## POTENTIAL OF ALKALI SILICA REACTION AS A FUNCTION OF REACTIVE FORM OF QUARTZ IN FINE AGGREGATE

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### ABSTRACT

In the present study the potential of alkali-silica reaction (ASR) in fine fraction of aggregate was analyzed. The investigation was focused on mineral composition of siliceous sand and its influence on ASR. Three siliceous sands from different origin and localization in Poland were tested. Petrographic analysis on thin sections was conducted. The automatic image analysis was used to estimate the content of reactive minerals (micro- and crypto-crystalline quartz). The XRD measurements were performed. Alkali-silica reactivity of fine aggregate was tested by mortar-bar test according to ASTM C1260 Standard.

Petrographic analysis showed that all tested siliceous sands contained reactive form of quartz, micro- and cryptocrystalline. Mortar-bar tests according to ASTM C1260 indicated that one from the selected sands exceeded expansion over the limit and was considered as reactive. The content of reactive minerals in sands estimated by automatic image analysis corresponded to ASTM C1260 results. The higher content of reactive form of quartz in siliceous sand, the larger expansion of mortar-bar test.

### **Keywords**

Siliceous sand, Alkali-Silica Reaction (ASR), digital image analysis, micro- and cryptocrystalline quartz, expansion.

## **INTRODUCTION**

The alkali–silica reaction (ASR) takes place between the alkaline pore solution in cement based composites and various metastable forms of silica contained in aggregate. The silica structure is dissolved by the nucleophilic attack of OH– ions, and the highly degraded silica structure behaves as a hygroscopic silica gel. Alkali silica reaction is an actual problem in many concrete constructions [1-4]. According to Kurtis and Monteiro [5], any aggregate with silica as its constituent has a potential to provoke and develop ASR. The alkali reactivity of aggregate depends on their geological origin, mineralogical composition and texture. Aggregates that contain reactive forms of SiO<sub>2</sub> react rapidly (like opal, tridymite or cristobalite) with sodium and potassium ions or react slower (like chalcedony, cryptocrystalline quartz and strained quartz).

While the coarse aggregate derived from the crushing of solid rocks is characterized by the homogeneity of physical and mechanical properties, the natural fine aggregate is considered as nonhomogeneous material. The natural fine aggregate in the form of natural sand is usually used in amount up to 30 percent of the total volume of aggregate in concrete.

The results regarding the application of various types and origin of sand in concrete technology were presented in [6-8]. Hasdemir et al. [6] analyzed the influence of natural sand composition on concrete strength. They showed that although all the tested sands revealed to be suitable for application as fine aggregate in high-strength concrete, but natural sands that contained smectite-type clays had insufficient quality compared to other sands. They stated that the chemical composition of natural sands, especially the Na<sub>2</sub>O content, was important for predicting the strength properties of hardened concrete. Olonade et al. [7] tested sands from quarries in Nigeria investigating their suitability for concrete production and considering standard physical and chemical properties. Xiao et al. [8] presented a critical review of existing studies on the effects of using sea-sand as raw materials for concrete production. As it concerns concrete durability, it has been shown that the use of sea-sand may have a significant effect on chloride-induced steel corrosion, but only a negligible effect on the carbonation process of concrete.

Very little information about natural siliceous sand according to the potential of ASR is available. The necessity to study fine aggregates for alkali-silica reactivity was discussed by Lukschova et al. [9]. They suggested to compare different test methods (petrographic analysis, mortar-bar test) to get more reliable results. Naziemiec [10] tested reactivity of Polish sands. Their mineral composition varied greatly and as a result they were characterized by various aggregate reactivity classes. Alkali-silica reactivity of natural sands was also tested by Hasdemir et al. [11]. They showed that some natural sands are more reactive than others, due to both high content of SiO<sub>2</sub> and of  $K_2O$  and  $Na_2O$ .

The investigation of the influence of the mineral composition of siliceous sand on the alkali-silica reactivity has been conducted. The research was focused on detailed microscopic observations and image analysis as well as on XRD measurements. The alkali-silica reactivity was measured by accelerated mortar-bar test according to ASTM C 1260 [12].

#### MATERIALS

Three different natural siliceous sands from Poland were used. The localization of their sources is presented in Fig. 1 denoted as it follows:

- S1-river sand;
- S2 fossil sand;
- S3 fossil sand.

For the estimation of potential alkali-silica reactivity of tested sands, according to ASTM C1260, cement CEM I 42.5R with 0.88%  $Na_2O_{eq}$  was used. The cement fineness, determined using the PN-EN 196-6 method, amounted to 525 m<sup>2</sup>/kg. The increase in gauging point spacing was lower than 1 mm.



Figure 1. Limits of Pleistocene periods of glaciations in Poland with marked locations of the analysed sands. Early Pleistocene: N, Narewian; Ni, Nidanian. Middle Pleistocene: Li, Liwiecian Limits of Middle Pleistocene: Odranian and Wartanian are not marked because they do not affect the tested material

#### **TESTING PROCEDURE**

Automated image analysis was used for characterization of mineral composition of siliceous sand in thin sections. Thin sections ( $20 \pm 2 \mu m$  thickness) of analyzed fine grained aggregates were prepared by using PELCON equipment. Microscopic analysis was conducted with OLYMPUS BX51 petrographic microscope with an automatic moving table and a DP25 digital color camera. Image analysis was performed by Image Pro Plus software, the single photographs of aggregate were stitched by Image Composite Editor software. The thin section analysis was performed in transmitted and cross-polarized light (XPL) and also with  $\lambda$  plate (XPL-G). The gypsum plate was applied for estimation of quartz particle sizes.

The X-ray diffraction (XRD) was used to identify the main minerals in the siliceous sands S1-S3. To evaluate the content of quartz in tested sands, semi-quantitative (S-Q) weight percentage analysis was used, which is one of the tools in EVA 5 evaluating software from Bruker. It was based on the intensity ratio of specific peaks in analyzed pattern and intensities of reference corundum patterns stored in device database. The minor minerals like albite were not taken into account. All sand specimens were powdered and sieved through a 0.045 mm sieve. A Bruker D8 Discover diffractometer was used with voltage ratio of 40 kV and 40 mA lamp current. As an X-ray source a copper lamp was used. The scan step size was  $0.02^{\circ}$  with collection time 1 s, and in the range  $2\Theta$  Cu K $\alpha$  from 5 to 65° [13].

Alkali-silica reactivity of selected sands was tested using ASTM C 1260 standard, as described in [1]. The sand were tested in fractions provided from producers. No crushing and sieving has been carried out. Three mortar-bar specimens from each sand have been made. Fine aggregate to cement ratio of 2.15 and water-to-cement ratio of 0.47 were maintained. Three mortar bars were tested in 1 M NaOH in 80°C. The criterion 0.15% of expansion after 14 days as a limit of

reactivity was used according to National Technical Guidelines [14].

#### TEST RESULTS AND DISCUSSION

Thin sections microscopic analysis has shown the predominant content of monomineral quartz grains (Fig.2 – 1). The micro- and cryptocrystalline quartz was found in all tested siliceous sand (Fig.2 – 2). The reactive form of SiO<sub>2</sub> was found in single fine grained chert particles. The examples of reactive form of quartz grains are presented on Figure 2. To compare potential alkali-silica reactivity of tested sands, the quantitative image analysis was made. The schematic schedule is presented on Figure 3.



Figure 2. Sections of micro- and cryptocrystalline form of quartz (fine grained chert), (S1-S3), 1 – monomineral quartz, 2 – fine grained chert, 3 – granitic rock



Figure 3. Scheme of the image analysis procedure of the micro- and cryptocrystalline quartz estimation

The differences in content of micro- and crypto-crystalline quartz in tested sands were observed. The highest content of reactive form of silica was found in S1 sand, the lowest in S3 sand, Table 1. The higher total content of  $SiO_2$  was in a river sand S1, and the lowest in fossil sand S3, similarly as in the case of reactive quartz content.

Table 1. Content of micro- and cryptocrystalline form of quartz in analysed sands obtained b	y
automatic image analysis	

	S1 (river sand)	S2 (fossil sand)	S3 (fossil sand)
Micro- and cryptocrystalline quartz content [%]	5.2	2.1	1.3
Total SiO <sub>2</sub> content [%]	99.9	98.5	86.3

In all tested sand specimens, the main peak in XRD measurements corresponds to the characteristic mineral of fine aggregate: quartz  $SiO_2$ . In each fine aggregate, the  $SiO_2$  and  $CaCO_3$  peaks are visible but with different intensity. The smallest peaks of calcite and the highest peaks of quartz were found in siliceous river sand S1 and fossil sand S2. The X-ray diffraction patterns are given in Figure 4, and content of quartz and calcite is given in Table 2.



Figure 4. X-ray diffraction (XRD) patterns with main characteristic peaks in the tested sands

Sand	quartz	calcite	quartz/calcite
Siliceous river sand S1	92.4	0.1	924.0
Siliceous fossil sand S2	84.4	3.0	28.1
Siliceous fossil sand S3	75.4	6.7	11.3

Table 2. The content of the quartz and calcite estimated by XRD method, %

The influence of the reactive form of  $SiO_2$  in siliceous sand on ASR potential was confirmed by accelerated mortar-bar test results. On Fig. 5 the results of mortar bar expansion exposure in 1 M NaOH in 80°C are presented. According to ASTM C1260 Standard, the aggregate is treated as non-reactive if expansion after 14 days is lower than 0.1% regardless of the aggregate fraction. For natural fine aggregate, 14-days criterion 0.15% of expansion according to National Technical Guidelines [14] was applied. River sand S1 is classified as reactive and fossil sands S2 and S3 as non-reactive aggregate. Additionally, after 28 days the S1 mortar-bar expansion is almost twice higher, than after 14 days, which indicates that the reaction does not diminish.



Figure 5. The results of sand mortar-bar expansion according to ASTM C1260 after 14 and 28 days of exposure in 1 M NaOH in 80°C

The results of mortar-bar expansion confirmed the image analysis results concerning the microand cryptocrystalline quartz. The highest expansion was observed in sand with the highest amount of micro- and cryptocrystalline quartz. The S3 sand, which was evaluated as non-reactive during expansion test, has the lowest content of micro- and cryptocrystalline quartz. The highest content of total quartz by image analysis was found in S1 river sand and it corresponded to the highest total content of quartz obtained by XRD measurements. Regardless the XRD results, where the total content of SiO<sub>2</sub> was found out it is necessary to carry out thorough tests on the mineral composition. As a conclusion it appears that XRD testing is insufficient to assess the alkali-silica reactivity of aggregates for roads.

#### CONCLUSIONS

From the experimental results, the following conclusions can be drawn:

- The tested siliceous sands have various contents of reactive form of quartz (micro- and crypto-crystalline).
- The highest content of micro- and cryptocrystalline SiO<sub>2</sub> was found in river sand S1.
- Expansion of mortar bars according to ASTM C1260 corresponded to the content of micro- and cryptocrystalline quartz in tested sand – the higher content of reactive SiO<sub>2</sub>, the higher expansion of mortar bars.
- Automated image analysis of mineral composition on thin sections allowed to pre-evaluate the ASR suitability of the aggregate.

The above conclusions should be taken into account when selection of sources of sand is made for application in high quality road pavements.

#### ACKNOWLEDGEMENTS

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# **BRITTLE MATRIX COMPOSITES**

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