Mix D specimens; that is typical of alkali induced corrosion (Fig. 7).

The deposits result from the washing of alkali and silica gel or calcium hydroxide from the deeper layers of the specimen. After 56 days of testing gel deposits on the surface of the rods was the only evidence of alkali induced corrosion; no surface cracking typical of alkaline induced corrosion was observed. Visual examination of the mortar bars did not reveal any surface damage, therefore we may state that granite rubble is not heavily contaminated with reactive rock that causes alkali induced corrosion and concrete degradation. Granite rubble (fr. 8/16) can be classified as Class I – very unlikely to be alkali-reactive (expansion after 14 days according to AAR-2 < 0.10 %).

Fig. 9. Photos of specimens with granite rubble fr. 5/11 (Mix G) after 56 days soaking in 1 M NaOH solution.

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Results of testing of the specimens with granite rubble fr. 5/11 (Mix G) are presented in Figure 8. The average elongation after 14 testing days was 0.079 % and after 56 testing days the average elongation of the prisms was 0.112 %.

The expansion of the specimens occurs when the prisms are immersed into 1M NaOH solution at 80 °C. The expansion is caused by the alkaline silica gel that develops in the specimen. Gel deposits on the surface were also observed on the specimens of Mix G; that is typical of alkali induced corrosion (Fig. 9). After 56 days of testing gel deposits on the surface of the rods was the only evidence of alkali induced corrosion; no surface cracking typical of alkaline induced corrosion was observed. Visual examination of the mortar bars did not reveal any surface damage; granite rubble (fr. 5/11) was not heavily contaminated with reactive rock that causes alkali induced corrosion and concrete degradation; therefore it can be classified as Class I – very unlikely to be alkali-reactive (expansion after 14 days according to AAR-2 < 0.10 %).

IV. Conclusions

The tests revealed that the tested granite rubble aggregates (fractions 2/8; 8/16; 5/11) are not highly polluted with reactive particles containing amorphous silica that reacts with sodium and potassium hydroxides causing alkali induced corrosion.

The test results showed that the tested granite rubble aggregates contain few reactive rocks containing amorphous silica because the expansion after 14 testing days did not exceed 0.1 % (0.059 % for 2/8 fraction mix, 0.066 % for 11/16 fraction mix, 0.079 % for 5/11 fraction mix) according to RILEM AAR-2 test method.

Visual examination of the mortar bars did not reveal any surface damage; granite rubble (fr. 5/11) was not heavily contaminated with reactive rock that causes alkali induced corrosion and concrete degradation; therefore it can be classified as Class I – very unlikely to be alkali-reactive (expansion after 14 days according to AAR-2 < 0.10 %).

Alkali induced corrosion tests with granite rubble did not show any signs of concrete degradation evidenced by visually seen microcracks, which spread in different directions on the surface, and local scaling on the surface at reactive aggregate location. Only surface gel deposits were observed.

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