Identification of Reactivity Level in Aggregates from Lithuanian Gravel Quarry

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Abstract – **The article analyses the reactivity level of aggregates from Lithuanian quarries and their effect on the alkali corrosion in mortars. The reactivity of aggregates was assessed according to Rilem Recommended Test Method: AAR-2. The hardened cement paste and cracked aggregate contact zones were tested by means of optical microscopy. Two gravel aggregates and one sand aggregate from Lithuanian quarries were selected for testing. The tests revealed that according to AAR 2 methodology fine and coarse aggregates from Lithuanian quarries shall be allocated to Group II, i.e. reactive aggregates. The expansion after 14 days exceeds 0.1 % in the case of fine aggregate, the average expansion after 14 days is 0.13 %, and in the case of coarse aggregates the average expansion ranges from 0.11 % to 0.12 %.**

Keywords – **Alkali-aggregate reaction, alkalis, aggregates, expansion, hygroscopic gel, reactivity.**

I. INTRODUCTION

Concrete is a very important building material often used in huge structures such as bridges, dams and platforms [1]. It consists of particles of aggregate, water, and a binding agent (cement and other cementitious materials). For a long time it has been observed that some aggregates incorporated in concrete develop reactions that damage the structures [2], [3]. The reactions between aggregates and binding agents are extremely complex. Many natural aggregates contain certain quantities of amorphous silica and carbonate, which both react with the alkalis (Na and K) of the mixture mainly coming from cementitious materials. This interaction is widely known as the alkali-aggregate reaction (AAR) [4], [5].

Alkali-aggregate reaction (AAR) is only one of the many factors that might be fully or partly responsible for the deterioration and premature loss in serviceability of concrete infrastructure [6].

Alkali-silica reactivity was recognized as a potential source of distress in concrete in the 1940s. Although reactive aggregates exist in many countries, ASR distress in concrete structures may remain undiagnosed. There are a number of reasons for this. Certain silica minerals present in aggregates do not produce deleterious expansion. Curing of concrete in-situ prevents ASR as does the use of various types of pozzolan or blast-furnace slag and low alkali content in concrete [7].

Alkali-aggregate reaction (AAR) is an internal chemical reaction between the alkaline components in the cement and certain active mineral constituents in some aggregates. The more fine-grained silica-phases react faster in the alkali silica-reaction. The most widespread type of AAR is Alkali-Silica Reactions (ASR) in which silica sensu lato in the aggregate react with the alkali from the cement paste forming a hygroscopic gel [8].

The alkali silica reaction is defined as the breakdown of silanol bonds in poorly crystallized silica present in some aggregates. In highly alkaline conditions, depolymerisation and dissolution of reactive silica in the aggregate creates sodium hydroxide and potassium hydroxides. Hydrolysis of these oxides produces alkali silica gel in solid form. The gel product expanding in the presence of moisture creates stresses in the microstructure. Expansion of concrete and degradation of its mechanical properties may result in malfunction of the structure. Prediction of such slow degradation process throughout the service life of a structure is vitally important in durable concrete design, construction and operational issues [9].

The discussed observations make it possible to conclude that the mechanisms and process of expansion in reactive carbonate rocks are multistage and complex processes [10]. In the first stage, hydroxyl ions attack the silanol groups (Si-OH) and the siloxane bonds (Si-O-Si); in the second stage, the formation of expansion products occurs. Depending on the gel formation generated, internal pressure is created within the cement matrix, triggering macroscopic expansion and subsequent degradation of the concrete matrix [11], [12].

Durability of normal Portland cement is a major issue in the civil infrastructure industry. Alkali silica reaction (ASR) occurs due to the chemical reaction between hydroxyl ions in the pore water within the concrete matrix and certain forms of silica [13].

Alkali-aggregate reaction (AAR) is a reaction in concrete between the alkali hydroxides, which originate mainly from the Portland cement, and certain types of aggregate [14].

This reaction can be stopped by ensuring that the alkalinity of the pore water within the concrete is reduced to a sufficiently low value to minimise or even prevent gel forming. The sources of high alkalinity are primarily found in the PC/CEM I component, though chlorides and alkalis from other sources cannot be ignored [15].
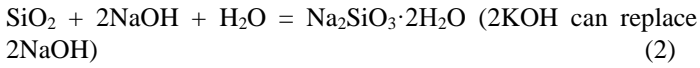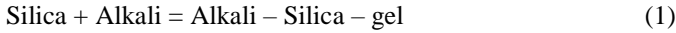
Conditions required for AAR:
- high alkali content of the cement;
- reactive aggregate;
- high moisture / water [16].

ASR may take from 3 to even more than 25 years to develop significantly in concrete structures depending on the main factors such as the nature (reactivity level) of the aggregates used, moisture and temperature conditions, and concrete alkali content [17].
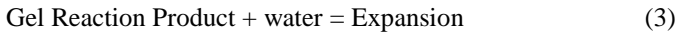
The reaction between reactive silica and alkali in the pore solution produces a hydrous alkali- silica gel, often referred to as ASR gel. Formation of the ASR gel alone does not cause cracking, however when the gel absorbs water, it shows

significant potential to swell. The resulting expansion often results in pressures greater than what the concrete can withstand, which in turn causes cracks in the concrete. The reaction process can be viewed as a two-step process [18]:

Step 1:

$$Silica + Alkali = Alkali - Silica - gel \qquad (1)$$

$$SiO_2 + 2NaOH + H_2O = Na_2SiO_3 \cdot 2H_2O \text{ (2KOH can replace 2NaOH)} \qquad (2)$$

Step 2:

$$Gel \ Reaction \ Product + water = Expansion \qquad (3)$$

Common visual symptoms of ASR generally consist of [19]:
– cracking;
– expansion causing deformation, relative movement, and displacement;
– localized crushing of concrete;
– extrusion of joint material;
– surface pop-outs;
– surface discoloration and gel exudations.

As a basis for calculating the total percentage of "reactive / possible reactive" rock types in an aggregate, each of the rock types detected should be placed, based on the petrographer's experience, in one of the three "reactivity classes". AAR is suggested to be divided into the following subgroups [20], [21]:
– very unlikely to be alkali-reactive;
– alkali-reactive uncertain;
– very likely to be alkali-reactive.

The rocks have different reactivity, because each country has a different history of rock formation.

After analysing the results of laboratory tests, aggregates can be classified according to the reactivity of alkali from "fast" to "normal" reactivity (5 to 20 years), "slow" reactivity (15 to 20 years), and "non-reactive" [20].

II. MATERIALS AND METHODS

4/16 mm fraction gravel and 0/4 mm fraction sand were used for the tests according to LST EN 13139+AC. Polycarboxylate super-plasticizer was used to achieve the required workability of the paste. Characteristics of the cement CEM I 52.5 R used for the tests are presented in Tables I and II.

TABLE I

MECHANICAL AND PHYSICAL PROPERTIES OF PORTLAND CEMENT

Fineness	Compressive strength, MPa		Densities, kg/m³	
Blaine apparatus, m²/kg	7 days	28 days	specific	bulk
520	52	64	3125	1250

TABLE II

PORTLAND CEMENT CHEMICAL COMPOSITION

CaO, %	SiO₂, %	Al₂O₃, %	Fe₂O₃, %	SO₃, %	Free CaO, %
64.02	20.09	4.84	3.87	2.83	4.35

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 at 80 °C are kept in 1M 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).

Classification of aggregates: Class I – very unlikely to be alkali-reactive (expansion acc. AAR-2 < 0.10 %); Class II – potentially alkali-reactive (expansion acc. AAR-2 from 0.10 % to 0.20 %); Class III – very likely to be alkali-reactive (expansion acc. AAR-2 > 0.20 %).

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 specific requirements presented in Table III and washed.

TABLE III

AGGREGATE SIFTING REQUIREMENTS

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

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 mm to 220 mm. Polycarboxylate-based superplasticizer was used to obtain the required mortar flow.

Mix compositions per one test specimen are presented in Table IV. The mortar was made in accordance with cement class requirements as per EN 196-1 [18].

TABLE IV

AMOUNT OF MATERIALS PER SPECIMEN

Mix	Cement, g	Aggregate, g	Water, ml	Plasticizer, g	V/C	Flow value, mm
I	200	450	94	0,2	0.47	220
II	200	450	94	0,5	0.47	205
III	200	450	94	0,5	0.47	205

The elongation of specimens is measured by a digital dial indicator with the scale interval of 0.001 mm. The indicator is fixed in the frame 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 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 with 1 M NaOH alkali solution at 80 °C.

Containers with the specimens are placed into the climatic chamber 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:

$$Expansion = \frac{100 \cdot (L_n - L_0)}{l}, \%, \qquad (4)$$

where: L_0 – measurement of the specimen just before the 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.

The density of hardened specimens was measured in accordance with LST EN 12390-7:2009, the flexural strength was measured in accordance with LST EN 12390-5:2009, and the compressive strength was measured in accordance with LST EN 12390-3:2009.

III. RESULTS

The density, flexural strength, and compressive strength values of hardened specimens are provided in Table V. The assessment of fine aggregate (sand) and coarse aggregate (gravel) revealed that in the case of fine aggregate higher compressive strength value up to 64.5 MPa and the lowest flexural strength of 3.6 MPa was obtained.

TABLE V

PHYSICAL AND MECHANICAL CHARACTERISTICS OF THE SPECIMENS AFTER 28 DAYS OF HARDENING

Mix	Density, kg/m³	Flexural strength, MPa	Compressive strength, MPa
I	2256	3.60	64.50
II	2221	4.39	58.50
III	2283	4.30	56.00

Figure 1 illustrates the results of the expansion test in the specimens with sand (Mix I). After 14 days of storing in 1 M NaOH solution the average elongation of the prisms was 0.2 mm, and expansion was 0.13 %. As observed in Figure 1, the elongation of prisms with sand is even, in contrast to the prisms with gravel. Sand is considered a very likely to be alkali-reactive aggregate. Compared to the specimens with coarse aggregates, the expansion of the specimens with sand is greater. After 56 days of storing the average elongation of the prisms is 0.51 mm, and the expansion is 0.32 %.

Fig. 1. Expansion of mortar prisms with sand (to Day 56). Mix I**.**
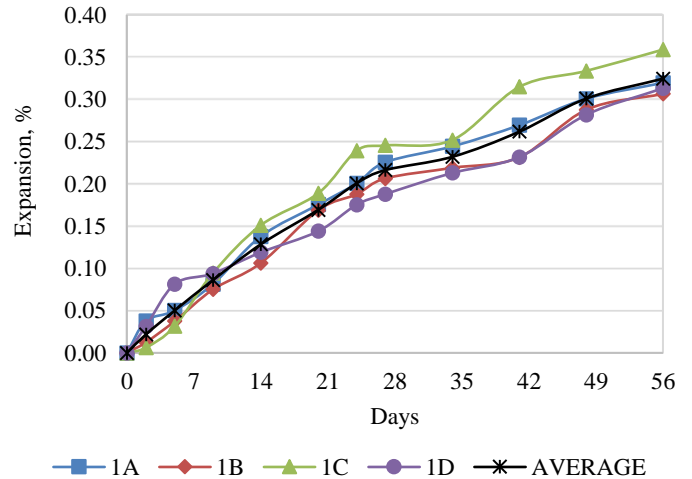
The picture of the specimen made of Mix I after 56 days of storing in 1 M NaOH is presented in Fig. 2. More significant damage occurred only on the surface of the specimen. The observed cobweb, micro-cracking and gel scales on the surface are the indicators of alkali corrosion. The scales result from the leaching of alkali and silica gel or calcium hydroxide (or both) from the cracked hardened cement paste. Intensive expansion of the prisms (Mix I) stored in 1 M NaOH alkali solution at 80 °C temperature occurs. Alkaline silica gel is the reason of destruction. Interactive destruction occurs in the mortar. Surface micro-cracking characteristic of alkali corrosion is observed. After 56 days of storing all prisms are damaged.
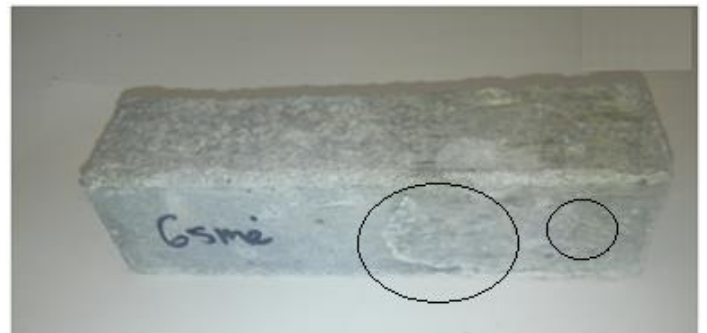

Fig. 2. Pictures of the sample (Mix I) after 56 days of storing in 1 M NaOH solution.
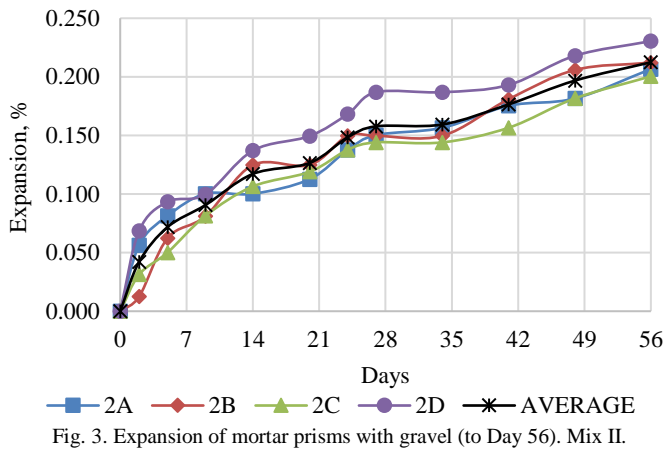
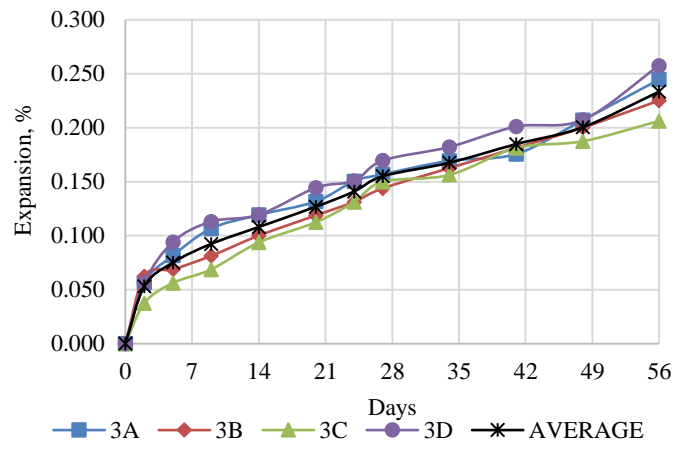Fig. 3. Expansion of mortar prisms with gravel (to Day 56). Mix II.

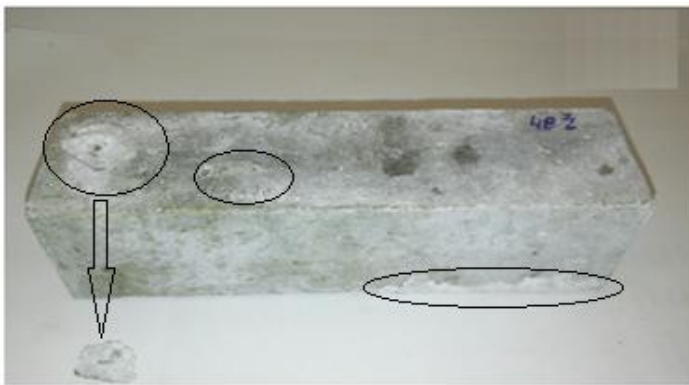Fig. 5. Expansion of mortar prisms with the second type of gravel (to Day 56). Mix III.

Fig. 4. Pictures of the sample (Mix II) after 56 days of storing in 1 M NaOH solution.

Fig. 6. Pictures of the sample (Mix III) after 56 days of storing in 1 M NaOH solution.

The testing results of prisms made of Mix II are presented in Fig. 3. After 14 days the average expansion of the prisms is 0.12 %.

The picture of the specimen made of Mix II after 56 days of storing in 1 M NaOH solution is presented in Fig. 4. Visual inspection revealed surface flaking, the contact zone of flakes and hardened cement paste, micro-cracking and gel scaling on the surface. The average elongation of the prisms was 0.36 mm, and the average extension was 0.21 %. The tests revealed that gravel is a very likely to be alkali-reactive aggregate.

The test results of the specimens made of Mix III with another type of gravel are presented in Fig 5. The tests showed that expansion after 14 days of storing in 1 M NaOH increased: the average elongation was 0.23 mm, and the average expansion was 0.11 %. After 56 days the average elongation was 0.42 mm, and the average expansion was 0.23 %. According to the test results, this gravel is also a very likely to be alkali-reactive aggregate.

The picture of the specimen made of Mix III after 56 days of storing in 1 M NaOH is presented in Fig. 6. The expansion resulting from alkali and aggregate reaction on the surface of concrete causes the breaking of cone-shaped parts of the surface above the aggregate, leaving the recess of unprotected reactive aggregate. The chipped-off cone-shaped recess is seen below the prism.

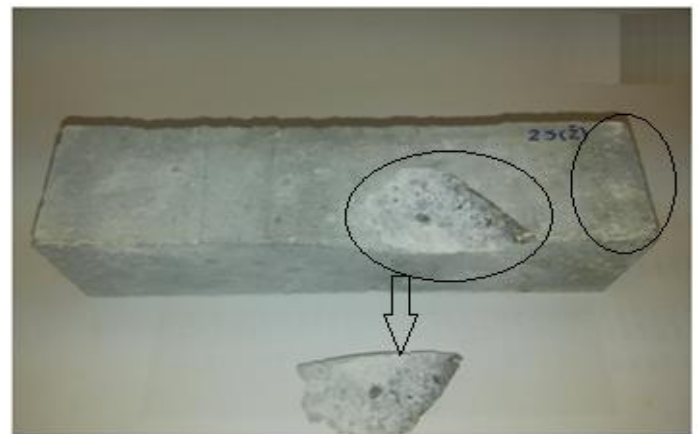Mortar expansion deformations in the prisms occur due to the impact of internal forces that cause cracking and mortar destructions. The extension is caused by alkaline silica gel. The interaction of mortar particles is disturbed and that is the reason of local spalling. Surface flaking is clearly seen in Figures 4 and 6.

Optical microscopy testing of the specimens revealed the contact zones of hardened cement paste and cracked surface. The microscopy images of the zones, where alkali corrosion reaction occurred, revealed a rough and cracked surface with clearly seen reacted particles (Fig. 7 and 8). The reaction products in the figure are white.

Figures 7 and 8 illustrate that alkali corrosion reaction, i.e. reaction products, caused a significant deterioration of the prisms, especially those made of Mix II and III. Alkali corrosion reaction involves three components: amorphous watery and cryptocrystalline SiO_2 (opoka, opal and flint), sodium and potassium hydroxide (alkali) and calcium hydroxide (portlandite). Deteriorated surface of the coarse aggregate is shown in the pictures. The coarse aggregate is surrounded by the zone of reaction products. This zone is very porous and has a great amount of glass phase. Behind the reaction product zone there is a hardened cement paste matrix, where reaction products are also seen.
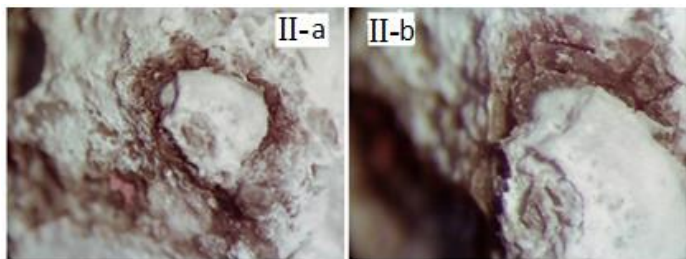
Fig. 7. Fragment of Mix II specimen. The contact zone of the hardened cement paste and aggregate is cracked (image of optical microscopy, image increased: a) × 25; b) × 50).
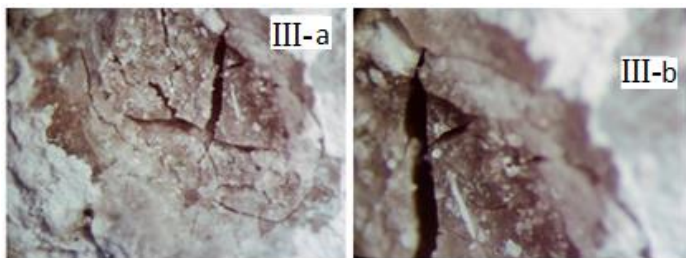

Fig. 8. Fragment of Mix III specimen. The contact zone of the hardened cement paste and aggregate is cracked (image of optical microscopy, image increased a) × 25; b) × 50).

IV. CONCLUSION

It was found that alkali corrosion of the aggregates in the mortar is a result of reaction between sodium and potassium hydroxide (alkali) from the cement and active SiO_2 present in the aggregates. This reaction creates big internal stresses in the mortar which cause deformations, cracking and destruction of concrete.

The tests revealed that Lithuanian gravel quarries are polluted with reactive particles containing amorphous silica dioxide that react with sodium and potassium hydroxide from the cement and cause alkali corrosion of the mortar.

It was found that alkali corrosion in the mortar results in the scaling of concrete surface and development of micro-cracks in all directions along the surface.

It was found that surface micro-cracking leads to the slitting-off of cone-shaped parts of the mortar leaving the unprotected reactive aggregate at the bottom of the recess. Such destructive behaviour is typical of the aggregates originating from Lithuania.

It was found that according to AAR 2 methodology fine and coarse aggregates from Lithuanian quarries belong to Group II, i.e. very likely to be alkali-reactive aggregates. The expansion of specimens made of the mortar containing these aggregates after 14 days exceeds 0.1 %: in the case of fine aggregate, the average expansion after 14 days is 0.13 %, and in the case of coarse aggregates the average expansion ranges from 0.11 % to 0.12 %.

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