

EFFECT OF ALKALI INGRESS AND BINDER COMPOSITION ON DAMAGING ASR

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Abstract

The effects of cement replacement by fly ash and the effect of alkali content of Portland cement and fly ash on damaging ASR in concrete due to ingress of deicing salt has been investigated using greywacke as reactive aggregate. Concrete specimens were subjected to cyclic storage scheme using moist and dry conditions as well as temperatures of 20 and 60°C and exposure to 10 wt.% NaCl solution. Exposure to salt resulted in a high degree of expansion and damage. Cement replacement by 30 wt.% fly ash reduced the damage by as much as a factor of 10. Investigations of pore structure, phase and pore solution composition indicate that, besides the densification of the pore structure due to fly ash, ASR due to external alkalis is affected by the alkali content of both cement and fly ash and the aluminium content of fly ash.

Keywords: avoiding ASR, alkali ingress, fly ash, pore solution, aluminium

1 INTRODUCTION

The recent observation of ASR damage to concrete pavement exposed to large quantities of deicing salt has reopened the discussion on ASR in Germany. Investigations on the effect of alkali ingress from deicing salt on ASR confirm the damaging effect of external alkalis on concrete [1-5]. It is well-known that cement replacement by fly ash reduces damaging ASR [6-8]. The present investigations focus on the effect of cement and fly ash alkali content and cement replacement by fly ash on ASR due to external alkalis. As well as using low alkali cement, different amounts of low and high alkali cement were replaced by fly ash in concretes produced with greywacke as reactive aggregate. The specimens were subjected to a cyclic storage scheme [9] which includes exposure to a NaCl solution. As well as monitoring expansion, pore solutions were expressed for chemical analysis, mercury intrusion porosimetry was used to investigate changes in pore structure and thin sections were investigated using optical microscopy.

2 MATERIALS AND METHODS

2.1 Materials and mixture proportions

Two Portland cements CEM I 32.5 R were used in the investigations representing high (HA) and low (LA) alkali equivalents, i.e. 1.02 and 0.56 wt.% $\text{Na}_2\text{O}_{\text{eq}}$, respectively. Up to 30 wt.% of the cement in the concretes was replaced by two hard coal fly ashes with different alkali contents (F1: $\text{Na}_2\text{O}_{\text{eq}}=3.6$ wt.%, F2: $\text{Na}_2\text{O}_{\text{eq}}=1.1$ wt.%). Precambrian greywacke graded at A/B 16 mm according to DIN 1045-2 [10] was used as reactive aggregate together with an inert quartz sand for the 0/2 mm fraction. The chemical composition and physical properties of the concrete constituents are listed in Table 1. The concretes were produced with 400 kg/m³ binder (cement and fly ash) at a w/b ratio of 0.45. Prisms measuring 75×75×280 mm³ were

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produced with gauges on the end faces. In addition, part of each concrete mix was poured into 500 ml polyethylene flasks for pore solution extraction at a later date.

2.2 Methods for assessment and analysis

General

The susceptibility of the concretes to ASR caused by external alkalis was tested using a modified 60°C test [9] with cyclic storage including exposure to a 10 wt.% NaCl solution, Table 2. For comparison, two concrete compositions were selected for non-cyclic storage in a performance test without NaCl. In this case, the initial storage period of 91 d comprised 1 d in the mould followed by 90 d moist storage over water (100 %RH at 20°C). This was followed by storage for 140 d over water at 60°C and one day at 20°C/ 100% RH before each measurement, [11]. Length changes of the prisms were measured at 20°C after initial storage and every 14 d. The specimens were assessed using an expansion criterion of 0.5 mm/m for the cyclic storage with NaCl and 0.2 mm/m for the performance test without NaCl, see [12]. The criteria apply for a storage period of 10 cycles or 140 d after initial storage.

Mercury intrusion porosimetry

Pore size distributions were determined with mercury intrusion porosimetry using concrete fragments (20 mm) removed from specimens at an age of 91 d after initial storage.

Pore solution and analysis

Pore solutions were expressed from concrete specimens (\varnothing 70 mm, 100 mm) removed from the flasks after the same initial storage (91 d) as the concrete prisms. In addition, pore solutions were extracted from concrete prisms after 10 storage cycles (age: 231 d). The chemical composition of the pore solution was analysed using ICP-OES and the hydroxyl ion concentration determined by titration. Good agreement was obtained between solution extracted from prisms and flask specimens after 91 d initial storage.

X-ray powder diffraction (XRD)

For the X-ray investigations a slice was sawn from each prism, crushed with a hammer and ground for 1 s in a vibration disc mill. The binder content was enriched by removing aggregate with a 63 μ m sieve. The powder was ground for 5 minutes in isopropyl alcohol in a laboratory ball mill and then dried at 40°C. For XRD quantitative phase analysis using CuK_α radiation and the Rietveld refinement, the samples were mixed with 20 wt% ZnO as an internal standard and stored in an argon atmosphere until measurement. This permitted the estimation of the amount of non-crystalline phases by the Rietveld fitting procedure. The XRD measurements were performed with a XRD 3003 TT diffractometer of GE Sensing & Inspection Technologies GmbH with θ - θ configuration und CuK_α radiation ($\lambda=1.54 \text{ \AA}$). The angular range was from 5 to 70 ° 2 Theta with a step width of 0.02 ° and a measuring time of 6 sec/step.

Microscopy

After 10 storage cycles, 40×40 mm² slices, 20 mm in thickness, were dry-sawn from the centre of the prisms and dried in an oven at 60°C. The slices possessed two external surfaces exposed to salt. The slices were vacuum-impregnated a number of times with fluorescent synthetic resin. Diamond polished thin sections (20 μ m) were prepared from the slices and examined optically using an Olympus BX 61.

3 RESULTS

Expansion of concrete

Figure 1 shows the expansion of the concrete specimens beginning at the end of the initial storage (91 d). After 10 cycles, the expansion of specimens made with 100% cement HA, Figure 1 (left), exceeds at 3.34 mm/m the 0.5 mm/m criterion. The 0.2 mm/m criterion for the performance test is also exceeded (0.31 mm/m) with this concrete. Replacement of this cement by ash reduces the 140 d expansion considerably, in particular in the case of the fly ash (F2) with a lower alkali content where the expansion was, at 0.31 mm/m, below the 0.5 mm/m criterion. The corresponding results for cement LA are shown in Figure 1 (right) where the expansion of concrete made with 100% cement is less (1.58 mm/m) than with cement HA, but still well above the expansion criterion. However, the performance test yields with cement LA an expansion of 0.1 mm/m which is below the 0.2 mm/m criterion. With the LA cement, 20% fly ash F1 was sufficient to marginally fulfill the 0.5 mm/m criterion for cyclic storage (0.55 mm/m). The expansion of 0.15 mm/m after 10 cycles with fly ash F2 is insignificant.

Porosity of concrete

The pores structure of concrete affects the ingress of external alkalis decisively. Table 3 shows the percentage of total porosity in pores above and below 30 nm for the different concretes. If it is assumed that pores larger than 30 nm contribute to capillary transport, see [13, 14], capillary porosity and therefore the ingress of external alkalis in concrete is reduced significantly for both cements on replacement by fly ash.

Pore solution composition

Table 4 and Figure 2 compares the chemical composition of pore solutions expressed from concretes made with high alkali cement (with 30 wt.% and without fly ash) and low alkali cement. The results are mean values over specimen volume where, in particular, the salt solution did not fully penetrate the specimens made with fly ash. The pH of the pore solution in concretes made without fly ash decreases from 13.57 or 13.31 after initial storage, for cement HA and LA, respectively, to 12.81 at the end of cyclic storage. At the same time the concentration of aluminium is also reduced. Opposed to this, the sulphate concentration of the pore solution increases significantly; from 63 to 136 mmol/L and from 6 to 112 mmol/L for cements HA and LA, respectively. Replacement of cement by fly ash has a significant effect on pore solution composition before and after cyclic storage. At first the pH is lower and the pore solution contains more aluminium. After cyclic storage little aluminium remains in solution, but the pore solution of fly ash concrete contains more sulphate. During cyclic storage of the fly ash concrete (30% F1, Table 4), the concentration of potassium almost doubles (238 to 444 mmol/L). This indicates that potassium is released into the pore solution from the fly ash (see Table 1) in the presence of dissolved NaCl.

X-ray powder diffraction (XRD)

Ettringite was no longer present after 10 cycles, but Friedel's salt and halite were present in the (dried) powders (100 %HA and HA 30 % F1).

Microscopy

The optical investigations revealed a high degree of damage to prisms made with 100% cement HA following exposure to NaCl solution. Numerous, strongly branching cracks with widths between 40 and 50 μm were present over the whole specimen cross section. The cracks started at greywacke aggregate particles then running through the matrix. Figure 3 (right) shows a crack filled with ASR gel which starts at a greywacke particle. The surface of a nearby pore is coated with gel. The manifestation of damage is similar for

specimens made with 100% cement LA in which numerous cracks (10 to 40 μm) and gel in pores and cracks are apparent. Again, the cracks start at the aggregate, Figure 4 (right). Replacement of cement by fly ash reduces the degree of damage considerably. Although the specimen HA 30% F1 contains a small number very fine cracks, they do not contain ASR gel. Ettringite was identified in a pore in Figure 4 (left), but not by XRD. Pores containing gel were not apparent. The concrete made with 100 % HA and subjected to the performance test without NaCl exhibited a small amount of damage with matrix cracks (5 to 10 μm) starting at aggregate particles. Gel was identified in pores and on cracks sides, Figure 3 (left).

4 DISCUSSION

As also observed elsewhere [4, 5], the exposure of concrete specimens with reactive aggregate to NaCl solution leads to considerable expansion, Figure 1. The results show that the expansion is mainly due to ASR rather than salt crystallization. Salt crystals were not observed on the (dry) concrete surfaces and, for example, the expansion of concrete made with 100% cement (LA and HA) is very different although a similar NaCl uptake is expected. The expansion due to NaCl ingress expansion is significantly smaller if the cement used is partially replaced by fly ash. Although after 10 cycles the expansion of the concrete HA 30% F1 is slightly above the limiting criterion, ASR gel is not present. However, fine cracks are apparent in the thin section micrographs. Assuming that the alkalis are just supplied by the cement, the alkali content of concrete made 100% cement HA is 4.1 kg $\text{Na}_2\text{O}_{\text{eq}}/\text{m}^3$ which is reduced to F1 2.9 kg $\text{Na}_2\text{O}_{\text{eq}}/\text{m}^3$ for 30% replacement with F1. However, although the alkali content of concrete with 100% cement LA is even lower (2.2 kg $\text{Na}_2\text{O}_{\text{eq}}/\text{m}^3$) it expands more than concrete made with cement HA and 30% fly ash. Moreover, at the same replacement level (30%) of cement HA, less expansion occurs with the lower alkali content (F2 as opposed to F1). This indicates that the alkali content of both the cement and fly ash affects the expansion produced by exposure to external alkalis. Schmidt [7] also observed a reduction in the final expansion of concrete specimens in a fog chamber at 40°C on using fly ash with lower alkali content. It is clear that the use of low alkali cement alone does not suffice to prevent ASR damage when concrete is subjected to external alkalis, as also observed by Stark et al. [5]. Thus the reduction in alkali content of concrete obtained by using low-alkali cement is not enough to offset the large amount of active alkalis (i.e. alkalis leading to damage) supplied by the NaCl solution.

Another aspect of ASR is the denseness of the concrete microstructure. The pozzolanic reaction of the fly ash results in a large reduction in capillary porosity [14], as is apparent in Table 3, and therefore a corresponding reduction in the capillary uptake of external alkalis. In the case of concrete made with 100% cement HA, salt completely penetrated the prisms (measured by spraying AgNO_3 as Cl indicator on dry-cut surfaces) after 10 cycles so that 100% of the reactive aggregate was in contact with NaCl. With fly ash, however, only 60 to 70% of the prism cross section contained NaCl after 10 cycles, i.e. 30 to 40% of the reactive aggregate was not exposed to NaCl. The penetration depth of NaCl decreased with increasing fly ash content. However, for a particular replacement level, different expansions occurred. This can only be explained by the different $\text{Na}_2\text{O}_{\text{eq}}$ of the fly ashes because the same cement was used. This argument also applies to the concretes made from cements HA and LA without fly ash; the expansion was different although NaCl completely penetrated all the specimens.

Differences in pore structure are also mirrored in the composition of the pore solution, bearing in mind that the values presented are averaged over specimen volume, i.e. it is not possible to distinguish between high near-surface concentrations and lower concentrations distributed over concrete depth. As can be seen in Table 4, exposure to NaCl leads to large amounts of Na and Cl in the pore solution. In the case of concretes made with 100% cement, the concentrations of Na and Cl in the pore solution accumulate during the cycles to values above the initial concentration of the 10 wt.% NaCl storage solution, i.e. 1900 mmol/L.

This effect is less pronounced for concretes with fly ash, Table 4. Sufficient alkalis are therefore available for the ASR reaction irrespective of binder composition or use of a low alkali cement. Nevertheless, different expansion and degrees of damage were observed for the concretes investigated.

It is well-known that penetrating chloride results in the formation of Friedel's salt from ettringite and calcium aluminate hydrate phases during which chloride is bound by sulpho aluminate phases and sulphate released into the pore solution. This accounts for the observed increase in sulfate concentration in Table 4 and Figure 2. If calcium aluminate hydrate dissolution in the presence of dissolved NaCl produces Friedel's salt an increase in pH of the pore solution is to be expected. In contrast, leaching and conversion of ettringite to Friedel's salt lower the pH of the pore solution, cf. Stark et al. [5]. Based on the present data, it is not possible to estimate the individual contributions to pH. According to [15], damaging ASR should not take place at the OH⁻ concentrations at the end of cyclic storage, i.e. between 60 and 80 mmol/L because the solubility of SiO₂ is relatively low. Nevertheless, ongoing ASR damage occurs despite the low pH the pore solution. It is therefore assumed that the solubility of SiO₂ in the pore solution at low pH is affected by dissolved NaCl. Calculations with the hydrogeochemical model PHREEQC [16] indicate that SiO₂ solubility may be enhanced by the formation of an aqueous complex NaHSiO₃⁰. Dove and Elston [17] observed an increase in the dissolution rate of quartz at higher NaCl concentrations. It is concluded that damaging ASR can still occur in concrete despite a significant reduction in pH.

Replacement of cement by fly ash reduces the amount of NaCl in the pore solution which is obviously due to the denser pore structure produced by fly ash. However, according to [18], C-S-H in fly concrete possesses a low C/S ratio and contains more aluminium. These C-S-H phases are able to bond more alkalis [18-20] thus reducing the alkali content of the pore solution. But in view of the high concentration of sodium in the pore solution, alkali bonding by C-S-H should not affect the ASR reaction. Nevertheless, expansion clearly depends on the alkali content of the fly ash. In Table 4, the concentration of aluminium in the pore solution of fly ash concrete after initial storage (91 d) is considerably higher than for concrete made without fly ash and therefore more aluminium is consumed during the subsequent cyclic storage. It is also possible that additional aluminium is supplied by the fly ash as hydration progresses. Aluminium is available for the formation of aluminosilicate complexes [21-23] in which the bound SiO₂ does not take part in the gel formation process. Moreover, aluminosilicate formation on the surface of the aggregate particles could also inhibit the dissolution of SiO₂ [24]. Thus aluminium controls the release of SiO₂ from the aggregate for ASR [22].

5 CONCLUSIONS

The effect of the alkali content of Portland cement and fly ash as well as cement replacement by fly ash on ASR in concrete due to ingress of deicing salt has been investigated using greywacke as reactive aggregate. Concrete specimens were subjected to a dry/moist cyclic storage scheme with exposure to NaCl solution during which expansion was monitored. Pore size distributions, pore solution compositions and phases were analysed. Section microscopy was used to observe damaging ASR. The following conclusions are drawn for damaging ASR in concrete exposed to external alkalis in deicing salt.

- Replacement of cement by fly ash reduces expansion and damage significantly.
- The use of low alkali cement does not prevent damaging ASR.
- Lower alkali contents of cement and fly ash reduce expansion.
- The denser pore structure produced by the pozzolanic reaction of fly ash affect the resistance of concrete to ASR favourably.

- Calculations indicate that the solubility of SiO₂ in the pore solution at lower pH is increased in the presence of dissolved NaCl owing to the formation of a complex NaHSiO₃⁰.
- Replacement of cement by fly ash increases the concentration of Al in the pore solution of concrete. The resulting aluminosilicates control the concentration of dissolved SiO₂ for the formation of ASR gel.

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Oxide [wt.%]	GW	HA	LA	F1	F2
	Greywacke	CEM I 32.5 R		Fly ash	
CaO	1.22	61.3	62.6	3.56	4.69
SiO ₂	68.7	18.7	22.1	47.8	46.2
Al ₂ O ₃	14.9	5.01	4.95	26.6	24.9
Fe ₂ O ₃	5.30	3.53	2.80	9.14	4.64
Na ₂ O	3.70	0.24	0.26	1.36	0.37
K ₂ O	1.76	1.20	0.46	3.41	1.10
Na ₂ O _{eq}	4.86	1.02	0.56	3.61	1.09
Fineness [wt.% > 45 μm]				21.4	18.1
Activity index 90 d [%] [25]				98	104
Glass content ¹ [wt.%]				80	68

¹ XRD with Rietveld refinement

Initial storage: total 91 d	1 d	in mould, 20 °C and 100 %RH
	69 d	20 °C and ~ 100 %RH
	14 d	20 °C and 65 %RH
	6 d	60 °C and ~ 100 %RH
	1 d	20 °C and ~ 100 %RH measurement
Cyclic storage: 10 cycles of 14 d total 140 d	5 d	60 °C (dry)
	2 d	20 °C in 10 wt.% NaCl soln.
	6 d	60 °C and ~ 100 % RH
	1 d	20 °C and ~ 100 %RH measurement

concrete	< 30 nm	> 30 nm
--	% of total porosity	
100 % HA	40.2	59.8
HA 20% F1	75.6	24.4
HA 30% F1	76.4	23.6
HA 20% F2	73.2	26.8
HA 30% F2	74.1	25.9
100% LA	46.5	53.5
LA 20% F1	60.9	39.1
LA 20% F2	72.4	27.6

concrete	storage	age	pH	OH ⁻	Na ⁺	K ⁺	Al	Cl	SO ₄ ²⁻
--	prism/flask	[d]	--	mmol/L					
100% HA	initial (f)	91	13.57	372	112	400	0.14	25	63
	cycle (p)	231	12.81	65	2657	329	0.02	2415	136
HA 30%F1	initial (f)	91	13.45	281	128	283	0.59	26	58
	cycle (p)	231	12.88	76	1843	444	0.07	1918	174
100% LA	initial (f)	91	13.31	205	112	104	0.10	2	6
	cycle (p)	231	12.81	64	2909	137	0.03	2567	112

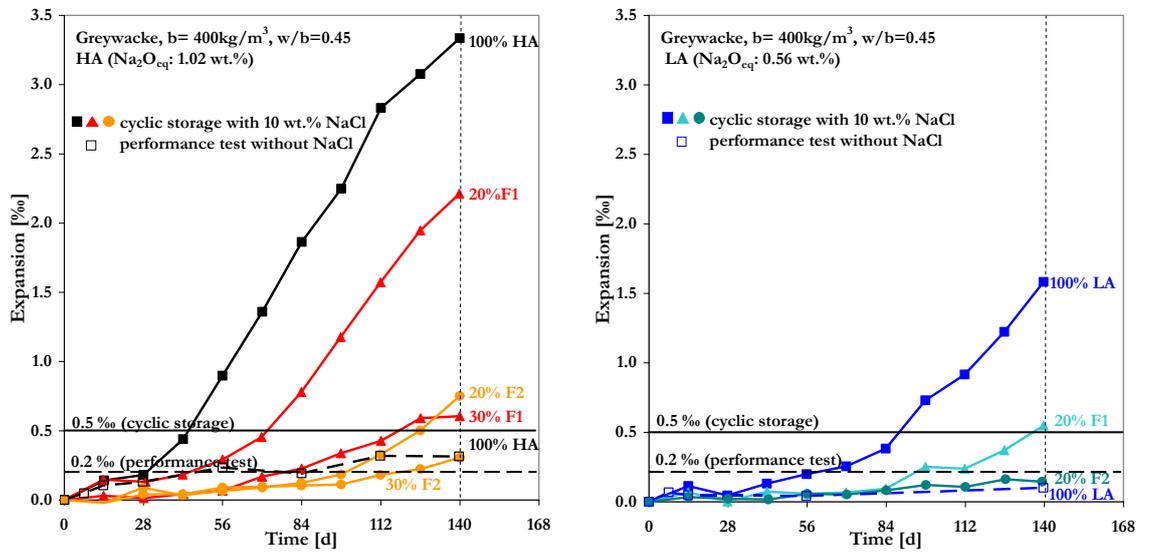


Figure 1: Expansion for concretes made with different binder compositions for high (left) and low (right) alkali cement

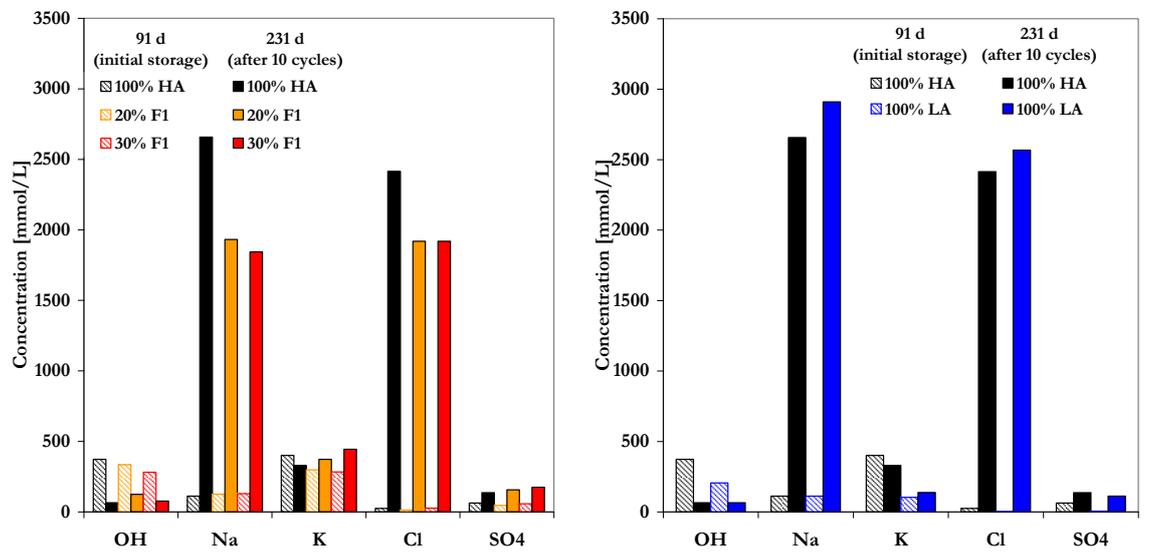


Figure 2: Expressed pore solutions after initial storage (flasks) and the cyclic storage (prisms) with 10 wt.% NaCl. Effect of replacement of HA cement by fly ash F1 (left) and effect of alkali content of cement (right)

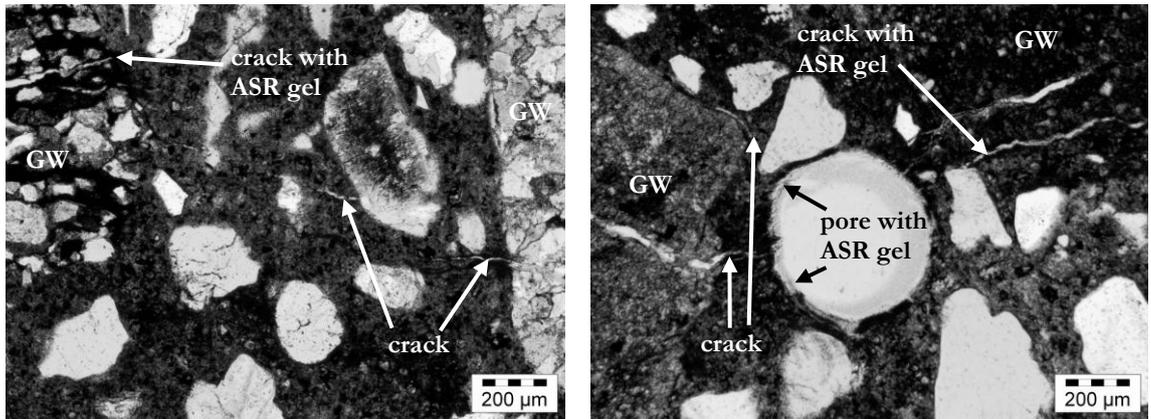


Figure 3: Microscopy at thin sections: 100% HA ($\text{Na}_2\text{O}_{\text{eq}} = 1.02 \text{ wt}\%$) performance test without NaCl (left) and cyclic storage with 10 wt.% NaCl (right)

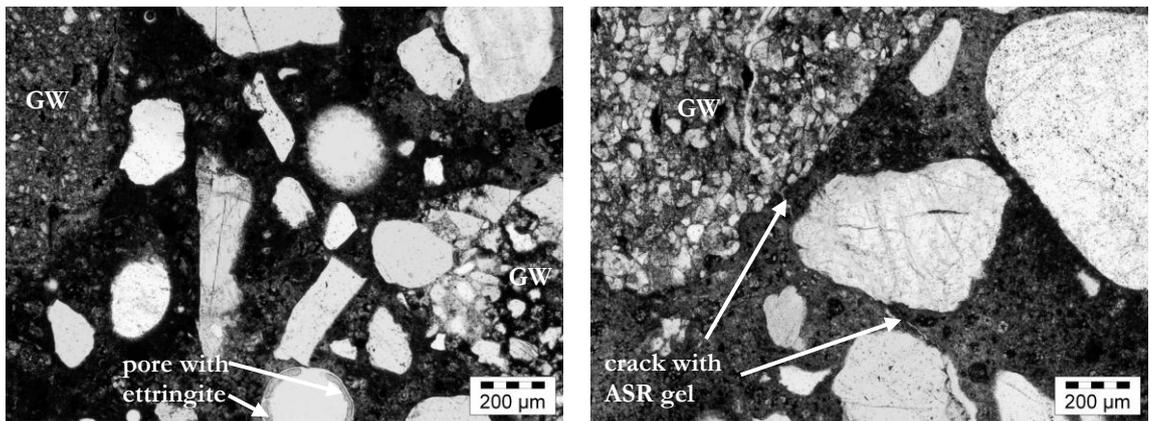


Figure 4: Microscopy at thin sections: HA 30 wt% F1 (left) and 100% LA with $\text{Na}_2\text{O}_{\text{eq}} = 0.56 \text{ wt}\%$ (right). Both with cyclic storage using 10 wt.% NaCl