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The influence of potassium–sodium ratio in cement on concrete expansion due to alkali-aggregate reaction

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ABSTRACT

In concrete containing potentially reactive aggregates, deleterious alkali-aggregate-reaction (AAR) can be prevented by the use of suitable mineral admixtures or by limiting cement content and alkalis (Na_2O-equivalent) of the cement. However, the Na_2O-equivalent of cement may not always accurately define the potential of cement to cause AAR. In this study, the potential reactivity of concrete produced with cements having similar Na_2O-equivalents but different K/Na-ratios has been measured and the composition of gel has been analyzed. Additionally, pastes and mortars have been produced to study the development of pore solution composition.

The expansion of the concrete mixtures shows significant differences depending on the cement used. The different K/Na-ratio present in the cements is reflected in the pore solution of pastes and mortars and in the gel present in aggregates of the concrete mixtures. As the hydroxide concentration in the pore solutions of pastes and mortars produced with the different cements is nearly identical, the difference in K/Na-ratio has to be the reason for the observed differences in concrete expansion.

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. Another way 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 (like fly ash, slag or silica fume) mitigating AAR [e.g. [1–5]], by limiting the cement content or by the use of cement with low Na_2O-equivalent [e.g. [6–11]]. Various national guidelines and standards take measures to reduce the content of soluble alkalis in concrete by limiting the cement content or by limiting the Na_2O-equivalent of the cement [e.g. [12–16]]. However, the Na_2O-equivalent may not always be a reliable parameter defining the potential of cements to cause AAR. Firstly, the alkali metals 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 concentrations of dissolved alkali metals during the first stage of hydration is different. A high amount of sodium or potassium sulfates in the cements will lead to high alkali metal concentrations during the first hours of hydration. The total amount of potassium and sodium present, i.e. alkali sulfates and alkalis present in the silicate and aluminate phases, will determine the alkali metal concentrations at a later stage of hydration. 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 use of the term "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 (unpublished data). 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, the composition of the pore solution of cement pastes and mortars produced with two of these cements was investigated.

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

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Table 1
Cement composition in weight-% and K/Na-ratio based on molar concentrations

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
Cement	A1	B1	A2	B2
SiO_2	19.66	19.33	16.24	16.09
Al_2O_3	4.79	4.87	3.84	4.19
Fe_2O_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_2O	1.08	1.06	0.96	0.99
Na_2O	0.18	0.29	0.16	0.23
Mn_2O_3	0.06	0.06	0.07	0.06
TiO_2	0.27	0.23	0.23	0.21
P_2O_5	0.14	0.13	0.09	0.12
Cr_2O_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
Na_2O-equ [%]	0.894	0.986	0.798	0.881
K_2O+Na_2O	1.26	1.35	1.12	1.22
K/Na-ratio	3.95	2.41	3.95	2.83

containing limestone powder (CEM II/A-LL 42.5 N / Table 1). The Na_2O-equivalent of the cements of producer A is about 0.1% lower than the one of the cements of producer B; potassium content is nearly identical but sodium content of the latter is higher. Rounded alluvial sand and gravel was used as aggregate (Table 2). Content of CEM I 42.5 N was adjusted in a way that both concrete mixtures had the same Na_2O-equivalent (Table 3). For the concrete mixtures produced with CEM II/A-LL 42.5 N, the cement content was identical. The superplasticizer used is based on polycarboxylate ether (dosages given as liquid). Per mixture of concrete three prisms (7×7×28 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 lower (0.31) but 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, put in zip-lock bags and stored in an oven at 60 °C in order to simulate the temperature present in the concrete performance test (see following chapter).

2.2. Methods for analysis

The petrography of the aggregates shown in Table 4 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 (7×7×28 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 concrete mixture.

Table 2
Petrography of the aggregates

Grain size	0–4 mm [wt.%]	8–16 mm [wt.%]
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

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_2O-equivalent [kg/m^3]	3.35	3.35	2.79	3.08
Total K_2O+Na_2O [kg/m^3]	4.73	4.59	3.92	4.27
K/Na-ratio (based on molar concentrations)	3.95	2.41	3.95	2.83

After conducting the concrete performance test, one prism of concrete C-A1 and C-B1 was selected in order to analyze the reaction products. Six samples (35×65×20 mm) were cut from each prism and dried for three days at 50 °C. After drying they were impregnated with epoxy resin and polished. In order to avoid an influence of local alkali metal contribution by feldspar or mica, the composition of the gel was only analyzed in quartzite aggregates. 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 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.

The pore solutions of the mortar samples were either collected by vacuum filtration (hydration time 2 h) or by using the steel die method and pressures up to 530 N/mm^2 (hydration time 1 day, 2, 4, 12 and 20 weeks). The cement pastes yielded enough pore solution for an analysis up to an age of four weeks. The solutions were immediately filtered using 0.45 μm nylon filters. After filtration an aliquot was diluted with HNO_3 (6.5%) to prevent the precipitation of solid phases. The remaining solution was used for pH measurements. The pH electrode was calibrated using KOH solutions of known concentrations. The total concentrations of the Na, K, Ca, Al, S, and Si were determined using inductively coupled plasma optical emission spectroscopy (ICP-OES). The measured sulphur (S) content corresponds to total sulphur content in the OPC system investigated. The K/Na-ratio given is based on the molar concentrations of the elements.

3. Results

3.1. Aggregate 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

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_2O-equivalent [kg/m^3]	6.03	6.03
Total K_2O+Na_2O [kg/m^3]	8.51	8.26
K/Na-ratio (based on molar concentrations)	3.95	2.41

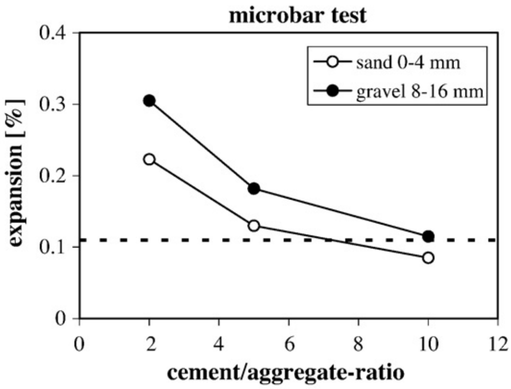

Fig. 1. Expansion of microbars.

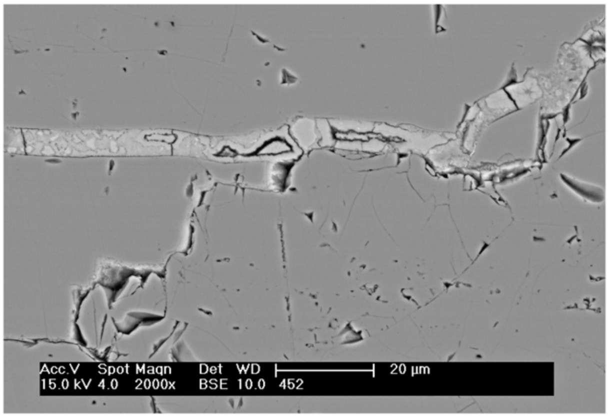

Fig. 3. Crack filled with gel in quartzite of concrete C-B1.

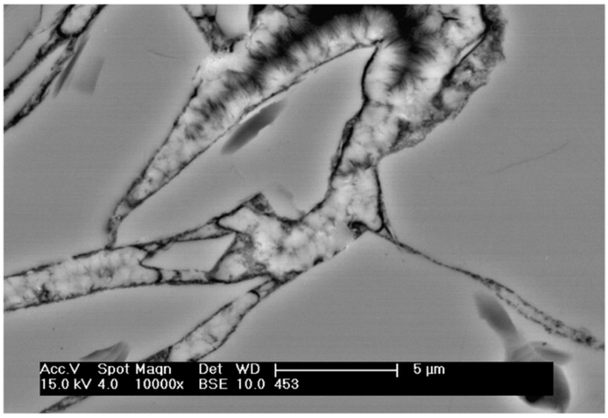

Fig. 4. Crack filled with gel in quartzite of concrete C-B1.

reactive (Fig. 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 (Fig. 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 (Figs. 3 and 4). The morphology of the gel varies. Sometimes it is structure-less and fills cracks entirely, sometimes it forms needle-like agglomerates growing inward from the edges of cracks present in aggregates. In between, all combinations occur. The gel in quartzite is mainly composed of Si, Ca, K and Na. Traces of Mg, S and Al are often present. The composition including the K/Na-ratio of the gel varies considerably. However, the results indicate that the average K/Na-ratio in concrete C-A1 is higher than in C-B1 (Fig. 5). In a ternary Ca–K–Na diagram this difference can be seen as well (Fig. 6). 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 (Fig. 7).

In some aggregates, gels of different generations are observed. Usually, the older or earlier gel is present in fragments in the central area of cracks (Fig. 8). In general, the gel of the later generation forms at the edge of the cracks. Without exception the composition of these gels is different; the gel of the earlier generation exhibits a higher K/Na-ratio than the gel formed later (Fig. 8).

3.4. Pore solution

The results of pore solution analysis are summarized in Table 5. Generally, the pore solutions of pastes and mortars show the same relative development with time. In the pastes the absolute concentrations of the main components sodium, potassium, sulphur and hydroxide ions are higher as a result of the lower w/c used in the pastes (e.g. Figs. 9 and 10). The calcium concentrations reach similar values in

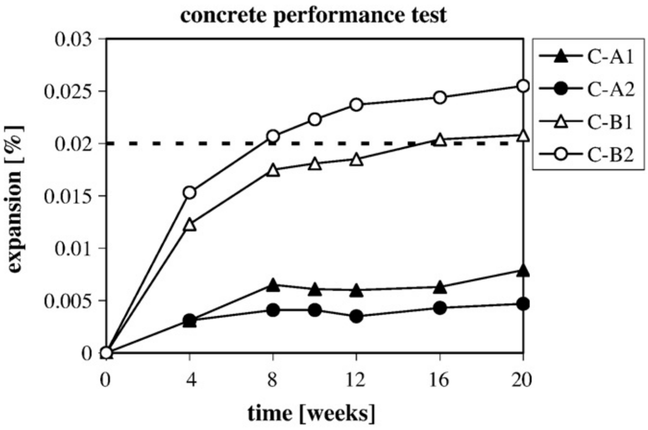

Fig. 2. Expansion of concrete prisms.

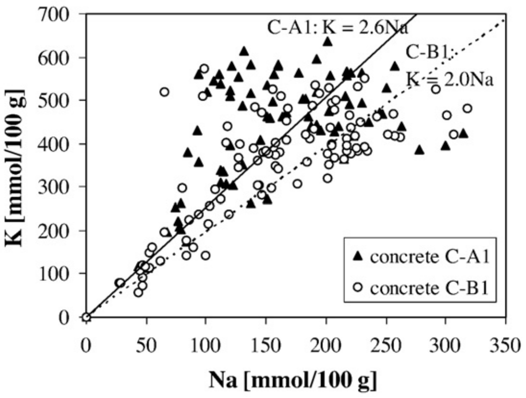

Fig. 5. K/Na-ratio of gel in quartzite of concrete C-A1 and C-B1.

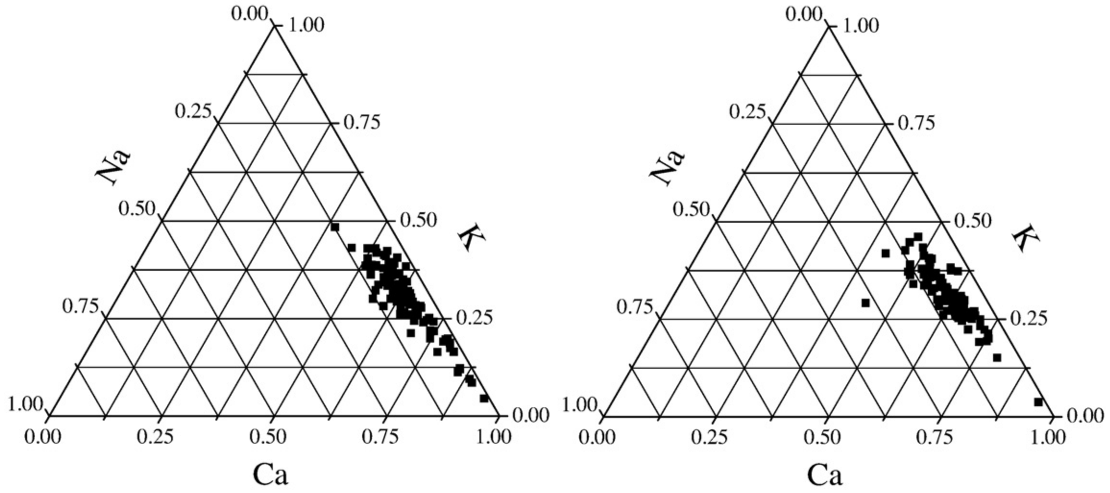

Fig. 6. Composition of gel in quartzite of concrete C-A1 (left) and concrete C-B1 (right) plotted in a ternary Ca–K–Na diagram (sum in mol% of Ca + K + Na = 1.0).

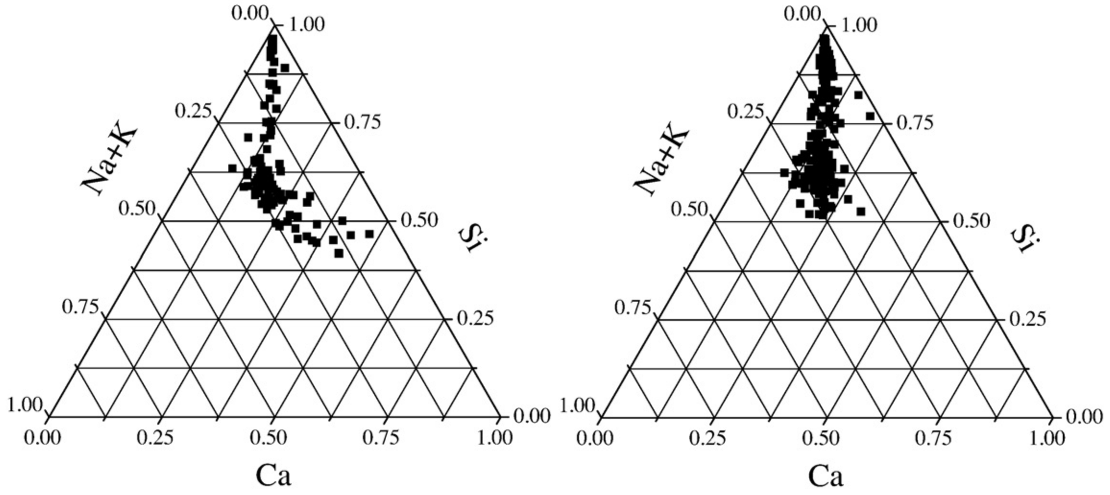

Fig. 7. Composition of gel in quartzite of concrete C-A1 (left) and concrete C-B1 (right) plotted in a ternary Ca–Si–(Na + K) diagram (sum in mol% of Ca + Si + (Na + K) = 1.0).

pastes and mortars and the silicon concentrations increase in the pore solutions of the mortars after the first day and exceed the concentrations present in the pore solution of the pastes (Figs. 11 and 12).

The influence of the cement on pore solution composition is the same in pastes and mortars. For sake of simplicity, their effect is described on the basis of the mortars. The alkalinity of mortar M-A1 and M-B1 is absolutely congruent during the measured period of 20 weeks. The maximum hydroxide concentrations are present at the age of one day. Afterwards, they decrease gradually while sulphate concentrations increase. Sodium concentration in the pore solution of mortar M-A1 is significantly exceeded by mortar M-B1. The difference reaches a maximum at the age of two weeks and starts to decrease afterwards (Fig. 13). Potassium concentrations are slightly lower in mortar M-B1 than in mortar M-A1, at the age of 20 weeks it is slightly higher (Fig. 14). Lithium concentrations are minor and they are identical in both mortars after the age of two weeks (Fig. 15). There are no significant differences in calcium and sulphur concentrations. However, the silicon concentration in the pore solution of mortar M-B1 reaches higher values after one day than in mortar M-A1 (Fig. 12).

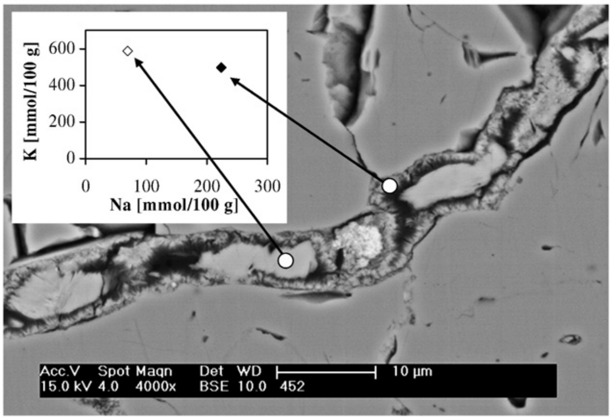

Fig. 8. Crack filled with gel in quartzite of concrete C-B1 and K/Na-ratio of early (open symbol) and late gel (filled symbol).

4. Discussion

Although the differences in composition of the cements of producers A and B are minor, the resulting potential reactivity of concrete mixtures is significantly different. The main difference in the chemical compositions of the cements is their Fe_2O_3, MgO and Na_2O content. Because of their low solubility Fe_2O_3 and MgO do not affect pore solution composition. However, the difference in Na_2O content of the unhydrated cement is reflected in a higher sodium concentration

Table 5
Measured concentrations in the pore solutions of pastes (P) and mortars (M) hydrated at 60 °C

p/m	Time	Na	K	Ca	Si	S	Al	Li	OH^-
	[weeks]	[mmol/l]							
Pastes									
P-A1	0.01	114	301	23.7	0.08	216	0.009	0.44	170
	0.14	140	573	2.6	0.55	85	0.038	1.93	660
	2	191	624	3.8	0.37	339	0.148	2.32	470
	4	187	647	3.6	0.40	342	0.026	2.26	540
P-B1	0.01	121	321	26.0	0.08	240	<0.015	1.57	180
	0.14	214	581	2.2	0.52	45	0.082	1.42	710
	2	271	524	3.2	0.33	246	<0.015	2.23	490
	4	275	512	3.2	0.39	240	0.038	2.51	570
Mortars									
M-A1	0.01	72	272	24.9	0.08	188	0.012	2.09	150
	0.14	85	334	3.0	0.30	9	0.050	1.39	450
	2	94	288	2.9	0.35	87	0.027	0.91	310
	4	90	273	2.5	0.44	83	<0.015	0.66	350
	12	106	262	4.0	0.46	142	0.032	0.48	210
	20	115	218	2.8	0.36	88	0.049	0.48	270
M-B1	0.01	77	246	26.0	0.06	160	<0.015	0.61	160
	0.14	119	309	3.1	0.28	6	0.067	0.79	450
	2	142	281	2.6	0.63	90	<0.015	0.78	330
	4	131	253	2.1	0.63	84	0.050	0.69	340
	12	132	215	2.7	0.82	107	0.047	0.46	210
	20	142	251	1.8	0.71	104	0.078	0.48	240

The values correspond to total concentrations with exception of OH^-, which refers to the free concentration.

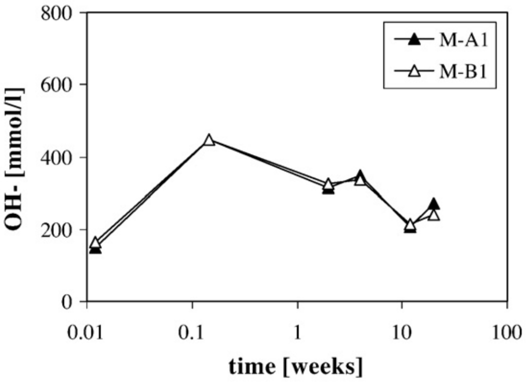

Fig. 10. Hydroxide concentration in the pore solution of mortar M-A1 and M-B1.

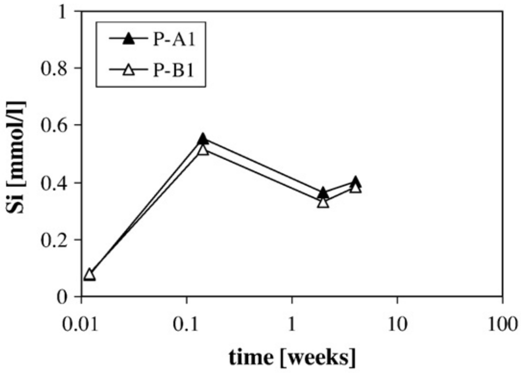

Fig. 11. Silicon concentration in the pore solution of paste P-A1 and P-B1.

in the pore solution of paste P-B1 and mortar M-B1. As a result, the K/Na-ratio is different as well (Fig. 16). The cements of producer B have a slightly higher Na_2O-equivalent and a higher combined content of K_2O+Na_2O. The slightly higher content of K_2O+Na_2O present in cement B1 is reflected in the hydroxide concentration of the pore solutions in the pastes but is not observed in the pore solutions of the mortars.

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 and were exposed to air. Nevertheless, it can be assumed that these differences do not influence the comparability of the different cements used. Generally, the hydroxide concentration in sealed samples is higher than in the ones exposed to high humidity due to leaching [23]. It is obvious that the hydroxide concentration of the pore solution in pastes and mortars cannot give an explanation for the substantial difference observed in concrete expansion. The most significant difference in the pore solution of pastes and mortars produced with the two different cements is the K/Na-ratio. Although there is a wide scatter in the results, the relative difference seems to be present as well in the gel as analyzed in cracks of quartzite aggregates in concrete C-A1 and C-B1. K/Na-ratio in the pore solution and in the gel are in the same range (Fig. 16) and they reflect the differences present in cement composition. Obviously, there is no preferential incorporation of either sodium or potassium in the reaction products.

The expansion due to AAR is a two step process consisting of a dissolution and an expansion process. Firstly, dissolution of reactive silica or quartz in aggregates is caused by a break down of Si–O bonds due to the attack of alkali hydroxides. Secondly, the resulting silica gel (containing also potassium, sodium and calcium) swells due to water take up and develops an expansive pressure. The kinetics of quartz dissolution depends strongly on pH and temperature. It is increased by the presence of cations; more notably in the presence of Na than K [19,24], which would agree with the greater expansion observed for

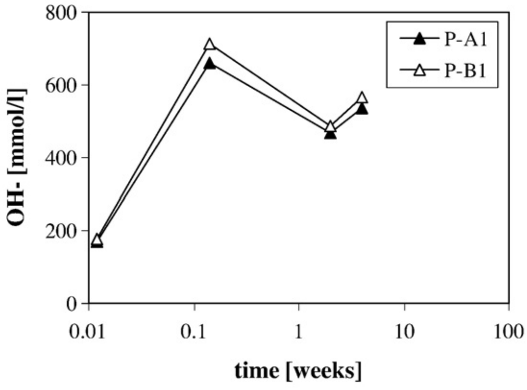

Fig. 9. Hydroxide concentration in the pore solution of paste P-A1 and P-B1.

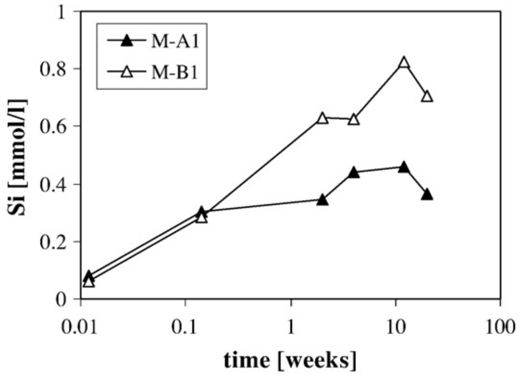

Fig. 12. Silicon concentration in the pore solution of mortar M-A1 and M-B1.

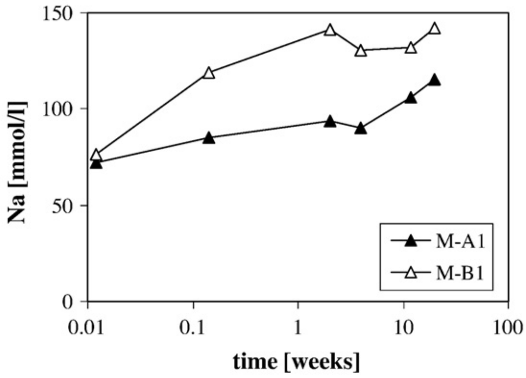

Fig. 13. Sodium concentration in the pore solution of mortar M-A1 and M-B1.

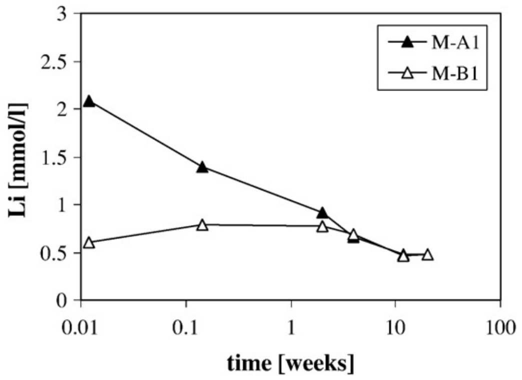

Fig. 15. Lithium concentration in the pore solution of mortar M-A1 and M-B1.

cement B. In contrast, for silica gel a higher rate of dissolution for potassium hydroxide than for sodium hydroxide has been observed experimentally [25]. Because little data exist on the combined effect of various ions [26], statements about the influence of K/Na-ratio on the rate of dissolution in alkaline conditions is difficult. The substantial differences in concrete expansion may be not only the results of different degrees of dissolution but are also substantially influenced by gel composition. The expansion mechanisms of alkali-aggregate reaction are still not clear [e.g. [27–31]. 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.

On the one hand side, gels with a high calcium concentration are supposed to show no or low expansion pressure behavior [27,31]. On the other hand side, the presence of calcium seems to be mandatory for an expansion to take place [32,33]. 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 indistinguishable with regard to the resulting surface charge density and expansive pressure respectively [34,35]. However, 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 [36]. The results of the present study indicate as well that the influence of potassium and sodium on expansion is not identical. However, the observed higher expansion of concrete B seems to be mainly caused by the lower K/Na-ratio (higher relative sodium content) both in pore solution and reaction products. The variations in calcium and silicon content of the gels are minor and they can hardly explain the measured concrete expansion.

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. This is in agreement with many observations reported in the literature 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 silicate and aluminate phases [e.g. [37]].

Expansion of the concrete prisms decelerates with increasing age. This could indicate that the alkalinity of the pore solutions has dropped below a critical level due to the consumption of alkali hydroxides (in the gels formed) and/or due to alkali leaching. 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 further supporting that the difference in K/Na-ratio is the trigger for the resulting expansions.

The only obvious difference between the four 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 Na_2O-equivalent to assess or decrease the potential reactivity of concrete by prescription of mix design can be misleading. In order to further investigate this problem more expansion tests have to be combined with pore solution analysis. In accelerated expansion tests the influence on temperature must not be neglected. While high temperatures do increase the reaction rates and thus the expansion, they also change the composition of the hydrate assemblages as well

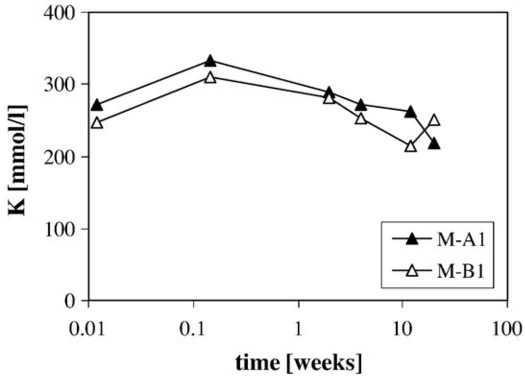

Fig. 14. Potassium concentration in the pore solution of mortar M-A1 and M-B1.

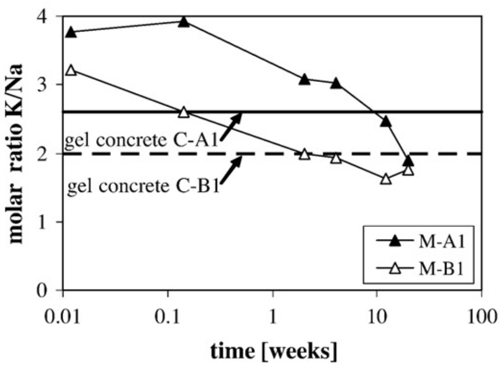

Fig. 16. K/Na-ratio in the pore solution of mortar M-A1 and M-B1. The horizontal lines show the mean K/Na-ratio of the gel in concrete C-A1 and C-B1 measured after 20 weeks.

as the composition of the pore solution [38,39]. The most distinct change with temperature concerns the sulphate concentrations which increase strongly with temperature. At 60 °C and higher these high sulphate concentrations found in the pore solutions do also lower the hydroxide concentrations in the pore solutions, thus possibly affecting AAR. Furthermore, dissolution 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 Na_2O-equivalent but different K/Na-ratios can expand considerably differently as an accelerated performance test to assess the potential reactivity of AAR shows. Consequently, the Na_2O-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 solution 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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