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Microstructural evaluation of the real concrete pavements with potential Alkali Aggregate Reaction signs

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Abstract

The paper presents results of the microstructural analysis of the expressway pavements concrete exhibiting signs of potential alkali-silica reaction (ASR). The analysis was performed on cores drilled from pavements from the northern part of Germany. Specimens were selected from regions of the pavements representing the highest degree of distress. The results obtained during microstructural characterization performed in the laboratory are presented and discussed.

The microstructure of concretes was investigated using microscopy in both transmitted and reflected light as well as in scanning electron microscope (SEM) operated in the backscattered (BSE). The chemical compositions of the phases of interest were determined using the Energy Dispersive X-ray analysis (EDS). The thin sections were analyzed using petrographic microscope (under plane, cross-polarized light and with gypsum plate). The petrographic analysis of aggregates was conducted to determine the presence of potentially reactive minerals. During the microstructural analysis particular emphasis was placed on establishing exactly the type of the reaction and the distribution and the composition of the resulting gel. In addition, the Damage Rating Index method has been used to ascertain the degree of concrete pavement damage caused by ASR.

During the microstructural analysis particular emphasis was placed on establishing exactly the type of the reaction and the distribution and the composition of the resulting gel. The results of the analysis revealed the presence of the deleterious reactions in pavements. Concrete showed evidence of the ASR. The reactive components were identified as schist and sandstone in the coarse aggregate and reactive siliceous fine aggregate. The resulting gel was predominantly of Si-Ca-K-Na composition.

Keywords: aggregate; alkali-silica reaction (ASR); gel; petrographic analysis; reactive minerals; SEM analysis

1. INTRODUCTION

Damage of concrete related to alkali-silica reaction (ASR) usually shows up after several dozen years. However, if the aggregate is highly reactive and the conditions are favourable, damage may occur within a few years [1][2]. The data collected during the in-situ visual examination of the damaged concrete structures as well as data obtained during microstructural characterization performed in the laboratory showed that problem of alkali-silica reaction is still valid and not fully recognized. Many examples of concrete damage caused by ASR from all over the world can be found in the literature. Fernandes *et al.* [3] analysed ASR products in 50-years-old concrete dam from Portugal. Custódio and Ribeiro [4] tested cores from a motorway underpass in Portugal built in early 2000's and they showed that swelling and cracking were caused mainly by ASR. Frýbort *et al.* [5] focused on composition of ASR gel in 18-years old highway concrete from Czech Republic. ASR products were also found in concrete pavements in Argentina [6].

In this paper the forensic investigations have been performed on core specimens extracted from expressway pavement structures situated in the northern part of Germany, constructed near the year 1996. After 15 years of exploitation, the concrete pavement showed signs of significant damage, Figure 1.1. Many cracks, especially D-cracking and along the joins were observed. The development of the alkali-silica reaction was suspected to be the principal cause of concrete deterioration. In spite of heavy

traffic and frequent application of deicing salts during winter season the observed damage could be also attributed to the combined effects of physical interactions of deicers with the concrete matrix and chemical reactions. The deterioration of concrete induced by pavement deicers has been thoroughly investigated, [7][8][9]. These researches demonstrated that exposure to chloride deicers could accelerate ASR in aggregates in pavement concrete.

In this study the microstructural evaluation of the damages of real concrete structure has been conducted. The research was focused on detailed microscopic observations including optical and scanning electron microscopy. These core specimens have been selected due to severe degree of distress observed. In all cases, the NaCl has been used as a deicer.



Figure 1.1: Examples of the deteriorated areas of pavement from the expressway

2. MATERIALS AND LABORATORY TEST METHODS

2.1 Materials

The damaged concrete pavement was located at the north of Germany. During the selection of the precise location of the cores, an attempt was made to collect the specimens from regions of the pavements representing varying degrees of distress.

Figure 2.1 shows an example of cores of expressway pavements with visible high levels of deterioration. Cores were extracted for the polished sections, thin sections and SEM-EDS analysis. The diameter of all cores was 300 mm. Additionally, in Figure 2.1c, a cross-section of the specimen with visible white gel filling cracks and air-voids is shown.



Figure 2.1: a, b) Photographs of a core from concrete pavement, c) lapped surface of Core 9 from concrete expressway showing a crack radiating from a rimmed, dark schist particle into adjacent paste. The crack is filled with white gel

2.2 Testing methods

2.2.1 Petrography

Petrographic analysis was performed using Olympus BX51 polarizing microscope with digital colour camera and automatic moving table Prior ES11BX/B. Thin sections with thickness of $20\pm 2\mu m$ were prepared. Observations were performed in petrographic microscope under plane (PPL) and cross-polarized light (XPL) at magnifications of 40 to 400X.

2.2.2 DRI

Each of the cores was cut vertically into two parts. After cutting, each longitudinal section was cleaned thoroughly with paper towels to remove remains from the surface. The specimens were dried at 50°C for 24 h. The estimation of the degree of concrete pavement degradation was performed using Damage Rating Index method proposed by Rivard *et al.* [10]. The assessment is based on summing up points of structural factors related to ASR. Individual factors have different weights, as is presented in Table 2.1.

Defect type	Factor	
Coarse aggregate with crack	0.25	
Coarse aggregate with crack and gel	2	
Coarse aggregate debonded	3	
Reaction rim around aggregate	0.5	
Cement paste with crack	2	
Cement paste with crack and gel	4	
Air void lined with gel	0.5	

Table 2.1: Structural factors related to ASR	[10]
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The specimens were cut into about 10 x 10 cm squares and polished on SiC powders (180, 320, 600, 1000). A grid composed of 1.5 cm squares was drawn on the prepared surface of the specimen [10].



Figure 2.2: Stereoscopic microscope with analyzed polished concrete section 10 x 10 cm with grid (1,5 x 1,5 cm) on an automatic table

On each square the number of defects occurring was counted using a stereoscopic microscope at 30x magnification. Summed values after taking into account the weights were normalized to 100 cm², giving the DRI number. The higher the DRI value, the higher ASR induced concrete degradation rate. In Figure 2.2 the stereoscopic microscope with polished specimen for DRI measurements is presented. The analysis was performed on 3 specimens.

2.2.3 SEM/EDS

Identification of alkali-silica gel was performed on lapped and polished specimens with an area of 45x30 mm, cut from concrete cores. The specimens were sputtered with carbon layer. A strip of conductive tape was then attached to each polished specimen. The microstructure of concrete was studied using Scanning Electron Microscope (SEM) with Energy Dispersive X-Ray Analysis (EDS). Specimens were tested by aid of a Zeiss sigma VP microscope, in the backscatter mode using an acceleration voltage of 20 kV.

3. RESULTS AND DISCUSSION

By petrographic investigations the type of aggregates used for concrete pavement were identified. The respective results are presented in Figure 3.1 - 3.3. As can be seen quartz sand was used as a fine aggregate, mainly it is monomineral quartz. But, the sand also contains reactive species of quartz (Figure 3.1). These reactive micro and cryptocrystalline quartz as well as strained quartz have caused an alkaline reaction in the concrete. Coarse aggregate was identified as schist and sandstone (Figure 3.2-3.3).



Figure 3.1: Cross sections of the fine aggregates (thin section, XPL, scale bar = 0.2 mm), 1 - microcrystalline quartz, 2 - micro- and cryptocrystalline quartz, 3 - monomineral quartz, 4 - strained quartz



Figure 3.2: Cross sections of the coarse aggregate grain recognised as a schist (thin section, scale bar = 0.5 mm, PPL (a) and XPL (b))



Figure 3.3: Cross sections of the coarse aggregate recognised as a sandstone (thin section, scale bar = 0.2 mm, PPL (a) and XPL (b))

Results of Damage Rating Index calculation are presented in Table 3.1. The average value form three specimens of DRI is 461. Very high value of DRI proves that concrete pavement was damaged mainly by ASR. For comparison, Rivard *et al.* [10] obtained DRI below 300 for reactive aggregates and below 100 for non-reactive aggregates. In [11] authors reported DRI over 400 for the most damaged specimen.

The largest influence on the high value of DRI had the number of pores filled with ASR gel and cracks in the cement matrix.

Specimen	Coarse aggregate with crack	Coarse aggregate with crack and gel	Coarse aggregate debonded	Reaction rim around aggregate	Cement paste with crack	Cement paste with crack and gel	Air void lined with gel	DRI
1	8.1	44.4	62.2	3.7	124.4	17.8	226.7	487
2	10.4	37.0	69.6	5.9	103.7	13.3	251.9	492
3	5.9	22.2	57.8	3.0	91.9	11.9	211.9	404
Average	8.1	34.6	63.2	4.2	106.7	14.3	230.1	461

Table 3.1: Detailed results of petrographic examination for Damage Rating Index calculations performed on the concrete cores

Alkali-silica reaction was observed in all of the cores as was cracking due to the reaction. The cracked concretes also appear to contain non-frost resistant air-void systems, respective air content, such that, some of the cracking is likely due to damage by freezing and thawing while critically saturated.

In the SEM analysis of concrete, cracks in the aggregate and in the cement matrix were observed. Most of the microcracks were localized parallel with the surface (what may suggest the freezing-thowing damage) and most of them were propagating through the matrix. Often, the part of the matrix crack extends towards the aggregate-paste interface causing debonding, Figure 3.4. On occasion the smaller cracks were filled with ettringite deposits' however the widest cracks in the matrix were typically empty, Figure 3.4. The cracks in the matrix didn't contain any traces of alkali-silica gel, but the cracks propagated through the aggregate were filled with ASR gel. The cracks located in the aggregate contained the Si-Ca-K-Na gel, Figure 3.5.

Ettringite deposits have been found in the cracks and very often in the air-voids, except in the carbonated very top layer.



Figure 3.4: Scanning electron micrograph of the air void filled with ASR gel (1) and ettringite (2)

The paste in the top face of the core was carbonated. CSH of the matrix didn't seem to contain deicer ions. Only very small amount of chloride ions have been found in the matrix, Figure 3.6. Much lower content of sodium ions compared to potassium ions was found in alkali-silica gel, Figure 3.4 and 3.5, what may suggest no influence of NaCl deicer. A very few signs of Friedel's salt has been found in the matrix, which occurred with ettringite, Figure 3.6.



Figure 3.5: Scanning electron micrograph of the cracked aggregate grain filled with Si-Ca-K-Na gel



Figure 3.6: Friedel's salt and ettringite signs in the matrix

Almost whole reacted grains of aggregate were also found (Figure 3.7-3.9). The quartz fine aggregate was converted into Si-Ca-K-Na gel. EDS analysis showed the presence of residues of pure quartz. Additionaly area of Figure 3.8 was analysed using EDS mapping. It is clearly visible whole grain consisting of ASR gel (stronger indication of K and Na) and point indications from pure quartz (Si). The analysis of sulfur (S) distribution shows the presence of ettringite in the air voids and cement matrix.



Figure 3.7: Scanning electron micrograph of the reacted aggregate grain almost completely converted into Si-Ca-K-Na gel, with remnants of quartz



Figure 3.8: Scanning electron micrograph of the reacted aggregate grain almost completely converted into Si-Ca-K-Na gel, with remnants of quartz

Marfil and Maiza [6] reported ASR induced damages in concrete from three different pavements in Argentina (3, 15, 18 years old). In all cases they found products of alkali-silica reaction inside the air voids and at the interface between aggregate and cement matrix. The reactive components were recognized as strained and microcrystalline quartz as well as volcanic glass. An ettringite situated in the air voids was also identified, but its impact on the destruction of concrete structures was not found. Kurdowski *et al.* [12] analysed the microstructure of the concrete highway pavement, constructed and open for exploitation near in 1934 in east of Germany at that time, now in the south-west of Poland. The construction was in excellent condition until 2008, when it was demounted. A part of siliceous quartz sand grains quartz, single feldspar grains and larger (up to 8 mm) rock fragments, chiefly sedimentary less frequently metamorphic and magmatic were found as fine aggregate. As a coarse aggregate, a basalt has been identified. The Authors conclude that the cement composition and low w/c ratio which assured a very good bond of cement matrix with aggregate grains, very compact interfacial transition zone and basalt aggregate affected the durability of the concrete pavement.



Figure 3.9: EDS mapping of the reacted aggregate grain (almost completely converted into a ASR gel)

In the conducted research opposite results were obtained regarding the microstructure analysis of the German concrete highway pavement. The differences result primarily from a different concrete composition but also from environmental conditions. Analyzed concrete specimens came from the A1

highway from northern Germany, close to the Baltic Sea, in contrast to specimens analyzed by Kurdowski *et al.* [12], which were taken from concrete pavement from the south once Germany.

4. CONCLUSIONS

Based on the results of studies on polished and thin sections and SEM specimens along with the microstructure analysis the following principal conclusions can be drawn:

- Based on the petrographic examination coarse aggregates used for concrete pavement were recognised as a sandstone and schist. The fine aggregate was a siliceous sand.
- Reactive form of quartz (micro- and cryptocrystalline quartz, strained quartz) was identified in the aggregate.
- The level of concrete pavement damage caused by ASR was quantified using Damage Rating Index calculations and was estimated at 461 which suggests significant influence of alkaline reaction in pavement destruction.
- The degradation of concrete structure is due to the alkali-silica reaction products which are present in the concrete cores.
- Numerous traces of ASR have been found in the received cores from the concrete pavement: cracks in the cement paste and aggregate grains, cracks filled with gel, air voids filled with gel.
- SEM/EDS measurements confirmed the presence of the K-Na-Si-Ca gel in the cracks and air voids.
- The composition of the gel consisted of Si, Ca and usually K, but also the Na was present.
- The part of cracks which were going through the matrix was found debonding the aggregates.
- Most air voids were filled (either partially or fully) with ettringite deposits.
- The NaCl deicer seems to not influence the concrete degradation by inducing the ASR distress. Only the small precipitation of the chloride ions was found in the gel in all specimens.

REFERENCES

- [1] Glinicki MA, Jóźwiak-Niedźwiedzka D, Antolik A, Dziedzic K, Gibas K (2019) Susceptibility of selected aggregates from sedimentary rocks to alkali-aggregate reaction. Roads and Bridges Drogi i Mosty 18:5–24. https://doi.org/10.7409/rabdim.019.001
- [2] Rajabipour F, Giannini E, Dunant C, Ideker JH, Thomas MDA (2015) Alkali silica reaction: Current understanding of the reaction mechanisms and the knowledge gaps. Cem Concr Res 76:130–146. https://doi.org/10.1016/j.cemconres.2015.05.024
- [3] Fernandes I, Noronha F, Teles M (2004) Microscopic analysis of alkali-aggregate reaction products in a 50-year-old concrete. Mater Charact 53:295–306. https://doi.org/10.1016/j.matchar.2004.08.005
- [4] Custódio J, Ribeiro AB (2019) Evaluation of damage in concrete from structures affected by internal swelling reactions - A case study. Procedia Struct Integr 17:80–89. https://doi.org/10.1016/j.prostr.2019.08.012
- [5] Frýbort A, Všianský D, Štulířová J, Stryk J, Gregerová M (2018) Variations in the composition and relations between alkali-silica gels and calcium silicate hydrates in highway concrete. Mater Charact 137:91–108. https://doi.org/10.1016/j.matchar.2018.01.012
- [6] Marfil SA, Maiza PJ (2001) Deteriorated pavements due to the alkali-silica reaction: A petrographic study of three cases in Argentina. Cem Concr Res 31:1017–1021. https://doi.org/10.1016/S0008-8846(01)00508-7
- [7] Sutter L, Dam T Van, Peterson KR, Johnston DP (2006) Long-Term Effects of Magnesium Chloride and Other Concentrated Salt Solutions on Pavement and Structural Phase I Results. Transp Res Recor 60–68

- [8] Wang K, Nelsen DE, Nixon WA (2006) Damaging effects of deicing chemicals on concrete materials. Cem Concr Compos 28:173–188. https://doi.org/10.1016/j.cemconcomp.2005.07.006
- [9] Jain J, Olek J, Janusz A, Jóźwiak-Niedźwiedzka D (2012) Effects of Deicing Salt Solutions on Physical Properties of Pavement Concretes. Transportation Research Record 69–75. https://doi.org/10.3141/2290-09
- [10] Rivard P, Fournier B, Gerard B (2002) The Damage Rating Index Method for ASR Affected Concrete - A Critical Review of Petrographic Features of Deterioration and Evaluation Criteria. Cem Concr Aggregates 24:1–11
- [11] Rivard P, Ballivy G (2005) Assessment of the expansion related to alkali-silica reaction by the Damage Rating Index method. Constr Build Mater 19:83–90. https://doi.org/10.1016/j.conbuildmat.2004.06.001
- [12] Kurdowski W, Witek A, Śliwiński J (2016) Concrete pavement on highway after 70 years of exploitation, the assessment of durability reason. Cement Wapno Beton 1:27–35