

THE Na_2O -EQUIVALENT OF CEMENT: A UNIVERSAL PARAMETER TO ASSESS THE POTENTIAL ALKALI-AGGREGATE REACTIVITY OF CONCRETE?

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Abstract

In numerous national recommendations and standards cement content or Na_2O -equivalent of cement used for specific concrete are limited to a certain value in order to prevent deleterious alkali-aggregate-reaction (AAR). However, the Na_2O -equivalent may not always accurately define the potential of cement to cause AAR. In this study, the potential reactivity of concrete produced with cements of two different producers having a similar Na_2O -equivalent but different K/Na-ratios has been measured and gel composition has been analyzed. Additionally, pastes and mortars have been produced to study the development of hydroxide concentration in the pore solution.

The expansion of the concrete mixtures differs significantly. The composition of the gel in the two concrete mixtures studied displays differences in K/Na-ratio while hydroxide concentration in the pore solutions of pastes and mortars is identical. This indicates that the differences in K/Na-ratio have to be the reason for the observed differences in concrete expansion.

Keywords: concrete expansion, K/Na-ratio, gel composition, pore solution

1 INTRODUCTION

There are different ways in concrete production to prevent deleterious alkali-aggregate-reaction (AAR) in new structures. One is the use of non-reactive aggregates. However, a concrete producer often has no access to such aggregates either due to regional unavailability or financial reasons. The other one is to reduce the alkalinity of the pore solution in concrete to a level where no deleterious AAR occurs with the aggregate used. This can be achieved by the use of suitable mineral admixtures mitigating AAR [e.g. 1-5] or of cement with low Na_2O -equivalent [e.g. 6,7]. It has been shown repeatedly that the expansion in mortar or concrete produced with low alkali cement is lower in comparison to high alkali cement [e.g. 7-11]. Consequently, various national guidelines and standards take measures to reduce the content of soluble alkalis in concrete by limiting the Na_2O -equivalent of the cement or by limiting the cement content [e.g. 12-16]. However, the Na_2O -equivalent may not always be a reliable parameter defining the potential of cement to cause AAR. Firstly, the alkalis in cement can either be present as potassium and sodium sulfate or be bound in silicate and aluminate phases [17]. Depending on the amount present in the different mineral phases the solubility during the first stage of hydration is different; potassium and sodium sulfate being the more soluble compounds. Secondly, the ratio of potassium to sodium can vary in different cements with consequences for the total amount of K_2O and Na_2O present. Thirdly, the Na_2O -equivalent implies that the effect of potassium and sodium is equivalent. However, there are numerous studies indicating that the influence of potassium and sodium on silica and quartz dissolution is not identical [e.g. 18, 19].

Recently, experimental results obtained with cements of two international cement producers have given indications that differences in the K/Na-ratio of cements may have consequences for the resulting potential reactivity of concrete. In this study the potential reactivity of four concrete mixtures produced with cements of these producers having a similar Na_2O -equivalent but different K/Na-ratios has been measured in an accelerated concrete performance test. The composition of gel in two concrete mixtures has been analyzed after the tests using EDX analysis. Furthermore, cement pastes and mortars have been produced with two cements in order to analyze the hydroxide concentration in the pore solution and monitor its change with time.

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2 MATERIALS AND METHODS

2.1 Materials and mix design

Four different concrete mixtures were produced using four different cements from two international cement producers; two ordinary Portland cements (CEM I 42.5 N) and two Portland cements containing limestone powder (CEM II/A-LL 42.5 N / Tables 1). The cements of producer A and B have a similar Na₂O-equivalent but show differences in their K/Na-ratio. Rounded alluvial sand and gravel was used as aggregate (Table 2). The concrete mixture produced with CEM I 42.5 N of producer A has a slightly higher total of K₂O and Na₂O, in the concrete mixture produced with CEM II/A-LL 42.5 N it is the opposite (Table 3). The superplasticizer used is based on polycarboxylate ether (dosages given as liquid). Per mixture of concrete three prisms (7 x 7 x 28 cm) were produced.

For the production of mortar and paste only CEM I 42.5 N was used. The mortar mixtures were produced with the same relative amounts of cement, sand, water and superplasticizer as used in the concrete (Table 4). The water-cement-ratio (w/c) of the pastes was 0.31 and the dosage of superplasticizer in relation to the weight of the cement was identical to the concrete and mortar mixtures. Mortar M-A1 and M-B1 and paste P-A1 and P-B1 were filled in plastic bottles with a volume of about 0.7 l right after mixing. The bottles were closed with air-tight screw-tops and put in zip-lock bags. Afterwards, they were stored in an oven at 60 °C.

2.2 Methods for analysis

The petrography of the aggregates shown in Table 3 was determined according to Swiss standard SN 670'115 [20].

The potential reactivity of the aggregates was measured with the microbar test according to AFNOR P 18-588 using standard cement [21].

The potential alkali-aggregate reactivity of the concrete was measured according to AFNOR P 18-454 [22]. In this test three prisms (70.7 x 70.7 x 28.3 cm) are stored at 60 °C and 100 % relative humidity for 20 weeks. When the expansion after this period exceeds 0.02 % the concrete is classified as potentially reactive. No NaOH was added to the mixing water during concrete production in this project.

After conducting the concrete performance test one prism of concrete C-A1 and C-B1 were selected in order to analyze reaction products. Six samples (35 x 65 x 20 mm) were cut from each prism and dried for three days at 50 °C. After drying, they were stored in an exsiccator for one day, then impregnated with epoxy resin and polished. The analysis took place within one week after the polishing. In order to avoid an influence of local alkali contribution by feldspar or mica only quartzite aggregates were used to analyze the composition of the gel. For the investigation of the microstructure an environmental scanning electron microscope (ESEM-FEG XL30) was used. The carbon coated samples were studied in the high vacuum mode (2.0-6.0 10⁻⁶ Torr) with an accelerating voltage of 15 kV and a beam current of 272-275 µA. The chemical composition of the gel was analyzed with energy dispersive X-ray spectroscopy (EDX) using point spectra. An EDAX 194 UTW detector, a Philips digital controller and Genesis Spectrum Software (Version 4.6.1) with ZAF corrections were used. Approximately 100 point spectra of gel were made per concrete mixture.

Pore fluids of the pastes and mortars were extracted using the steel die method and pressures up to 530 N/mm². pH of the pore solution was measured with a glass electrode. Hydroxide concentrations were calculated based on pH and temperature measurements. Mortar pore solution was analyzed at an age of 2 hours, 1 day, 2, 4, 12 and 20 weeks. At an age older than four weeks, the pastes did not yield enough pore solution for an analysis.

3 RESULTS

3.1 Microbar test

The expansion of the tested grain size fractions both exceed the limit value of 0.11% and can therefore be classified as potentially reactive (Figure 1). The microbars produced with gravel exhibit higher expansion rates than the ones produced with sand.

3.2 Concrete performance test

Both concrete mixtures containing cement of producer B expand above the limit value of 0.02 % and can be classified as potentially reactive (Figure 2). Mixture C-B2 exceeds this limit after eight weeks and mixture C-B1 after 12 weeks. In contrast, mixtures C-A1 and C-A2 expand very little. All mixtures expand mainly during the first 8 weeks showing decreased expansion rates afterwards.

3.3 EDX analysis of gel

Concrete C-A1 and C-B1 both contain aggregates with gel-filled cracks (Figures 3 and 4). Crack and gel formations are mainly present in quartzite and gneiss. Sandstone shows no signs of reactivity and only a small part of siliceous limestone exhibits a few gel-filled cracks. The morphology of the gel varies. Sometimes it is structure-less and fills cracks entirely, sometimes it forms needle-like agglomerates crowing inward from the edges of cracks present in aggregates. In between, all combinations occur. In order to prevent an influence of alkali release by other silicates, only gel in quartzite was analyzed. It is mainly composed of Si, Ca, K and Na. Often traces of Mg, S and Al are present. The composition varies considerably. The K/Na-ratio is part of this variation as well. About 95% of all data lie between ratios 10:1 and 2:1. However, the average K/Na-ratio in concrete C-A1 is higher than in C-B1 (Figure 5). In a ternary Ca-K-Na diagram this difference can be seen as well (Figures 6 and 7). Furthermore, in concrete C-A1 some gel analysis show a higher Ca content than in concrete C-B1. A ternary Ca-Si-(K+Na) diagram reveals a trend in concrete C-A1 to a higher Ca but a lower Si content compared to concrete C-B1 (Figure 8 and 9).

In some aggregates gel of different generations is observed. Usually, the older or earlier gel is present in fragments in the central area of cracks (Figure 10). In general, the gel of the later generation forms at the edge of the cracks. Without exception the K/Na-ratio of these gels are different; the gel of the earlier generation exhibits higher values than the gel formed later (Figure 10).

3.4 Pore solution

Paste P-A1 and P-B1 show only a slight difference in hydroxide concentration (Figures 11 and 12). After two hours hydroxide concentration sharply increases and after 14 days it stabilizes at a value of about 0.5 mol/l. Alkalinity of mortar M-A1 and M-B1 is absolutely congruent. Like in the pastes there is a sharp increase in hydroxide concentration from two hours to one day. However, after 14 days there seems to be a trend to decreasing concentrations. The absolute values for the mortars are between 30 and 40 % lower compared to the ones of the pastes at the age of four weeks as the w/c used for the mortars is higher than the one for the pastes.

4 DISCUSSION

Although the differences in composition of the cements of producers A and B may seem minor the resulting potential reactivity of concrete mixtures is significantly different. The main difference in the chemical compositions of the cements is their Fe₂O₃, MgO and Na₂O content. However, only the difference of Na₂O should have an impact on the composition of the pore solution as both iron and magnesium have a low solubility. The cements of producer B have a slightly higher Na₂O-equivalent and a higher combined content of K₂O + Na₂O. This slight difference is reflected in the hydroxide concentration of the pore solutions in the pastes. It is not present in the pore solution of the mortars. A possible reason is the consumption of hydroxide ions at reaction sites.

There are some differences in the storage of mortar and concrete. While the mortar was stored in sealed containers the concrete prisms were able to take up water during the concrete performance test. Nevertheless, it can be assumed that these differences do not influence the comparability of the different cements used. Based on this assumption it is obvious that the alkalinity of the pore solution neither in paste or mortar can give an explanation for the substantial difference observed in concrete expansion. The chemical composition of the cements indicates that the only difference in the composition of the pore solution is the K/Na-ratio. This suggestion seems to be confirmed by the chemical analysis of the gel as analyzed in cracks of quartzite aggregates of concrete C-A1 and C-B1. Although there are large variations in K/Na-ratio and in the absolute concentration of these cations, the relative differences in the K/Na-ratio of the cements seem to be reflected in gel composition of the concrete mixtures.

The solubility of quartz depends strongly on pH and temperature. It is increased by the presence of cations; more notably in the presence of Na than K [19,23]. Experimental data showed a higher rate of dissolution of silica gel for potassium hydroxide than for sodium hydroxide though [24]. Little data exist on the combined effect of various ions [25] making statements about the influence of K/Na-ratio on the rate of dissolution in alkaline conditions difficult. No quantification was made about the degree of reaction in the two concrete mixtures studied microscopically. However, assessed qualitatively there seem to be slightly more dissolution phenomena and gel formations in concrete C-B1 than concrete C-A1. Therefore, the differences in concrete expansion can not only be the results of different degrees of dissolution but have to be substantially influenced by gel composition. These differences are not limited to K/Na-ratio but include calcium and silicon as well. Gel in concrete C-A1 contains slightly more calcium and less silicon than concrete C-B1.

The expansion mechanisms of alkali-aggregate reaction are still not clear [e.g. 26-30]. The cations present in the double layer of reactive minerals seem to determine the resulting expansive forces, but there is no consensus so far about the decisive parameters. The importance of calcium in the expansion mechanism is undisputed. In regard to the monovalent cations involved a combination of experimental results and calculations based on double layer theory indicate that potassium and sodium should be indifferent cations in regard to the resulting surface charge density and expansive pressure respectively [31,32]. On the other hand, expansion tests conducted on mortars in different alkali salt solutions indicate a greater expansion in the presence of potassium compared to sodium at a given pH and ionic strength [33, 34]. However, the significant differences in the expansion of concrete observed in the present study seem to be mainly caused by the lower K/Na-ratio both in pore solution and reaction products of mortar M-B1 and concretes C-B1 and C-B2.

The observation that the composition of early gel and gel formed at a later stage differ indicates that cationic composition of the pore solution changes with time. The results indicate that a higher fraction of potassium is present as easily soluble potassium sulfate, while a higher fraction of sodium is bound in relatively slow reacting clinker phases.

Expansion of the concrete prisms decelerates with increasing age. In the relatively short testing period of 20 weeks leaching is not a likely reason for the decelerating expansion. This could indicate that the alkalinity of the pore solutions has dropped below a critical level due to a consumption of hydroxide ions in reaction sites. This decrease as the likely reason for the simultaneous flattening out of the expansions curves is supported by the pore solution analysis of the mortars. Firstly, the concentrations of hydroxide ions in mortars produced with the different cements change congruently. Secondly, after the age of one day hydroxide concentrations decrease and lower the potential for quartz dissolution and ensuing expansion. However, the simultaneity of the process indicates that hydroxide ions in mortar and concrete are consumed at the same rate at reactions sites further supporting the difference in K/Na-ratio as the trigger for the resulting expansions.

The only obvious difference between the concrete mixtures is the K/Na-ratio of the cements used. If this is indeed the reason for the considerable difference in concrete expansion the use or limitation of the Na₂O-equivalent to assess or lower the potential reactivity of concrete by prescription of mix design can be misleading. In order to further investigate this problem expansion tests have to be combined with pore solution analysis. Furthermore, solubility experiments on quartz have to include various alkali and earth alkali ions in different proportions. More knowledge is needed about the relation between pore solution, gel composition and the mechanisms finally leading to the expansion in order to explain the phenomena observed in this study.

5 CONCLUSIONS

Concrete mixtures produced with cements having a similar Na₂O-equivalent but different K/Na-ratios can expand considerably different as an accelerated performance test to assess the potential reactivity of AAR shows. Consequently, the Na₂O-equivalent as a parameter to assess the potential reactivity of concrete mixtures might be misleading in certain cases.

The pore solution analysis of mortars shows that the differences in expansion are not caused by differences in the concentration of the hydroxide ions. However, the gel as analyzed in cracks of quartzite reflects the different K/Na-ratios of the cements indicating that the differences in expansion are due to the differences in the cationic composition of the pore solution.

In order to better understand the expansion mechanisms of AAR, the influence of alkaline solutions containing combinations of cations on the solubility of quartz has to be investigated. Additionally, the role of monovalent cations on AAR induced expansion has to be further investigated.

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TABLE 1: Cement composition in weight-%.

cement producer	A	B	A	B
cement type	CEM I 42.5 N	CEM I 42.5 N	CEM II/A-LL 42.5 N	CEM II/A-LL 42.5 N
SiO ₂	19.66	19.33	16.24	16.09
Al ₂ O ₃	4.79	4.87	3.84	4.19
Fe ₂ O ₃	3.07	2.34	3.02	2.18
CaO	62.87	62.35	61.20	61.49
MgO	1.82	2.73	1.53	3.62
SO ₃	3.30	3.16	3.00	3.06
K ₂ O	1.08	1.06	0.96	0.99
Na ₂ O	0.18	0.29	0.16	0.23
Mn ₂ O ₃	0.06	0.06	0.07	0.06
TiO ₂	0.27	0.23	0.23	0.21
P ₂ O ₅	0.14	0.13	0.09	0.12
Cr ₂ O ₃	0.01	0.01	0.01	0.01
SrO	0.10	0.56	0.11	0.54
Cl	0.02	0.02	0.02	0.04
Loss on ignition	2.63	2.86	9.52	7.17
SUM TOTAL	100.00	100.00	100.00	100.00
Na ₂ O-equ	0.894	0.986	0.798	0.881
K ₂ O + Na ₂ O	1.26	1.35	1.12	1.22
K/Na-ratio	6.71	4.09	6.71	4.82

TABLE 2: Petrography of the aggregates.

grain size	0-4 mm [weight-%]	8-16 mm [weight-%]
quartzite	30.3	19.8
gneiss	5.2	9.5
sandstone	40.9	28.8
siliceous limestone	5.9	3.6
limestone	17.1	36.6
porous rocks	0.6	1.7
SUM TOTAL	100.0	100.0

TABLE 3: Mix design of the concrete.

concrete mixture	C-A1	C-B1	C-A2	C-B2
sand 0-4 mm [kg/m ³]	761	790	778	778
gravel 4-8 mm [kg/m ³]	190	197	195	195
gravel 8-16 mm [kg/m ³]	476	493	486	486
gravel 16-32 mm [kg/m ³]	476	493	486	486
CEM I 42.5 N [kg/m ³]	375	340	-	-
CEM II/A-LL 42.5 N [kg/m ³]	-	-	350	350
superplasticizer [kg/m ³]	2.7	2.7	2.8	2.8
water [kg/m ³]	162	147	155	155
SUM TOTAL [kg/m ³]	2443	2463	2453	2453
w/c	0.43	0.43	0.46	0.46
Na ₂ O-equivalent [kg/m ³]	3.35	3.35	2.79	3.08
total K ₂ O + Na ₂ O [kg/m ³]	4.73	4.59	3.92	4.27

TABLE 4: Mix design of the mortar.

mortar mixture	M-A1	M-B1
sand 0-4 mm [kg/m ³]	1370	1420
CEM I 42.5 N [kg/m ³]	675	612
superplasticizer [kg/m ³]	4.9	4.9
water [kg/m ³]	292	264
SUM TOTAL	2342	2301
w/c	0.43	0.43
Na ₂ O-equivalent [kg/m ³]	6.03	6.03
total K ₂ O + Na ₂ O [kg/m ³]	8.51	8.26

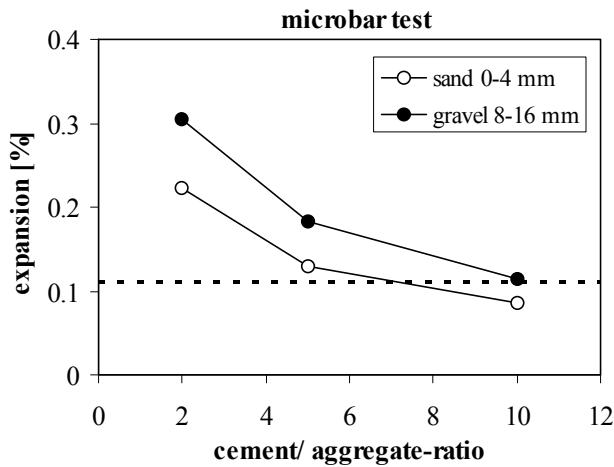


Figure 1: Expansion of microbars.

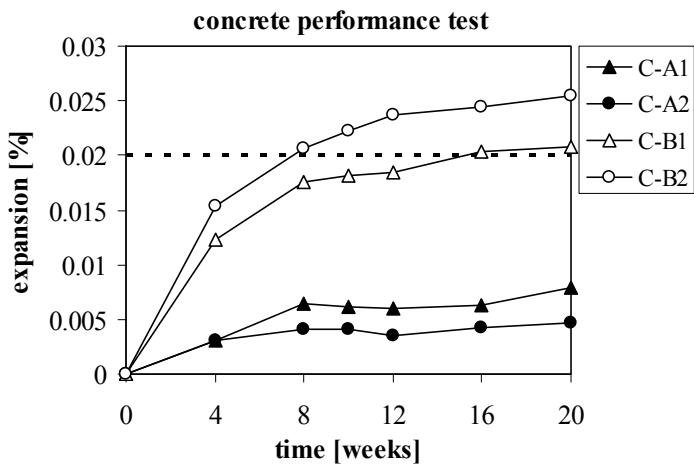


Figure 2: Expansion of concrete prisms.

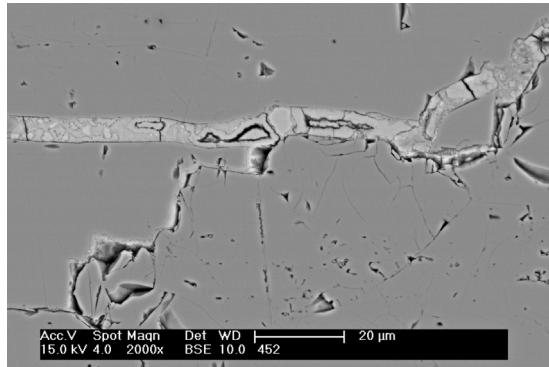


Figure 3: Crack filled with gel in quartzite of concrete C-B1.

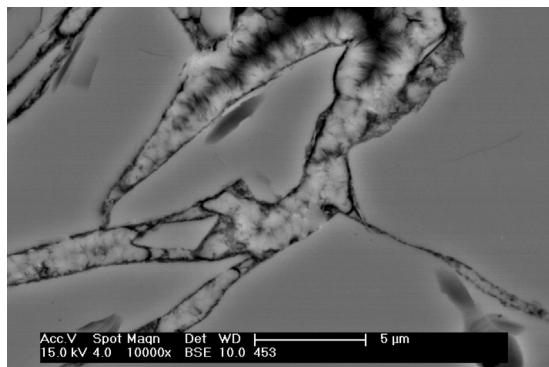


Figure 4: Cracks filled with gel in quartzite of concrete C-B1.

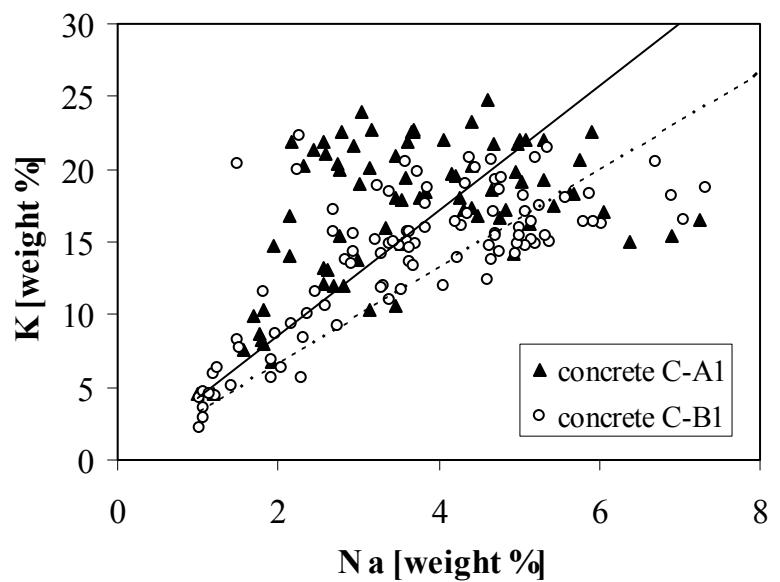


Figure 5: Potassium and sodium in ASR gel in quartzite of concrete C-A1 and C-B1.

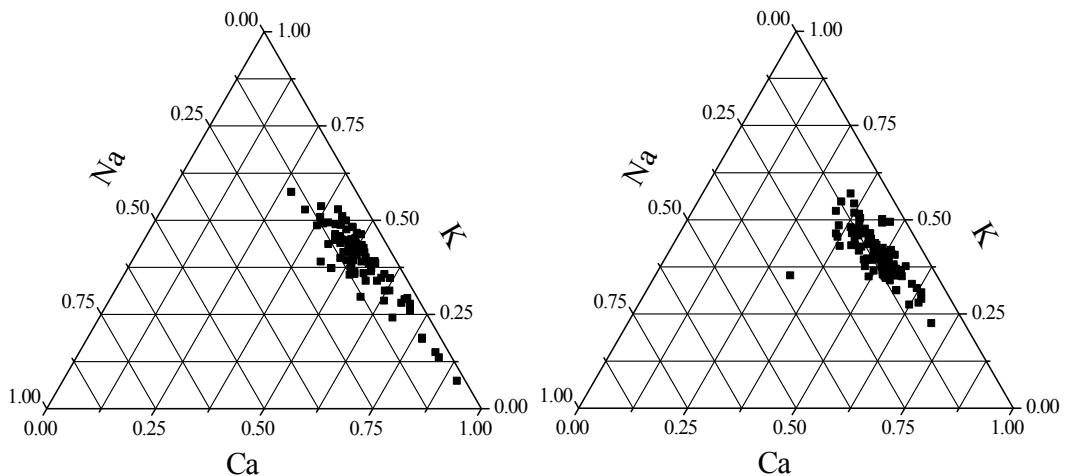


Figure 6 and 7: Composition of ASR gel in quartzite of concrete C-A1 (left) and concrete C-B1 (right) plotted in a ternary Ca-K-Na diagram (sum in weight-% of Ca-K-Na = 1.0).

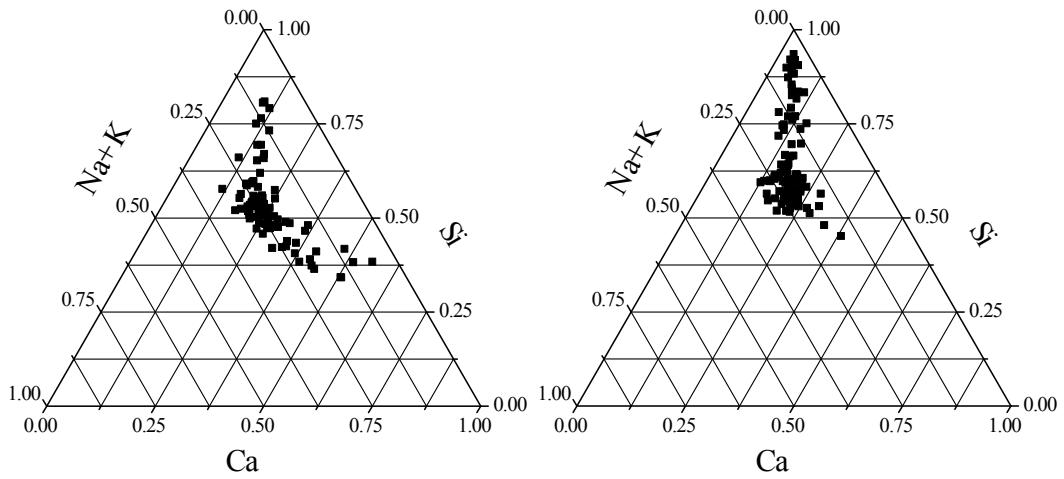


Figure 8 and 9: Composition of gel in quartzite of concrete C-A1 (left) and concrete C-B1 (right) plotted in a ternary Ca-Si-(K+Na) diagram (sum in weight-% of Ca-Si (K+Na) = 1.0).

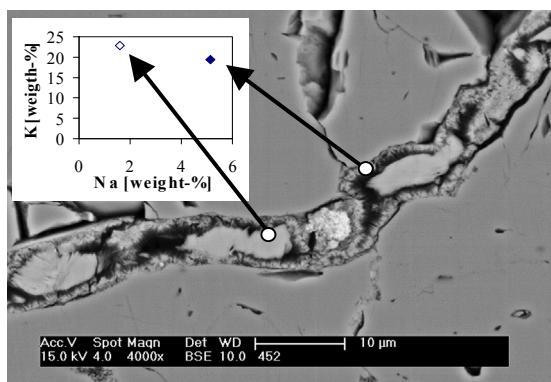


Figure 10: Crack filled with gel in quartzite of concrete C-B1 and K/Na-ratio of early (open symbol) and late gel (filled symbol).

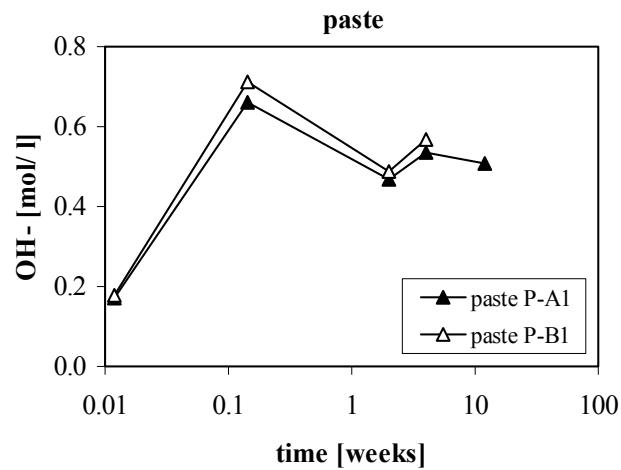


Figure 11: Hydroxide concentration in the pore solution of paste P-A1 and P-B1.

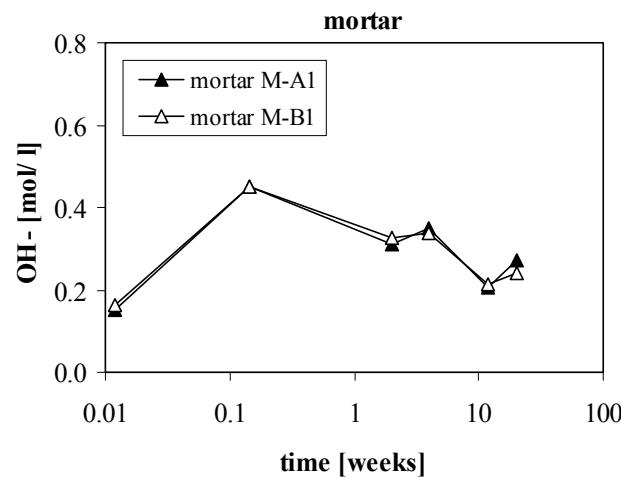


Figure 12: Hydroxide concentration in the pore solution of mortar M-A1 and M-B1.