

## **ASR PERFORMANCE TESTING OF AIR ENTRAINED CONCRETE EXPOSED TO EXTERNAL ALKALIS**

**Karolina Gibas (1), Michał A. Glinicki (1), Mariusz Dąbrowski (1), Daria Józwiak-Niedźwiedzka (1), Aneta Antolik (1) and Kinga Dziedzic (1)**

(1) Institute of Fundamental Technological Research, Polish Academy of Sciences, Warsaw, Poland

### **Abstract**

The risk of occurrence of deleterious alkali-silica reaction (ASR) in concrete should be properly minimized in major highway pavements and bridges. Real-life experiences show that even in concrete made with aggregates potentially not susceptible to ASR, it may occur under unfavourable conditions of external alkali supply at wet conditions and heavy traffic load. An experimental investigation was performed to study the susceptibility of selected Polish mineral aggregates to ASR at external alkali supply. The test method „60°C concrete test with external alkali supply” covered by draft procedure RILEM AAR-12 was implemented at IPPT PAN laboratory. Air entrained concrete specimens were exposed to cyclic temperature changes and wet-dry exposure as well as NaCl solution exposure. Several combinations of coarse crushed aggregate and fine natural aggregate were tested in concrete designed as for heavy duty highway pavement. SEM evaluation of microstructure of concrete with glacial deposit aggregate revealed visible alkali-silica gel. The effects of fine aggregate on the expansion of concrete were also revealed.

**Keywords:** air entrained concrete, alkali-silica reaction, cyclic exposure, external alkali, highway pavement

### **1. INTRODUCTION**

The issue of susceptibility of some mineral aggregates to the reaction with alkali hydroxides in concrete is still valid, despite numerous worldwide studies and publications [1]. The alkali-aggregate reaction occurs between the pore solution of concrete and the reactive minerals in aggregate grains, eventually leading to excessive expansion and cracking of concrete elements. ASR research on Polish mineral aggregates revealed reactivity of certain sands and gravels containing opal and chalcedony, sandstone, silica limestone and dolomite [2-4]. The expansion of cement mortar or concrete specimens with the tested aggregate depends on the content of potassium and sodium in cement. The impact of external alkalis

was demonstrated using the climate simulation concrete prism test [5] and such a test was used successfully to evaluate job mixtures for pavements.

For the ASR performance testing of concrete the "60°C concrete test with external alkali supply" method was developed at the German VDZ Institute [6]. The method involves cyclic exposure of concrete specimens to wetting-drying, soaking in solution of sodium chloride, while the temperature change takes place within the range from 20°C to 60°C. It has been recently included in RILEM AAR-12 draft procedure. The test method allows to evaluate the resistance to alkali-silica reaction of concrete mixtures for the moisture classes WA (concrete element exposed to extraneous moisture and to external supply of alkalis by de-icing agents) and WS (concrete element exposed to extraneous moisture, to external supply of alkalis by de-icing agents and to fluctuating loads – concrete pavements of motorway constructions) [7].

The "60°C concrete test with external alkali supply" method was implemented at the Institute of Fundamental Technological Research, Polish Academy of Sciences to assess the risk of ASR reactions in domestic aggregates in concrete [5]. The paper describes preliminary results of ASR performance tests on air entrained concrete containing crushed granite and aggregates crushed from glacial deposits in the northern part of Poland. The expansion tests with external alkali supply are compared with the standard expansion testing of mortar specimens in 1 N NaOH solution at 80°C.

## 2. EXPERIMENTAL SECTION

### 2.1 Materials and specimens

Air-entrained concrete containing crushed coarse aggregates and siliceous sand was tested (Table 1). Portland cement CEM I 42.5R was used, with an alkali content of  $\text{Na}_2\text{O}_{\text{eq}}=0.58\%$ . The water-to-cement ratio was selected following an assumed limiting value adequate for slip-formed concrete highway pavement to be exposed to XF4 aggressive environment.

Concrete mixes contained domestic aggregates with maximum grain size of 16 or 22 mm. The grain size distribution of aggregates was set according to Polish standards for concrete highway pavements. The content of fine aggregates was 30%. Air entraining admixture was used to obtain the target fresh air content of 5% to 7%.

The mixes were manufactured using a laboratory mixes of 50 litres capacity. Concrete prisms 75x75x285 mm with steel studs were made of each mix for ASR performance testing. Companion cube specimens were also manufactured for standard compression testing.

Table 1: Composition and properties of fresh and hardened concrete mixture

Mixture Designation	GD1_QS	GD2_QS	G1_QS	G2_QS	G3_QS
Type of Cement	CEM I 42.5R				
Cement [ $\text{kg}/\text{m}^3$ ]	360				
w/c	0.45				
Coarse aggregate	Glacial deposit 1, 2-16 mm	Glacial deposit 2, 2-16 mm	Granite 1, 2- 16 mm	Granite 2, 2-16 mm	Granite 3, 2- 22 mm
Fine aggregate	Natural quartz sand, 0-2 mm				
Fresh air content, A [%]	6.2	4.6	7.2	5.5	6.7
Slump [mm]	180	180	130	140	150
Compressive strength, $f_{c28}$ [MPa]	42.6	48.1	41.6	44.5	42.4

## 2.2 Test methods

The petrographic analysis of minerals in aggregate grains was performed using RILEM AAR-1 Recommendation. Thin sections of aggregate grains were prepared with a thickness of  $20 \pm 2 \mu\text{m}$ . The petrographic analysis was focused on characterisation of potentially reactive forms of silica using Olympus BX51 microscope with digital colour camera and automatic moving table Prior ES11BX/B.

Fresh mix properties were tested using standard EN methods (the slump, the fresh air content). After 28 days of curing the compressive strength of concrete on 100 cubes was tested according to PN-EN 12390. The air void content in hardened concrete was measured on polished flat sections using Image Pro 7 digital analysis of microscopic images according to PN-EN 480-11.

The ASR performance test consists of pre-storing specimens for 28 days and an alternating storage for 140 days.

Pre-storage: After casting, the prisms were protected from moisture loss and stored in the moulds at  $20 \pm 2^\circ\text{C}$  for 1 day. After demoulding the prisms were stored in a water bath for 20 days at the temperature of  $20 \pm 2^\circ\text{C}$ . The prisms were inserted into sealed containers and were placed in the thermostatic chamber (the reactor) for 6 days at a temperature of  $60 \pm 2^\circ\text{C}$  and a minimum relative humidity of 98%. Then stored 1 day in an unopened condition in a room with  $20 \pm 2^\circ\text{C}$ . At the age of 28 days the zero measurement of the mass and the length of specimens were taken.

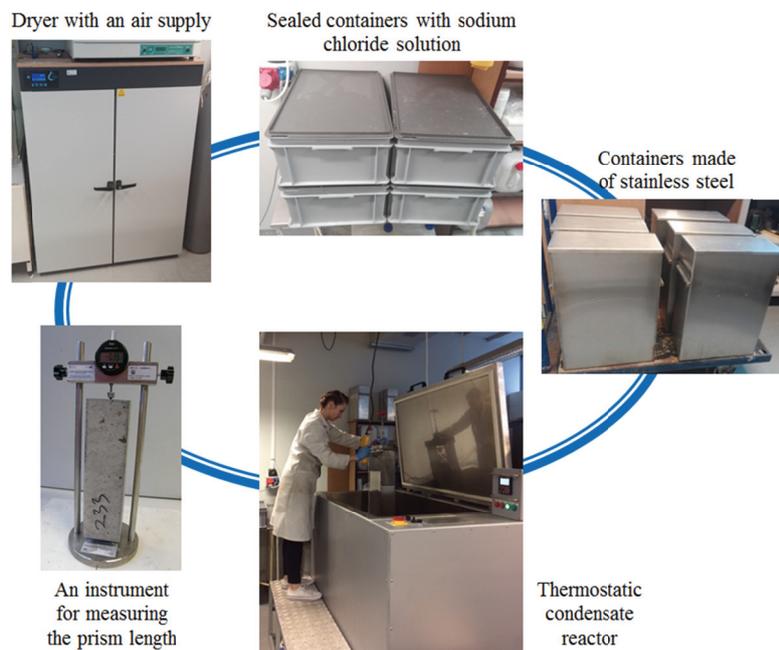


Figure 1: Alternating storage of concrete prisms with external alkali supply

Alternating storage: At the age of 28 days the prisms were exposed to the alternating storage condition. The single cycle lasted for 14 days and it was repeated 10 times. After the zero length measurement, the prisms were placed in a dryer at  $60 \pm 5^\circ\text{C}$  with an air supply for 5 days, Figure 1. Then the prisms were removed from the dryer to cool off and to be placed in containers with 3% NaCl solution at  $20 \pm 2^\circ\text{C}$  for 2 days. Afterwards the prisms were placed

over a water bath in a sealed container. The container was placed in the reactor for 6 days at  $60\pm 2^\circ\text{C}$  and  $\text{RH} \geq 98\%$ . Containers removed from the reactor were stored in an unopened condition at  $20\pm 2^\circ\text{C}$  for 1 day. Then the mass and the length of prisms were determined at the laboratory environment at  $20\pm 2^\circ\text{C}$  and  $\text{RH}=65\pm 5\%$ . The alternating storage was continued until the specimens reached the age of 168 days following their preparation. The concrete has sufficient resistance to the alkali-aggregate reaction when the final expansion is less or equal to 0.30 mm/m.

Accelerated mortar bar test (ASTM C1260/RILEM AAR-2) was used to evaluate the ASR expansion potential on specimens exposed to 1 N NaOH solution at  $80^\circ\text{C}$  during 14 days. In accordance with these standards, if after 14 days of testing the expansion of the mortar bars is lower than 0.10%, the aggregate may be considered non-reactive, and above 0.20% is highly reactive. Three mortar bar specimens  $25\times 25\times 285$  mm were prepared using Portland cement CEM I 42.5R ( $\text{Na}_2\text{O}_{\text{eq}}=0.88\%$ ) for each aggregates, which were processed by crushing and sieving to the appropriate specified by standard gradation.

Identification of alkali-silica gel was performed on polished sections with an area of  $45\times 30$  mm, cut from concrete prisms after 10 cycles of the alternating temperature exposure and out of mortar specimens after 14 days of the exposure to 1N NaOH solution at  $80^\circ\text{C}$ . The microstructure of mortar and concrete was observed using Scanning Electron Microscope (SEM) with Energy Dispersive X-Ray Analysis (EDX) [8]. Specimens were tested using Zeiss sigma VP microscope, in the backscatter mode using an acceleration voltage of 20 kV.

### 3. TEST RESULTS

The total air content (A) in hardened concrete specimens was measured and it was higher than 4.6% and smaller than 7.2%. The dispersion of the air content between concrete mixtures with different aggregate type was probably caused by a dust fraction of rocks. A large proportion of dust fraction causes the absorption of air entraining admixture and thus reduces its effectiveness. The compressive strength after 28 days of curing was from 42 MPa to 48 MPa (the average values for three specimens).

A variety of rock types was identified in crushed aggregate grains from glacial deposits: limestone, granite, diorite, sandstone, mudstone, spongiolite, quartzite and siliceous rock (chalcedony-rich rocks). Each of aggregates from glacial deposits contained quartz in its pure form, strained quartz and quartz crystals smaller than  $60\ \mu\text{m}$  and thus classifying as microcrystalline material. According to the RILEM classification this corresponds to the Class II of reactivity potential. All granites mainly consisted of plagioclase, albite and K-feldspar, quartz and biotite. The strained quartz and microcrystalline quartz were also present in granite aggregates as well as myrmekitic quartz which can increase the potential for ASR [8].

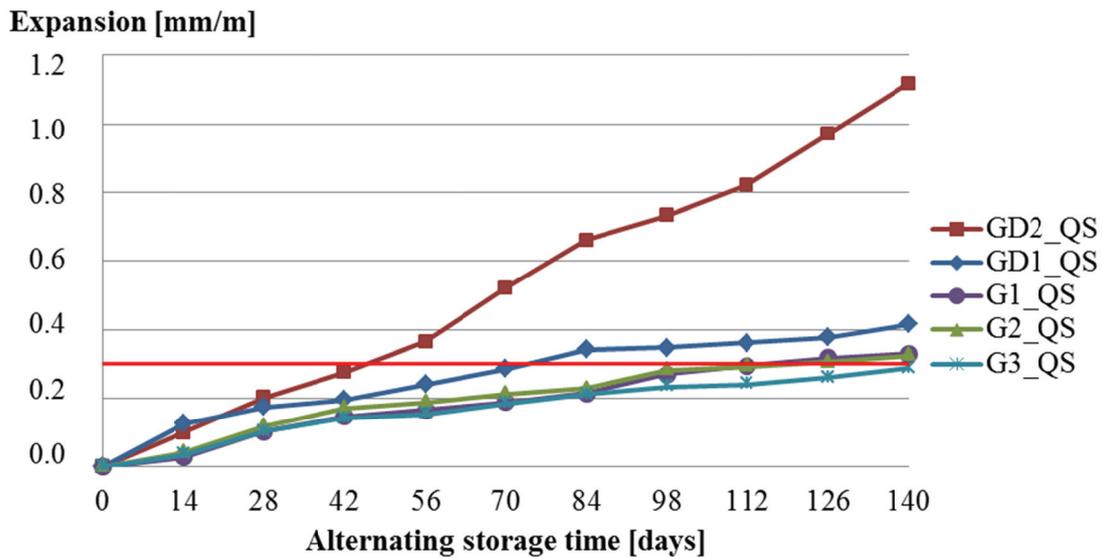


Figure 2: The expansion of concrete prisms in time during ASR performance testing with external 3% NaCl solution exposure

The relative increase of the length of concrete specimens in time is presented in Figure 2. The horizontal axis covers the time of 10 exposure cycles of specimens to alternating temperature, moisture and external alkalis. The mass of specimens increased over time: the mass gain was from 0.7% to 1.4%.

The ASR reactivity of single aggregates evaluated in accordance with ASTM C1260/RILEM-AAR-2 recommendation is shown in Figure 3.

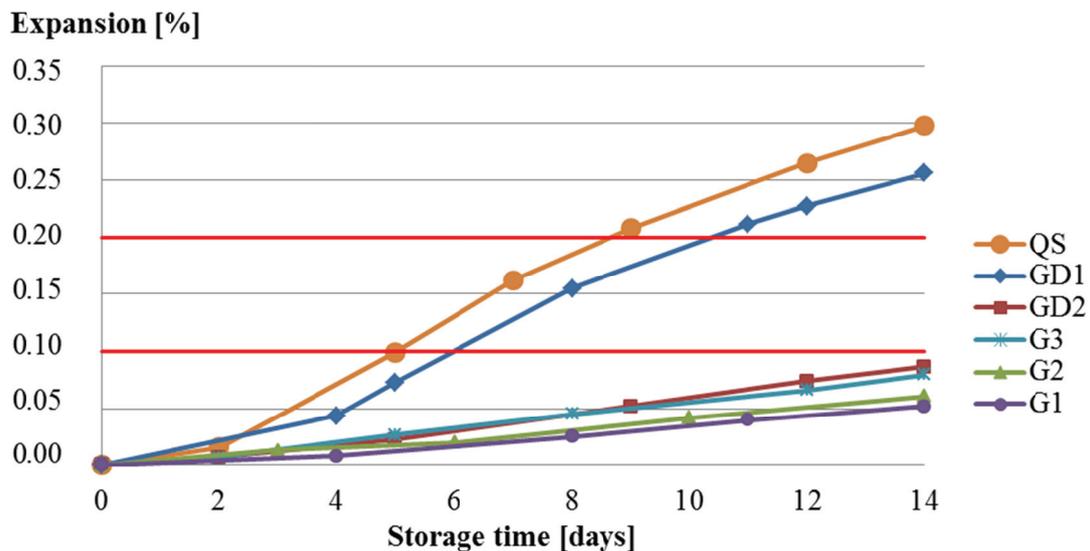


Figure 3: The expansion of mortar specimens in time in 1M NaOH solution at 80°C following ASTM C1260 procedure and RILEM AAR-2 recommendation

Examples of SEM images of specimens containing crushed aggregate from glacial deposit 1 after the termination of VDZ and ASTM tests are shown in Figures 4 – 5 along with EDS spectra at selected locations. Some cracks in the matrix were present in concrete specimens the most severe cracking was observed in concrete with aggregates from glacial deposits. ASR gel of typical composition was found; it was filling cracks and air-voids in air entrained concrete specimens.

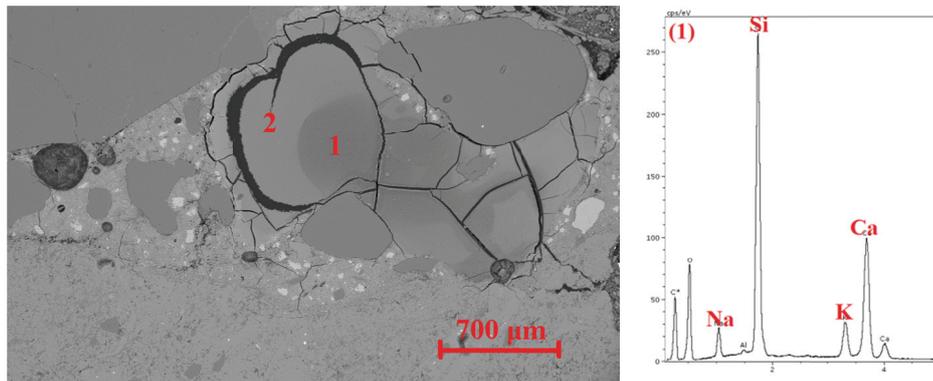


Figure 4: The microstructure of concrete with glacial deposit aggregate (GD1\_QS) after VDZ test with visible alkali-silica gel (1)(2); the scale bar is 700 μm

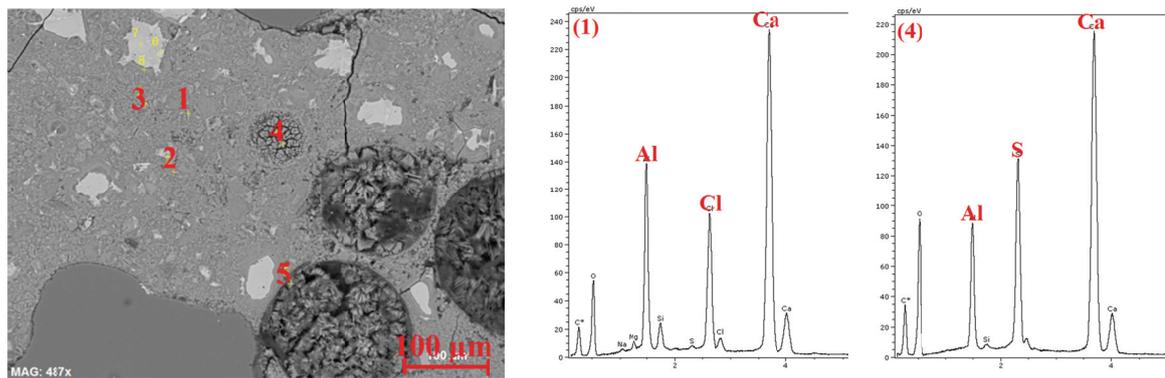


Figure 5: The microstructure of concrete with glacial deposit aggregate (GD1\_QS) after VDZ test with visible Friedel's salt in cement matrix (1)(2)(3) and ettringite deposit in air-voids (4)(5); the scale bar is 100 μm

#### 4. TEST ANALYSIS

The ASR reactivity of single aggregates was evaluated using ASTM C1260/ RILEM AAR-2 (Figure 3). Specimens containing crushed aggregate from glacial deposit 1 demonstrated the greatest expansion, above 0.25% after 14 days of storage in NaOH solution. Also the natural quartz sand exhibited a clear potential for ASR since the final expansion of mortar bars was about 0.298%. These aggregates can be considered as potentially harmful, reacting with sodium and potassium hydroxides in concrete pore solution. The other four coarse aggregates can be considered innocuous as indicated by modest final expansion of specimens.

The prolonged exposure of concrete specimens to cyclic changes of temperature and moisture, and external alkali supply resulted in steady increase of specimen length. For most

concrete mixes made with the combination of siliceous sand and crushed coarse aggregates the final expansion was above 0.30 mm/m (Figure 2). Only concrete specimens with granite 3 aggregate after 10 cycles showed the final expansion of 0.29 mm/m, slightly below the limiting value. The highest length increase was observed for concrete specimens made with crushed aggregate from glacial deposit 2 (1.12 mm/m).

To confirm that such a large expansion was caused by the formation of an expansive alkali-silica gel in concrete and mortar specimens, the microstructure was examined using SEM\_EDX after termination of the VDZ and ASTM tests. ASR gel of typical composition was found. The weakest aggregate grains were completely or almost entirely replaced by alkali-silica gel. Many of the aggregate particles were found to contain cracks filled with alkali-silica gel from which is penetrated to the cement matrix (Figure 5). The chemical analysis in microsurface revealed that it was Si-Ca-K-Na gel. The chemical composition didn't differ from the range of composition known from the literature [9-13]. The ratios of Na/Si, K/Si, Ca/Si, (Na+K)/Si were 0.27, 0.03, 0.52, 0.31, respectively, i.e. within the range characteristic for ASR gel. The alkali-silica gel partially or completely filled the air-voids. After the exposure of concrete specimens to NaCl solution, ettringite appeared in the air-voids and Friedel's salt in the matrix (Figure 5).

The crushed coarse aggregate classified as innocuous using the data in Figure 3 when combined with QS sand exhibited quite substantial expansion during the concrete performance testing. The significant reactivity of fine aggregate clearly contributed to the length increase of concrete. That is a demonstration of the significance of sand reactivity in evaluation of such aggregate combinations.

## 5. CONCLUSIONS

The following conclusions can be drawn:

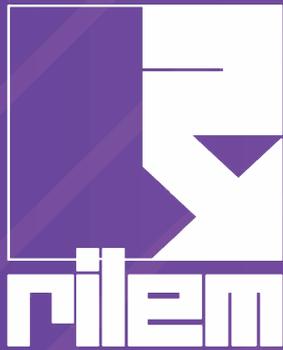
- The cyclic „60°C concrete test with external alkali supply” test method covered by draft procedure RILEM AAR-12 was effective to reveal the influence of domestic aggregate combinations on expansion of concrete specimens. The expansion of air entrained concrete specimens containing siliceous sand and crushed aggregate from glacial deposits or granite aggregate reached 0.287 to 1.119 mm/m when exposed to external 3% NaCl solution.
- Extensive amounts of ASR gel, identified using SEM-EDS, was found in specimens showing the largest length increase. The gel was filling the air-voids and replaced some aggregate grains.
- The exposure of concrete specimens to alternating temperature, moisture and 3% NaCl solution resulted in an increased occurrence of Friedel's salt and ettringite in the matrix.
- For the investigated range of slowly reacting mineral aggregates the results of concrete performance tests are broadly consistent with the prediction based on accelerated mortar bar tests.

## ACKNOWLEDGEMENTS

This paper is part of the Project “Alkali-Aggregate Reactivity of Domestic Aggregates” funded by the National Centre for Research and Development (NCBiR) and the General Directorate for National Roads and Highways (GDDKiA) in Poland in 2016-2019.

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