

## **EFFECTS OF CYCLIC WET-DRY- EXTERNAL ALKALI EXPOSURE ON MICROSTRUCTURE AND WATER PERMEABILITY OF AIR-ENTRAINED PAVEMENT CONCRETE**

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### **ABSTRACT**

Penetration of external alkalis from deicing salts into concrete pavement can promote deleterious alkali-silica reaction (ASR) and therefore reduce its long-term performance. In severe exposure conditions (wet and heavy traffic load) the susceptibility of aggregates to ASR may be aggravated. To study this phenomenon on concrete specimens the combined cyclic exposure method was developed at VDZ Düsseldorf. Such a cyclic wet-dry-external alkali exposure was used on air entrained concrete specimens to investigate its influence on the microstructure and the permeability of concrete. Air voids characteristics, mercury intrusion porosimetry, porosity accessible to water and water absorption of concrete was tested. Concrete microstructure was observed in scanning electron microscope to reveal the specific features. The applied cyclic wet-dry-external alkali exposure induced a marked decrease of total porosity of concrete and the appearance of ASR gel in cement matrix. An increase of the rate of water absorption of concrete was also found.

### **KEY WORDS**

ALKALI-SILICA REACTION / VDZ TEST / MICROSTRUCTURE / PERMEABILITY / WATER ABSORPTION

### **1. INTRODUCTION**

Presence of potential reactive minerals in aggregates and appearance of sodium and potassium hydroxide in the pore liquid of concrete can cause degradation of concrete in road pavements. Therefore, concrete mix design to avoid the ASR reaction involves appropriate selection of components, especially non-reactive aggregates and low alkaline cement. However, alkalis could ingress into concrete from deicing salt dissolving in water covering concrete pavement during winter time. The deterioration is not clearly connected with expansion of concrete (CHATTERJI 2015), because the ASR gel can fill air voids and thus disperse excess volume of ASR reaction product in air-entrained concrete (LINDGÅRD 2013). Effects of such severe environmental exposure on concrete microstructure and its transport properties are not fully understood. The paper presents the results of microstructure and permeability tests on concrete specimens exposed to cyclic wet-dry- external alkali loads, promoting ASR reaction if potentially reactive minerals are present in the aggregates.

### **2. MATERIALS AND TESTING METHODS**

Evaluation of concrete microstructure and water permeability was performed on specimens produced using 6 mixtures with different aggregate types. Concrete mixtures were designed with the same amount of cement paste according to BORCHERS (2016) assumption for bottom layer of concrete pavement – 360 kg/m<sup>3</sup> of Portland cement (CEM I) and w/c = 0.45. Crushed aggregates were selected from larger population based on the appropriate physical and mechanical properties including grain size, shape, abrasion and frost resistance. Representative aggregates of basalt, granite, limestone, dolomite, glacial gravel, amphibolite were selected. Proportion of fine and coarse aggregates was constant. Quartz sand constituted 30% by weight of all aggregates. Coarse aggregate accounted for the remaining share of 30% and 40% by weight for 2-8 mm and 8-16 mm

fraction, respectively. Cumulative grading curves of each concrete mixtures were similar, which makes it possible to compare the properties of concrete. After initial conditioning as indicated by BORCHERS (2016) one part of specimens was placed in laboratory conditions ( $22 \pm 2$  °C and RH  $\approx 60\%$ , reference specimens) and the other part was subjected to ten VDZ cycles. One cycle lasting 14 days comprised of high humidity storage at 60 °C, drying at 60 °C, soaking in NaCl solution at 20 °C. After 140 days of VDZ method exposure the concrete prism revealed linear expansion above limit ( $>0.5$  mm/m) and obtained significant expansion up to 1.45 mm/m for concrete with granite aggregates, thus we assume occurrence of ASR reaction. Small slices were cut from the specimens before and after exposure for tests using mercury intrusion porosimetry, porosity accessible to water and rate of water absorption. The air void characteristic in hardened concrete was determined using a computer-driven system of automatic image analysis (GLINICKI & ZIELIŃSKI 2008). Tests were performed using polished concrete specimens 100x100x25 mm cut from 150mm cube specimens. The measurement procedure complied with standard requirements imposed by PN-EN 480-11.

Mercury intrusion porosity (MIP) measurements were carried out using separated cement matrix from small three cores drilled from concrete specimens ( $\varnothing = 14$  mm;  $h = 20$  mm; weight  $\approx 3$  g). Cement matrix from cores were dried at 35 °C until a constant weight to avoid microcracks and then they were tested. The size of specimens for MIP analysis was linked to the size of measurement container of Quantachrome POREMASTER 60 mercury porosimeter.

The rate of water absorption was tested in accordance with ASTM C1585. Concrete prisms 76x76x50 mm were cut from concrete prisms 285x76x76 mm and were conditioned according to the ASTM standard procedure. The initial sorptivity ( $S_i$ ) was calculated based on mass intake during the first 6 h. The porosity accessible to water (E) was tested in accordance with NF P18-459. Measurements were performed on concrete prisms 76x76x50 mm cut from concrete prisms.

### 3. RESULTS AND DISCUSSION

Air voids characterization revealed the air void characteristics adequate for frost resistant concrete. The spacing factor was within the range from 0.15 to 0.20mm,  $A_{300}$  in the range from 2.2 to 3%. Obtained results allow to make an assumption about similar air voids microstructure of concrete tested specimens.

The wet-dry-external alkali exposure enhancing ASR reactivity caused a decrease of total pore volume by 3-100% in concrete specimens in respect to pore volume in reference specimens stored at dry laboratory conditions. A pronounced effect was observed for concrete containing crushed basalt, granite, dolomite and glacial gravel aggregates. The largest, twofold decrease of porosity was revealed in concrete with glacial gravel aggregates. Concrete specimens with limestone and amphibolite aggregate did not show any significant changes of total pore volume. The volume of pores larger than 1  $\mu\text{m}$  was found quite similar in specimens exposed to VDZ cycles and in reference specimens (Fig. 1) for all concrete mixtures. SEM studies revealed mesh of cracks in cement matrix close to aggregate surface (Fig. 2). According to observation by (LINDGÅRD et al. 2013) an increase of number of cracks is related to higher expansion of concrete prisms. The highest degradation of concrete was observed in concrete with glacial gravel aggregates, where cracks were observed across quartz aggregates (Fig. 2a). The width of cracks was larger than 1  $\mu\text{m}$ . This range of pore size is a limitation of use MIP technique for description of large deterioration due to ASR reaction.

The decrease of total pore volume of concrete after cyclic wet-dry-external alkali exposure was caused by decrease of volume of the most numerous pores in cement matrix from range 0.05-1  $\mu\text{m}$  (Fig. 1). After VDZ cycles pores in this range were not dominant. A probable reason of such changes was related to formation of ASR gel and Friedel's salt inside the pores. At SEM images ASR gel inside cracks and air voids was observed more often than Friedel's salt. Friedel's salt was generally present inside a compact cement matrix. According to (Gibson et al. 2016) Friedel's salt crystals do not indicate the presence of ASR reaction. It is only another reaction which is going on without impact on formation of ASR. Ingress of  $\text{Cl}^-$  ions into cement matrix could take place before

ASR reaction as is evidenced by formation Friedel's salt as a first layer in air voids before formation next layer of ASR gel (Fig. 2b). Measurements of the porosity open to water penetration according to French standard NF P18-459 did not give any significant information about microstructural changes. The porosity was from 12 to 13.5% for reference specimens. Porosity changes after the wet-dry-external alkali exposure were within a range of up to 5%. According to (BAROGHEL-BOUNY 2006) classification such porosity accessible to water corresponds to medium durability class (12-14% of porosity accessible to water). Porosity accessible to water measurements confirm the observation from MIP, which indicated no effect of VDZ cycles on large pores above 1 μm diameter.

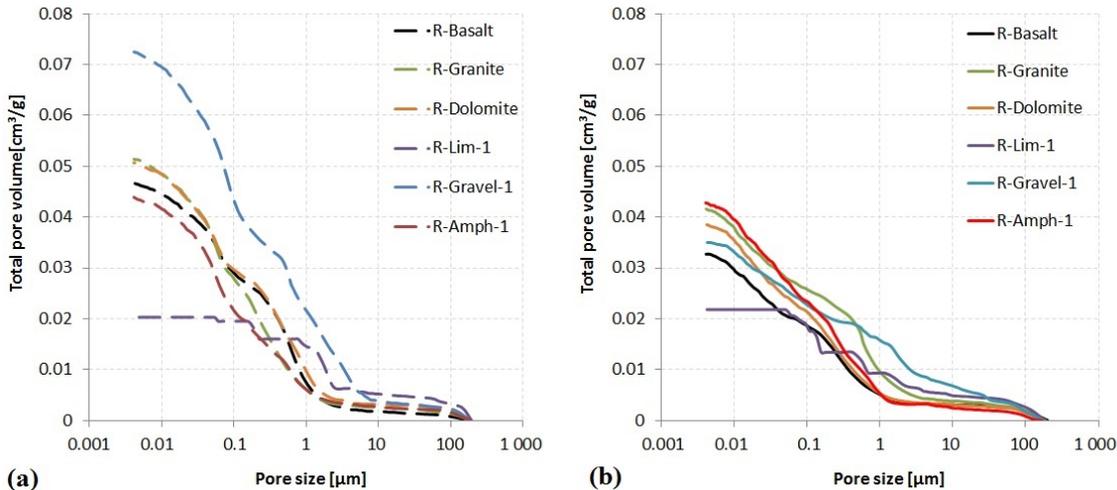


Figure 1. Changes of porosity of cement matrix (a) reference specimens, (b) specimens after VDZ cycles – MIP measurements

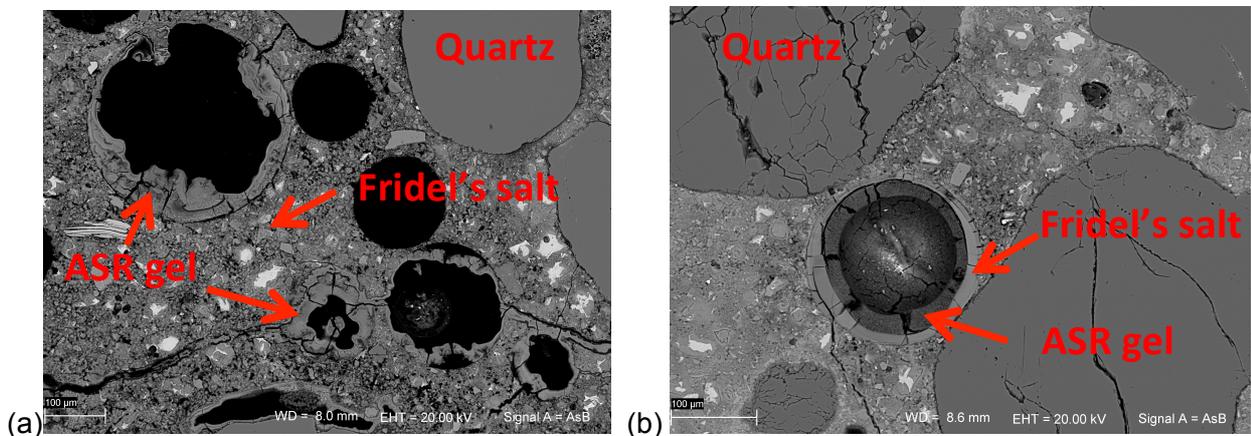


Figure 2. Example of SEM observation of concrete microstructure with different aggregate type: (a) glacial gravel (b) limestone

Capillary suction measurements revealed significant microstructural changes generated by cyclic dry-wet-external alkali exposure. Occurrence of microcracks in concrete after VDZ cycles contributed to significant increase of rate of initial water absorption (Fig. 3). In comparison to concrete specimens stored in dry laboratory conditions an increase of rate of water absorption by 2 to 5 times was obtained. The highest rate of initial water absorption was found in concrete with glacial gravel aggregates ( $27 \cdot 10^{-4} \text{ mm/s}^{1/2}$ ) and smallest for concrete with crushed basalt aggregates ( $13 \cdot 10^{-4} \text{ mm/s}^{1/2}$ ). Such results correspond to the measured expansion of concrete prisms in VDZ method.

#### 4. CONCLUSIONS

The cyclic dry-wet-external alkali exposure method significantly changed microstructure of concrete. A decrease of total porosity volume measured by MIP test was found, which resulted from a decrease of volume of dominant pores within the range 0.5-1 μm. SEM observation

revealed the appearance of many cracks in cement matrix, which were filled generally by ASR gel. Cracks most frequently appeared in concrete close to glacial gravel and rarely in concrete with basalt aggregates. Appearance of Friedel's salt was observed especially in bulk cement matrix, and rarely inside the air voids. Another information given from SEM observation was earlier formation of Friedel's salt than ASR gel in air voids. Measurement of porosity accessible to water did not show any changes after VDZ cycles but water absorption test revealed significant increase of initial water absorption. Results from water absorption measurement correspond to the expansion of concrete prisms and the number of cracks observed by SEM microscope.

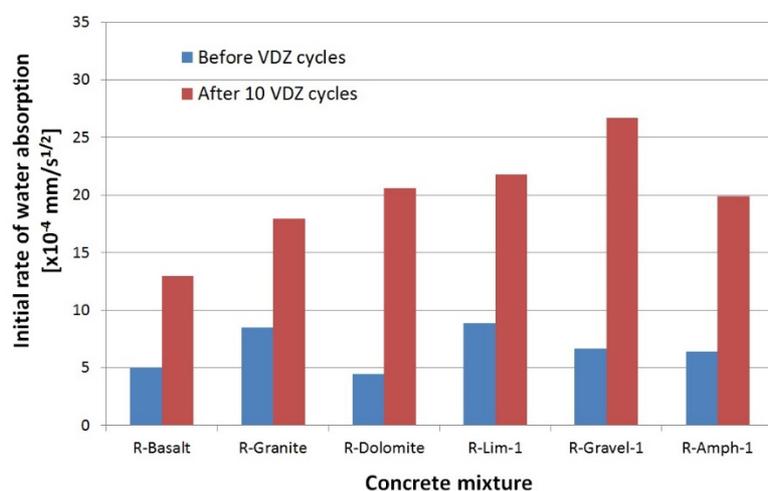


Figure 3. Initial rate of water absorption before and after 10 VDZ cycles

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