

Application of RILEM test methods for alkali-silica reactivity evaluation of Polish aggregates for concrete road structures

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Abstract

Results of experimental investigations on the reactivity of crushed aggregates produced from sedimentary and igneous rocks are presented. Applied test methods, consistent with RILEM and ASTM standards are implemented in technical specifications of Polish national highway administration. The range of the investigations covered coarse aggregates produced from crushed limestone, dolomite, sandstone and greywacke rocks, as well as from basalt, melaphyre, porphyry, granite and gabbro. The aggregates were selected on the basis of their mechanical and physical properties to be adequate for making durable concrete for highway applications. The mineral composition of the aggregates was evaluated with regard to their reactive SiO₂ content. A considerable amount of reactive minerals: chalcedony, tridymite and microcrystalline quartz were found in the grains of the porphyry and melaphyre aggregates. No reactive forms of quartz were found to be present in the mineralogical composition of the limestone aggregates and the dolomite aggregates. Micro- and cryptocrystalline quartz occurred in the particles of the greywacke aggregates and the sandstone aggregates. On the basis of the expansion of mortar and concrete of specimens, the coarse aggregates produced from crushed rocks were classified into three reactivity categories. A sodium-potassium-calcium silicate gel was found and its composition was consistent with that reported in the literature as characteristic of the alkali-aggregate reaction products. The tests revealed consistent assessment of the reactivity of the aggregates.

Keywords: alkali silica gel; expansion tests; microcrystalline quartz; petrographic analysis; technical specifications

1. INTRODUCTION

During a period of intense economic growth after 1989, Poland has become a significant aggregate producer in Europe [1]. Since 2004, the aggregates for concrete are classified according to PN-EN 12620 [2] standard that is in common use in European countries. The standard requirements in respect to alkali-aggregate reactivity are left to be decided locally at the place of use. Aggregate producers in Poland issue declarations of performance in accordance with PN-EN 12620 on the basis of the degree of potential alkali-reactivity reactivity that is determined as per PN-B-06714-46 [3]. This is a quick method that consists in testing the aggregate reaction with sodium hydroxide and determining the aggregate mass loss. So called zero degree of potential reactivity determined in this way has been considered for three decades as the adequate indicator of the lack of reactive aggregate components, and that it provides adequate protection against the harmful alkali-aggregate reaction. Unfortunately, in several cases this was not a reliable rating [4]. The contradiction in the assessment of the reactivity of some domestic aggregates by the chemical method and RILEM methods was demonstrated for crushed

aggregates from igneous and sedimentary rocks [5], as well as for some domestic gravel and carbonate aggregates.

Symptoms of concrete deterioration due to alkali-silica reaction (ASR) are not reported frequently in Poland. Góralczyk [6] made a survey of concrete structures deteriorated due to ASR and found only few road viaducts and concrete buildings affected. Actually, identification of symptoms of an adverse alkali-silica reaction is not a routine activity during structural inspections. Symptoms of ASR are often difficult to distinguish from concrete damage caused by freezing-thawing, deicing salts, sulphate attack, steel corrosion or excessive drying shrinkage. The difficulty to differentiate ASR signs from other destructive mechanisms that occur simultaneously may explain a little importance of detailed ASR risk analysis. Many engineering structures built in the '70s and' 80s of the 20th century in Poland have already been demolished and replaced, without special search for symptoms of ASR (Fig. 1.1). Based on the data provided by General Directorate for Roads and Motorways (GDDKiA), it is known that about 3/4 of the bridge structures on national roads are younger than 30 years [7]. Therefore, there are relatively few objects old enough to exhibit advanced ASR symptoms that could be expected, if such potential would exist. For more than two decades the use of low-alkali cement $<0.6\% \text{Na}_2\text{O}_{\text{eq}}$ was the most widespread for bridge construction. In the context of planned major investments in new highways system and some negative experience on German highways [8], [9], it becomes important to provide an up-to-date solution for materials selection adequate for a long term durability of concrete.



Fig. 1.1: Viaducts of Trasa Łazienkowska in Warsaw before demolition in 2012 with concrete spalling, corroded steel reinforcement, cracks and exudations, possibly due to ASR [4]

Extensive knowledge and practical experience collected in North America, Europe and Japan enabled the development of strategies for recognizing reactive aggregates and counteracting the harmful effects of reactions in concrete. These are contained in such guidelines and standard recommendations as AASHTO [10], RILEM [11], DAfStB [12] as well as in numerous up-to-date review papers. The appropriate approach to aggregate selection for concrete transportation infrastructure was developed in Poland too [13]. The objective of this paper is to discuss the developed test procedures to evaluate the potential alkali-silica reactivity of crushed aggregates produced from sedimentary and igneous rocks and to provide local insight into the risk of harmful alkali-aggregate reaction in concrete.

2. MATERIALS AND METHODS

2.1 Materials

Sedimentary rock aggregates as well as aggregates of igneous origin were selected (Table 2.1) because of their abundance in Poland's geological resource balance. Basalt, granite and gabbro rock aggregates are often used for road structures of high durability. Selected crushed aggregates have particularly good physical and mechanical properties and are potentially suitable for making durable concrete bridges and pavements. When used to manufacture concrete specimens, the aggregates listed in this table were

used in combination with non-reactive coarse aggregate (amphibolite). Physical properties of aggregates determined according to standard procedures PN-EN 12620 are presented in [14] and [15]. Portland cement CEM I 52.5R (Cement 1) was used, with an alkali content of $\text{Na}_2\text{O}_{\text{eq}}=0.88\%$, the highest available Na_2O equivalent in cement which is present on the Polish market. As a reference cement, CEM I 42.5R (Cement 2) from Norcem A.S (Norway) as recommended in RILEM Recommendation [11] was been used. The fineness of Cement 1, determined using the PN-EN 196-6 [16] method, amounted to $525 \text{ m}^2/\text{kg}$ and the cement soundness (Le Chatelier method) was lower than 1 mm. The Blaine specific surface in Cement 2 amounted to $546 \text{ m}^2/\text{kg}$ and the soundness (Le Chatelier) was 0 mm determined using the PN-EN 196-3 [17] method. The sodium oxide equivalent was equal to 1.12%.

Table 2.1: Set of selected domestic aggregates

Rock origin		Aggregate designation	
igneous	volcanic	basalt, 2/8 mm	B
		melaphyre, 2/8 mm	M
		porphyry, 2/8 mm	P
	plutonic	granite, 2/8 mm	Gr
		gabbro, 2/8 mm	Ga
sedimentary	clastic	sandstone, 2/8 mm	S
		greywacke, 2/8 mm	Gw
	chemogenic	limestone, 2/8 mm	L
		dolomite, 2/8 mm	D

2.2 Testing methods

2.2.1 Petrographic analysis

The epoxy-impregnated thin sections were prepared from each aggregate. The largest individual grains were selected, washed, dried, glued into the reference glass and then epoxy vacuum impregnated. The specimens were ground and polished to get thin sections of $20 \pm 2 \mu\text{m}$ in thickness. The Pelcon automatic thin section machine was used (Fig.2.1) and the specimen preparation procedure was described elsewhere [18]. The analysis of thin sections was performed using an Olympus BX51 microscope in plane-polarized light (PPL), cross-polarized light (XPL), XPL with λ plate (XPL-G). Images were acquired using an Olympus DP25 digital color camera, automatic moving table Prior ES11BX/B, and analySIS software. The petrographic analysis was performed according to RILEM AAR 1.1 in [11] and ASTM C 295 [19].

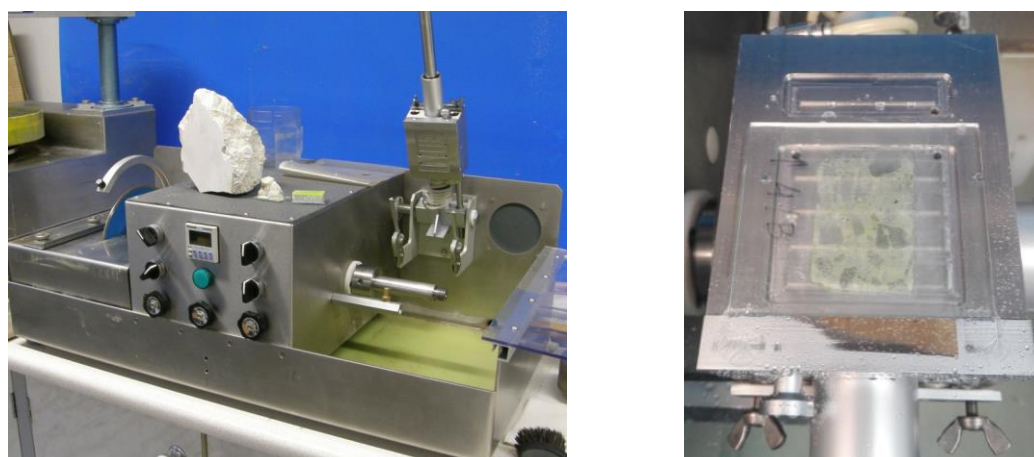


Fig. 2.1: The view of automatic thin section machine and the thin section

2.2.2 Accelerated mortar bar tests

The accelerated mortar bar test was performed according to PB/1/18 procedure included in [13] based on RILEM AAR-2.1 in [11] and ASTM C1260 [20] procedures. Fifteen mortar compositions (9 aggregates with Cement 1 and 6 aggregates with Cement 2) were tested. Three 25x25x285 mm mortar bars with steel studs were prepared using each aggregate-cement combination. After 24h, the specimens were demoulded, submerged in distilled water and heated to 80°C. After next 24h, the bars were removed from water, measured (zero reading) and immersed in 1M NaOH solution previously heated to 80°C. The specimens inside the containers filled with NaOH were stored in thermostatic chamber, their length was periodically measured for at least 14 days.

2.2.3 Long term concrete prism tests

Selected aggregates (greywacke, porphyry, dolomite, basalt, granite and limestone) were subjected to concrete prism testing according to PB/2/18 procedure included in [13] based RILEM AAR-3 in [11] and ASTM C1293 [21] procedures. Standard procedures were adjusted to allow for locally available cement and metric sieve gradation. The aggregates were crushed and tested in combination with non-reactive coarse aggregate (amphibolite). The content of cement was 420 kg/m³, w/c ratio was 0.45 Concrete mixes were prepared with Cement 1 and addition of NaOH to mixing water in order to boost equivalent alkali content to 1.25% of mass of cement. Three concrete prisms 75x75x285 mm with steel studs were cast per each aggregate. The prism were stored at 38±2°C and high humidity (RH>95%) above water layer inside the tightly sealed containers for a period up to 1 year. Length change measurements were taken periodically: immediately after demoulding and after 1, 2, 4, 13, 26, 52 weeks of conditioning.

2.2.4 Microstructural investigation using SEM

After completion of the mortar bar test, the specimens for microstructural investigation were obtained by slicing these specimens with a slow speed diamond saw. The specimens 50x30x10 mm were then dried in an oven at 50 °C for 3 days, vacuum-impregnated with a low-viscosity epoxy, lapped and polished using a special procedure for SEM specimens. Each specimen was prepared in such a way that the polished face which was to be examined was a cut surface from the middle of the bar. The specimens were coated with a thin layer of carbon for about a minute and a strip of conductive tape has been attached to each specimen. Each of the specimen was thoroughly examined using JEOL JSM-6380 LA SEM-EDX in the backscatter mode using an acceleration voltage of 15 kV. The recommendations for ASR gel identification [22-23] were followed.

3. RESULTS AND DISCUSSION

3.1 Reactive minerals identification

The petrographic analysis focused mainly on the presence of potentially reactive minerals and the content and size of SiO₂ crystals. In all the analysed grains of the aggregates produced from igneous rocks, the SiO₂ crystals have been found. Potentially reactive components, such as chalcedony and tridymite, have been found in melaphyre M while microcrystalline quartz and volcanic glass were present in porphyry P, Fig. 3.1. Plagioclases, pyroxenes and olivine phenocrystals were distinguished as the primary minerals in basalt aggregate B (Fig. 3.1.); the secondary minerals in the form of brown titanite hornblende, and the accessory minerals – the opaque minerals and apatite. Porphyry aggregate P was classified as trachyandesite with a porphyritic texture. The aphanitic quartz-feldspar mass consisted mainly of feldspars, plagioclases and phenocrystals (orthoclases), amphiboles and pyroxenites. The accessory components were opaque minerals and volcanic glass. The melaphyre aggregate M was composed mostly of feldspars, mainly plagioclases and opaque minerals (iron oxides), which were scattered quite regularly in the whole aggregate grains, as shown in Fig. 3.1. The sparse melaphyre grains contained calcite crystals. All the analysed grains contained voids and cracks filled with chalcedony and micro and cryptocrystalline forms of quartz (Fig. 3.1). Gabbro aggregate Ga contained mainly plagioclases (labradorite-bytownite) and metamorphosed amphiboles (hornblende), while granite aggregate Gr contained quartz and alkali feldspars as the primary components, biotite as the secondary component and amphiboles as the accessory components (Fig. 3.1).

No potentially reactive minerals like opal, chalcedony, tridymite, highly strained quartz or microcrystalline quartz (crystal size 10-100 µm) were found in aggregate grains originating from sedimentary rocks in the group of chemogenic rocks (limestone L and dolomite D). Characteristic rhomboidal dolomite crystals were found in aggregate D, Fig. 3.2. In the aggregates from the group of clastic rocks

(greywacke Gw and sandstone S), quartz constituted most of the mineral content. Medium-grained and fine-grained monomineral quartz crystals were main constituents in the grains of sandstone aggregate S. Also feldspars, muscovite and biotite (the latter was partially converted into chlorite) were present (Fig. 3.2). The greywacke aggregate Gw was characterized as quartz-lithic wacke, where numerous fine quartz and mica grains were visible in the matrix (Fig. 3.2). Among the numerous lithic components in the greywacke aggregate, microcrystalline and cryptocrystalline form of quartz (crystal size less than 10 μm) were observed. A detailed description of domestic rock aggregates containing the potentially reactive minerals was presented in [24].

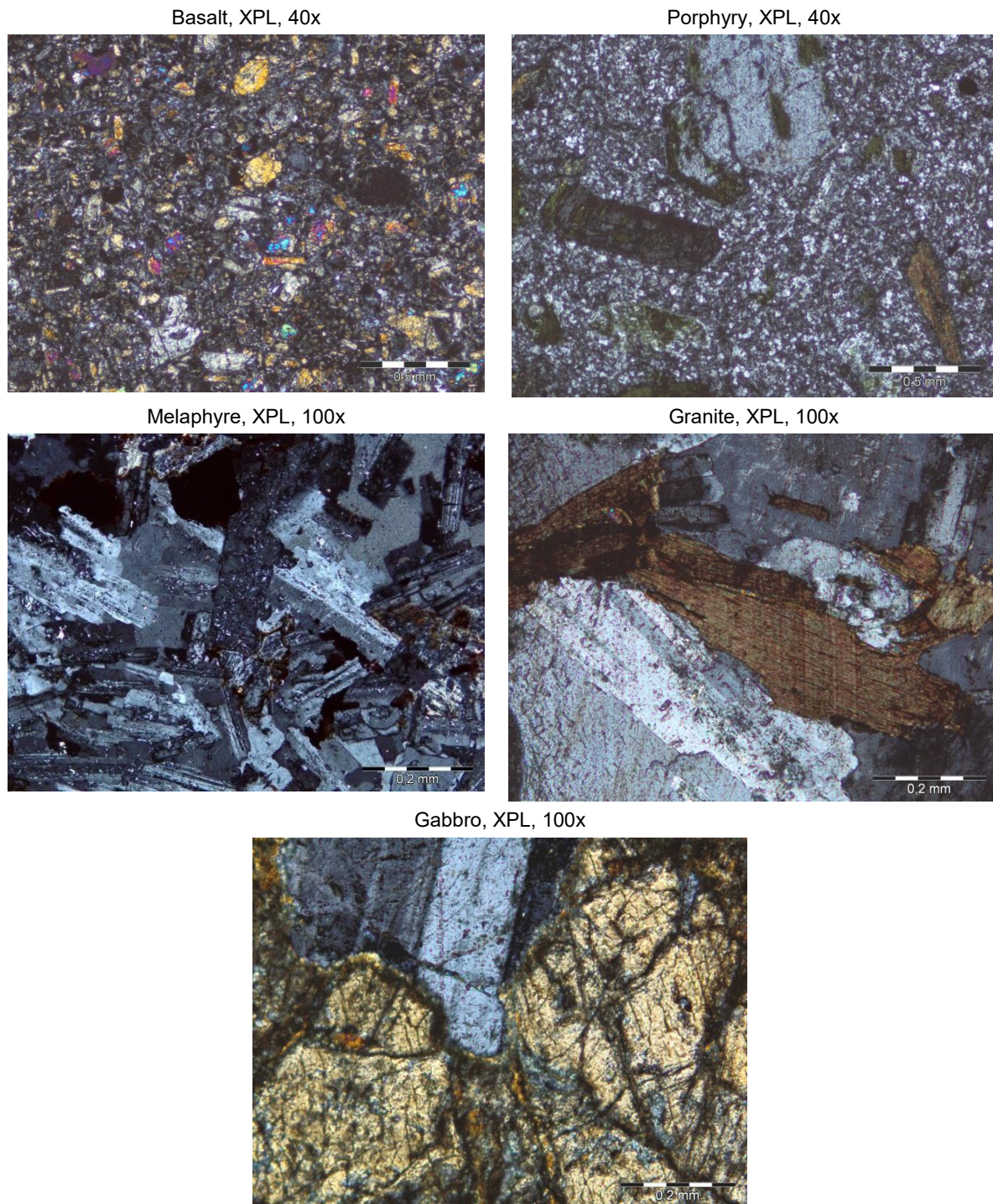


Fig. 3.1: Thin section photomicrographs with visible characteristic minerals in analyzed aggregate produced from igneous rocks

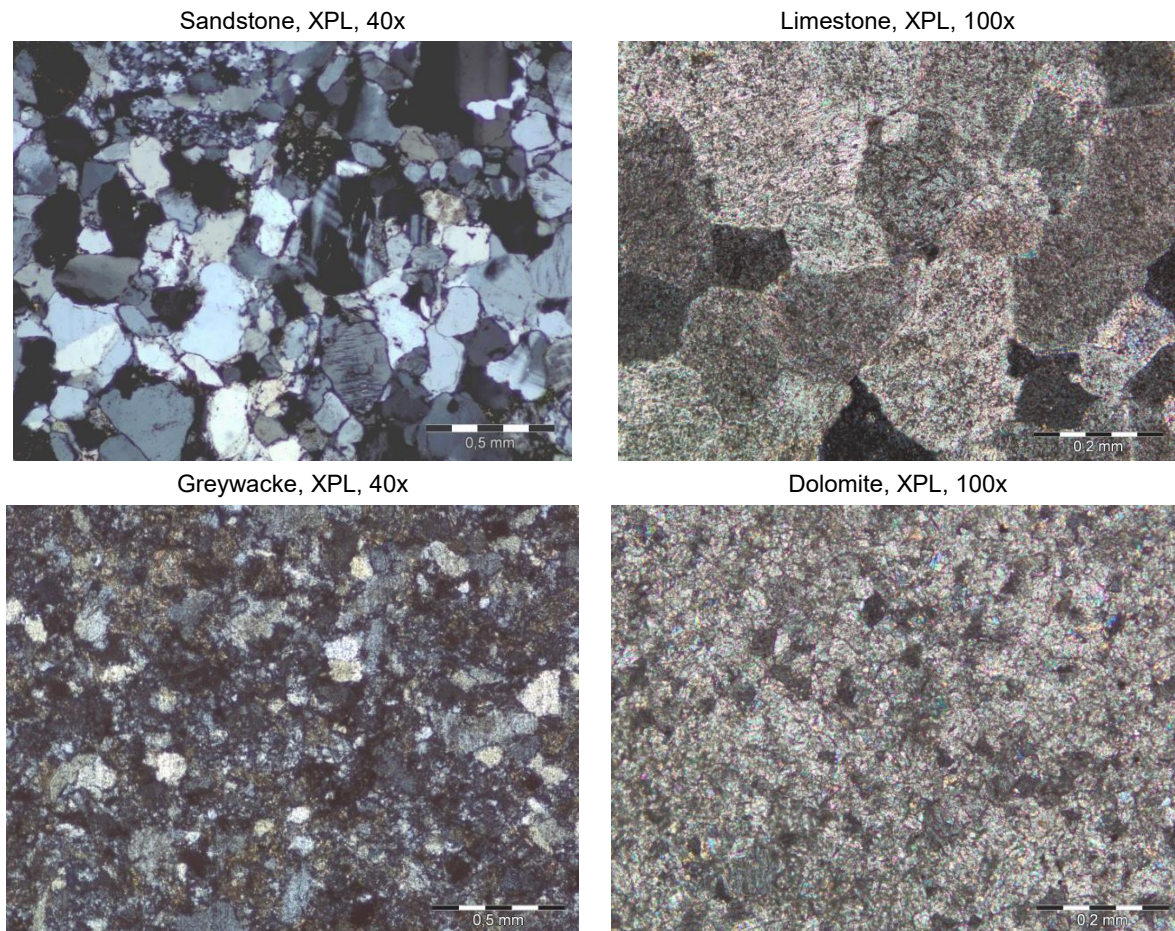


Fig. 3.2: Thin section photomicrographs with visible characteristic minerals in analyzed aggregate

3.2 Expansion of mortar specimens

The expansion of mortar bars with Cement 1 and Cement 2 is presented in Fig. 3.3 and Fig 3.4, respectively. The length change (the average of 3 specimens) versus time of exposure to sodium hydroxide solution at the temperature of 80°C is shown. Aggregates of clastic origin showed the highest expansion at 14 days of exposure among all tested aggregates – sandstone 0.341% and greywacke 0.258%. Igneous rocks exhibit diverse reactivity – from moderately reactive (the 14 day expansion higher than 0.10% but not higher than 0.30%) like melaphyre, porphyry to non-reactive (the 14 day expansion not higher than 0.10%) like basalt, gabbro, granite. Specimens with chemogenic sedimentary aggregates (limestone and dolomite) had quite low expansion – 0.029% and 0.004% respectively. The mortar bars with Cement 2 showed equal or greater expansion than specimens with Cement 1 in all analyzed aggregates.

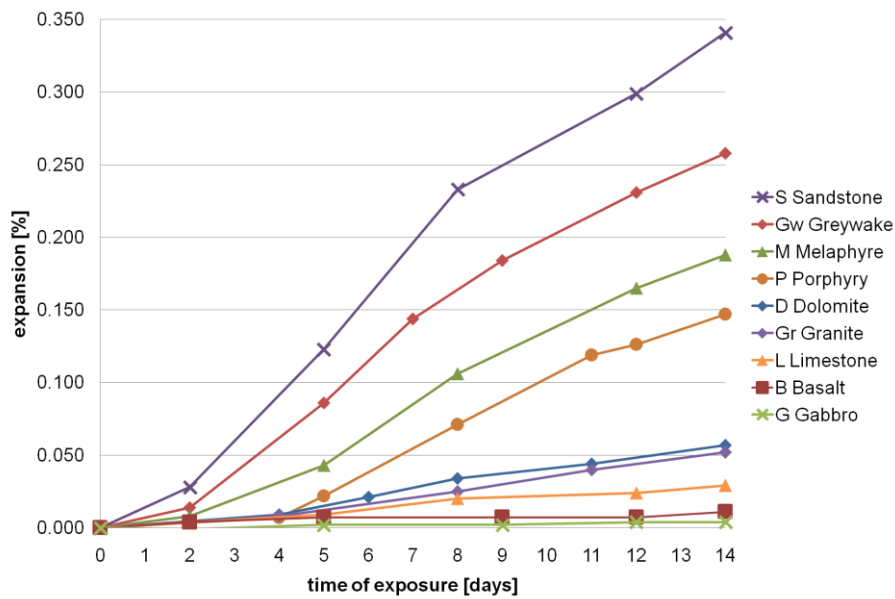


Fig. 3.3: Expansion of mortar bars with Cement 1 exposed to 1 M solution of NaOH and temp. of 80°C

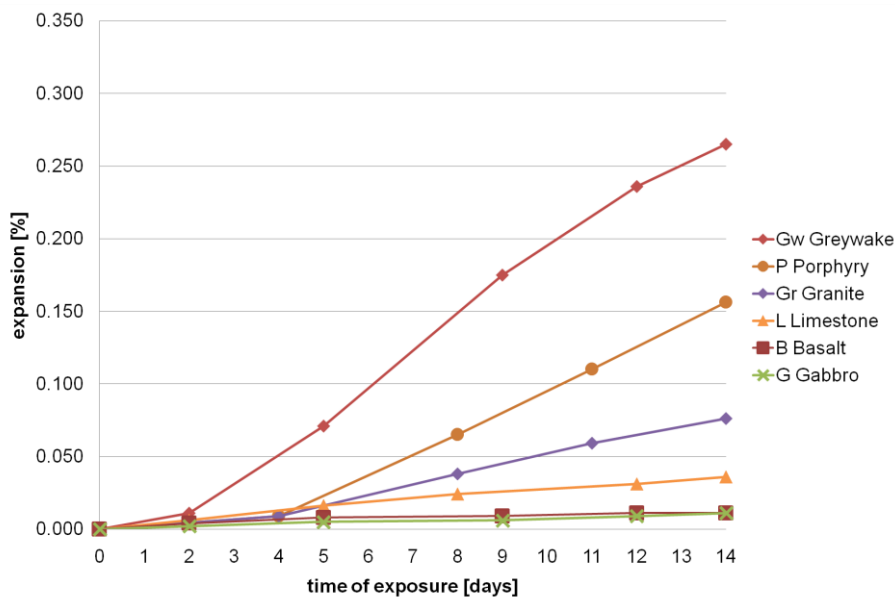


Fig. 3.4: Expansion of mortar bars with Cement 2 exposed to 1 M solution of NaOH and temperature of 80°C

3.3 Expansion of concrete prisms

In Fig. 3.5 the expansion of concrete specimens exposed to high humidity for a period up to 1 year is shown. The highest expansion was observed in prisms with greywacke aggregate – more than 0.09% after 52 weeks. The porphyry had elevated expansion – 0.045% after 1 year. The rest aggregates showed low expansion, approx. 0.03% after 1 year. The applied limits for the expansion at 1 year were: not more than 0.04% for non-reactive aggregate, more than 0.04% but no more that 0.12% for moderately reactive aggregate. These results are consistent with the results of mortar bar test.

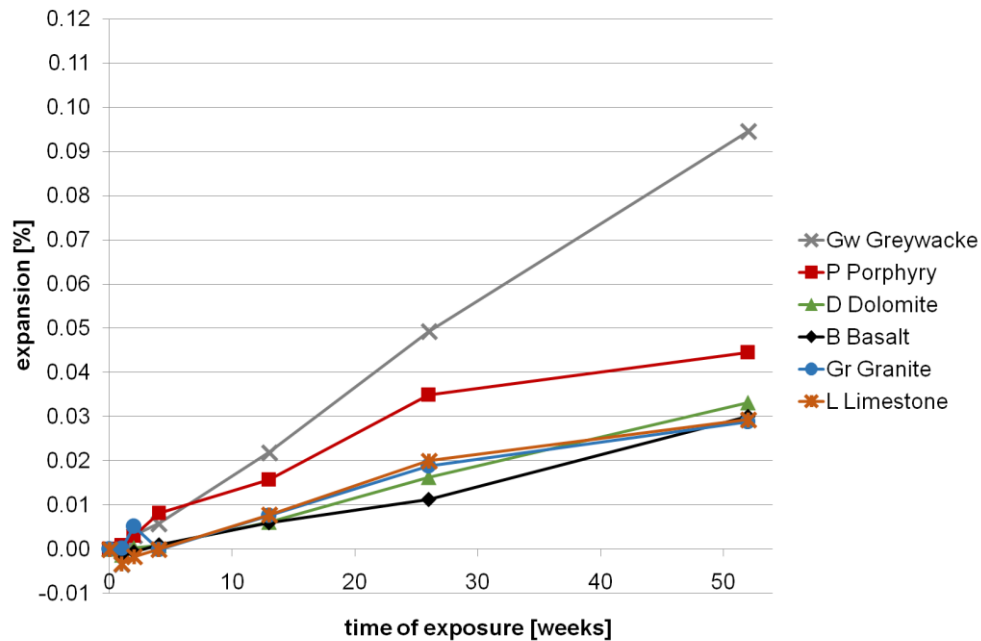


Fig. 3.5: Expansion of concrete prisms exposed to high humidity conditions and temp. of 38°C

3.4 Presence of ASR reaction products in mortars

In Fig. 3.6 the polished surface of a mortar bar specimen is shown as observed in reflected light under the stereoscopic microscope. Fig. 3.7 shows images seen using scanning electron microscope on polished sections of the same specimens (after the carbon sputtering). The crack network which was visible on the mortar bar surface was also present in the whole volume of specimen made with porphyry, melaphyre and greywacke. The characteristic numerous cracked grains of porphyry and greywacke, filled with a transparent or white gel are shown in Fig. 3.7. Also cracks in the cement matrix and air voids were found to be filled with the gel. The continuity of the filling of cracks in the aggregate grains and cracks as well as voids in the cement matrix is indicative of the intensity of the alkali-silica reaction and shows the direction of gel filling.

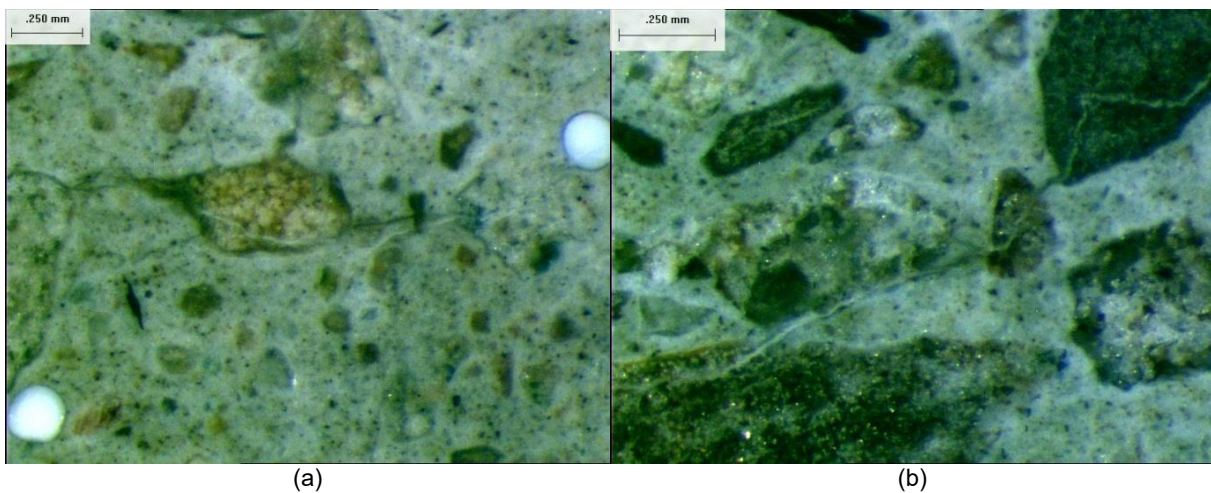


Fig. 3.6: Polished sections of mortar specimens observed using an optical microscope: visible voids and cracks filled with the white gel present in cement matrix and in aggregate grain: a) porphyry, b) greywacke; scale bar 0.250 mm

SEM microphotographs with the EDS results of the alkali-silica gel composition analysis in microareas are shown in Figs. 3.7-3.8. The results of the mortar bar microstructure analysis are consistent with the

results of the accelerated expansion measurements. The expansion of mortar bar specimens was caused by the expansive Si-Ca-Na-K gel resulting from the presence of reactive SiO₂ mineral components in the aggregate. An alkali-silica gel was detected, with much more higher content of sodium than potassium, what follows from testing of specimens in 1 M NaOH solution. Mapping of the distribution of silicon, calcium, sodium and potassium elements in the greywacke aggregate was performed. The results suggest that the alkali-silica gel displaced from the aggregate grains to the cement matrix and continued to fill the air-voids in the matrix. The composition of ASR gel was characterized by the indicators provided in Table 3.1. The alkali and calcium/silicon ratios based on the EDS analysis for at least 35 microareas (points) within the ASR reaction products filling the cracks are presented. The composition of reaction products, estimated on the basis of a SEM-EDS analysis, is typical [25] and the proportions: (Na+K)/Si and Ca/Si are within the range reported in the literature.

Table 3.1: Composition of ASR reaction products in mortar specimens

Composition indicator	Greywacke	Sandstone	Melaphyre	Porphyry
Na/Si	0.31	0.24	0.25	0.18
(Na+K)/Si	0.37	0.31	0.30	0.20
Ca/Si	0.47	0.59	0.76	0.45

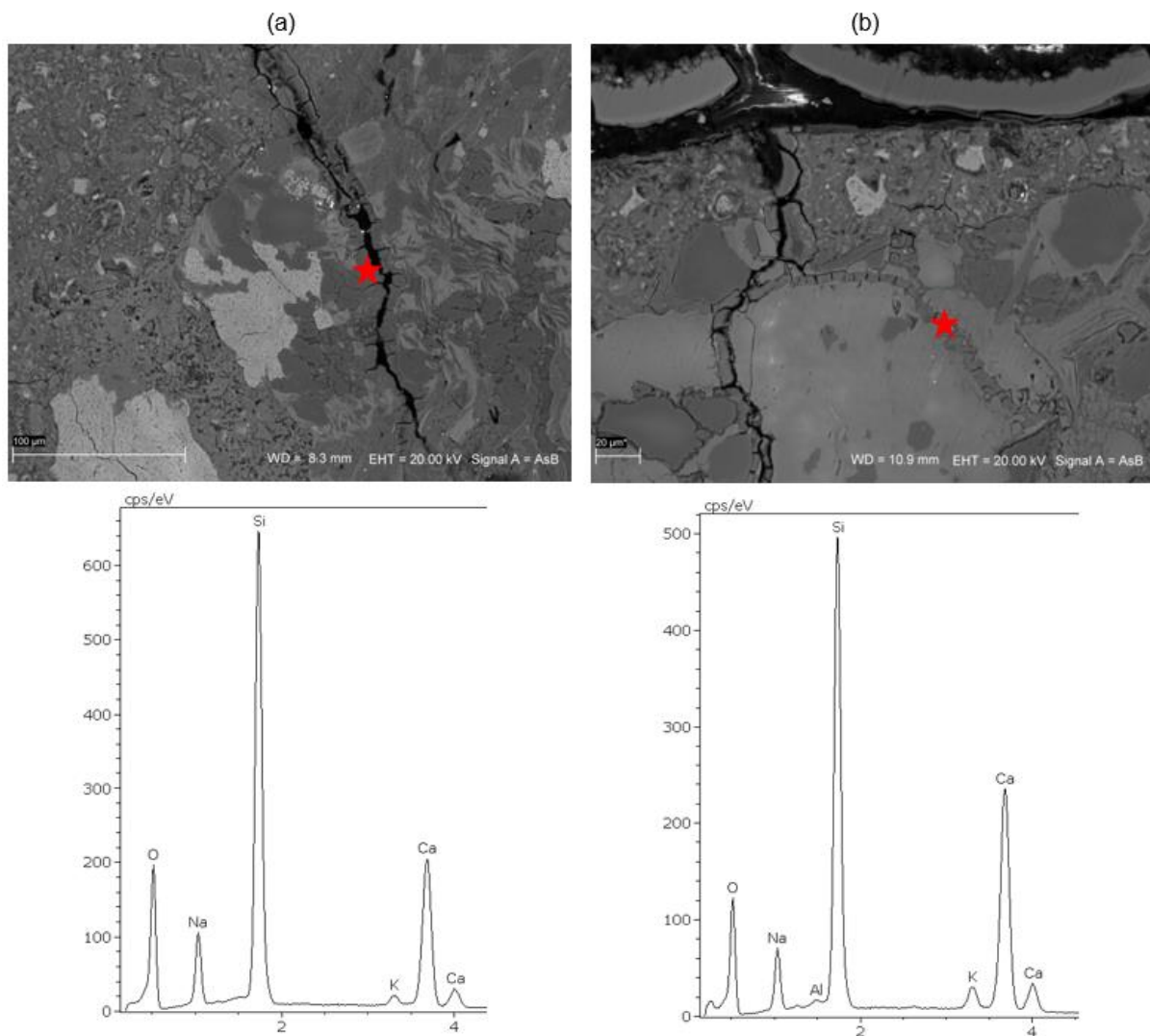


Fig. 3.7: SEM microphotograph with visible cracked aggregate and cement matrix filled with Si-Ca-Na-K gel: a) greywacke, scale bar 100 µm b) sandstone, scale bar 20 µm

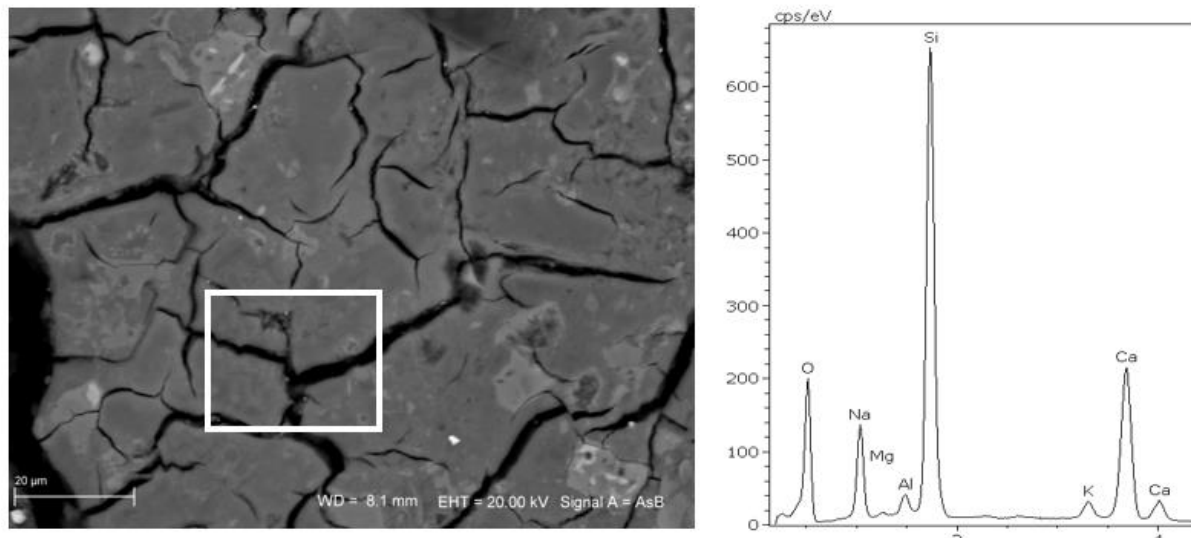


Fig. 3.8: SEM microphotograph of ASR reaction product filling cracks in a greywacke aggregate particle and the results of the EDS analysis in selected area; scale bar 20 μm

4. POLISH TECHNICAL GUIDELINES

The developed technical guidelines related to concrete transportation structures and highway pavements [13] include also recommendations for preventing damage due to alkali-silica reaction in new concrete structures. The recommendations follow the strategy defined by AASHTO [10] and RILEM AAR-7,1 in [11]. The general concept of the guidelines includes the following essential elements:

- the analysis of documented aggregate use history,
- petrographic analysis of aggregate and expansion tests, as presented in previous sections 2.2.1, 2.2.2 and 2.2.3,
- aggregate suitability classification for intended use, primarily identification of non-reactive aggregates,
- selection of preventive measures, as appropriate,

The sustainable use of available natural aggregates for the production of concrete, while eliminating or reducing the effects of a harmful ASR reaction, requires specific preventive measures. These measures are selected according to the analysis of risks due to alkali-silica reactions. The analysis consists of:

- categorization of aggregate reactivity (R reactivity category);
- categorization of environmental impacts on concrete (environmental impact category E), taking into account the size and purpose of concrete elements and the design time of use, as well as other factors and impacts that may affect the risk of a harmful alkali-silica reaction;
- classification of building and engineering objects (object class S) depending on their importance and the economic, social and environmental effects of a possible alkali-aggregate reaction.
- Technical guidelines [13] provide the following solutions for preventing premature deterioration of concrete as a result of the alkali-silica reaction:
- the basic solution consists in the selection of mineral aggregate with the R0 reactivity category (non-reactive aggregate – i.e. with negligible content of components susceptible to the alkali-silica reaction) and limitation of the use of reactive aggregates;
- strongly and very strongly reactive aggregates (reactivity categories R2 and R3) are excluded from the use in concrete intended for road engineering structures and highway pavements;
- in the case of moderate reactivity of the aggregate (reactivity category R1), the reduction of its harmfulness consists in the use of prescriptive restrictions in the composition of concrete; the restriction concerns the reduction of alkali concentration in the concrete pore liquid by the use of low-alkali cements and / or the use of mineral additives: silica fly ash or granular blast furnace slag.

When determining the suitability of aggregate for concrete components of the structure, it is necessary to take into account the operating conditions of the environment, such as changes in humidity and

temperature, the use of de-icing agents and variable mechanical loads. The sodium chloride is the most frequently used deicer. The penetration of NaCl into concrete is thought to enhance the ASR phenomena. That is reflected by a relevant formulation of distinct moisture classes for concrete elements exposed to extraneous moisture and to external supply of alkalis by de-icing agents and for concrete elements exposed to extraneous moisture, to external supply of alkalis by de-icing agents and to fluctuating loads, like in highway pavements [12]. For expert ASR performance evaluation of aggregate in concrete exposed to external alkali supply, the test method “60°C concrete test with external alkali supply”, developed originally at VDZ in Germany [26] and covered by draft procedure RILEM AAR-12 [27] is implemented. Several combinations of coarse crushed aggregate and fine natural aggregate were tested in concrete designed as for heavily trafficked highway pavement [28] and significant effects of fine aggregate on the expansion of concrete was revealed.

5. CONCLUSIONS

New technical guidelines were developed in Poland for the classification of domestic aggregates in respect to alkali-aggregate reaction in concrete used in road pavements and engineering structures. The specified test methods and criteria are based on RILEM recommendations with local modifications to allow for the use of local Portland cement of the highest available alkali content 0.9% Na₂O_{eq}. Experimental tests on domestic coarse aggregates from crushed sedimentary and igneous rocks performed using these test methods resulted in the consistent conclusions.

Petrographic analysis on thin sections revealed no reactive mineral components in the aggregates produced from limestone and dolomite as well as from basalt, gabbro and granite rocks. Significant amounts of reactive minerals such as chalcedony, tridymite and microcrystalline quartz were found in porphyry and melaphyre aggregates.

Mortar bar and concrete prism expansion tests resulted in the consistent classification of aggregates:

- basalt, gabbro and granite, limestone and dolomite – category R0 (non-reactive),
- porphyry and greywacke– category R1 (moderately reactive).

Si-Ca-Na-K reaction products from ASR were found in mortar bars with the R1 and R2 category of aggregates, confirming their susceptibility to the reaction with sodium and potassium hydroxides. Microscopic examinations showed that the expansive Si-Ca-Na-K gel had moved from the aggregate grains to the cement matrix and completely filled the voids in the matrix.

6. ACKNOWLEDGEMENTS

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