

# THE INFLUENCE OF SUPPLEMENTARY CEMENTING MATERIALS ON THE DISSOLUTION OF AGGREGATES IN ALKALINE SOLUTIONS

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## Abstract

Starting from expansion measurements on concrete prisms, produced with two different alkali reactive aggregates, the influence of two aluminium containing SCMs on the expansion of concrete prisms and on the solubility of the aggregates in an alkaline solution were investigated. The solubility of aggregates in an alkaline solution reflects the alkali reactivity of the corresponding aggregate. Initially, the solubility experiments with the aggregates and SCMs were made separately and then in mixtures of each aggregate and SCM. In a further step, the SCMs were stored in KOH solutions containing  $\text{Ca}(\text{OH})_2$ . The concentrations of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  were determined to investigate the effect of calcium on the solving processes of SCMs. The combination of concrete expansion test and dissolution experiments of two alkali reactive aggregates and two aluminium containing SCMs allows first conclusions about the influence of such SCMs on the reactivity of aggregates.

**Keywords:** alkali silica reaction, supplementary cementing materials (SCM), solubility of aggregate, solubility of SCM

## 1 INTRODUCTION

The addition of supplementary cementing materials (SCMs) to avoid AAR in concrete is known and accepted worldwide. But it seems that the effect and inhibition mechanism of this reaction, especially with aluminium containing SCM, is unclear and therefore current subject of international investigations [1]-[10]. The effectiveness of SCM in the cement stone matrix under constant conditions like type of cement, water/cement ratio and the same exchange level is essentially dependent on the aggregate. This is an indication that interactions between SCMs and aggregate arise that are decisive for the dosage of the required additive amount for safe prevention of AAR. This interaction between aluminium-containing additives and reactive aggregate should be investigated by means of dissolution experiments.

As part of a research project funded by the German Research Foundation "Deutsche Forschungsgemeinschaft" (DFG), the influence of different supplementary cementing materials on the solubility of aggregates is investigated. First results of solubility studies of two different reactive aggregates and two aluminium-containing SCMs in an alkaline solution at 40°C are presented in this paper.

## 2 MATERIALS AND METHODS

### 2.1 General

The aim of the experimental study is to show at first the influence of SCM on the reactivity of aggregates in concrete based on expansion measurements. Further dissolution experiments aim to draw knowledge, as the solubility of  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  from the aggregate is influenced by the additives.

In a first step the dissolution of the aggregate grains and the SCM in the alkaline solution was determined separately. Afterwards the same procedure was applied to the mixture of aggregate and SCM. Step into another the aggregates are stored in mixtures of KOH and  $\text{Ca}(\text{OH})_2$  solution in order to investigate the influence of calcium ions on the dissolution processes, because  $\text{Ca}^{2+}$  ions are always available in the pore solution of the concrete.

The comparison of the chemical dissolution values and the expansion of the concrete prisms will provide insights regarding the interactions between SCM and aggregate.

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## 2.2 Materials and mix designs

Two aggregates with a different reactivity, a greywacke G9 and a rhyolite R2, were selected. The classification as reactive aggregates refers to the results of the concrete prism test (CPT). The mineralogical compositions determined by XRD on finely powdered samples are summarized in Table 1. As SCMs were used two aluminium containing commercially available mineral admixtures: metakaolin MK and fly ash FA. MK called Powerpozz was purchased from NEWCHEM AG and Powerment as FA was used by the company Powerment AG. The chemical compositions are listed in Table 2.

For the CPT, the aggregates with a grading curve A/B 16 mm according to [11] were used as reactive components and inert quartz sand for the fraction 0/2 mm. Furthermore, portland cement CEM I 32.5 R was used with an alkali equivalent of 1.07 wt.%  $\text{Na}_2\text{O}_{\text{eq}}$ . The mineral admixture amount of 4% is related to the content of the aggregate in the fraction 2/16 mm. The concrete prisms had a size of 100x100x500 mm<sup>3</sup> and were produced with 400 kg/m<sup>3</sup> binder (cement and SCM) at a water/binder ratio of 0.45. The cement respectively binder was doped with  $\text{K}_2\text{SO}_4$  to an alkali equivalent of 1.30 wt.%  $\text{Na}_2\text{O}_{\text{eq}}$ , corresponding to 5.2 kg  $\text{Na}_2\text{O}_{\text{eq}}$ /m<sup>3</sup> concrete, in order to guarantee the same alkali level in all concrete species. The concrete compositions are listed in Table 3.

## 2.3 Methods for assessment and analysis

### *Concrete prism test*

The concrete prisms were stored in a fog chamber at 40°C till 1 year, meanwhile the expansion is detected. These conditions exceed the demands of [11], where the limiting value is 0.6 mm/m at 280 days.

### *Solubility analyses*

For the solubility investigations of the aggregates G9 and R2, the fraction 2/8 mm was used. 90 g of the aggregate were put into 900 g of alkaline solution with a concentration of 1 Mol/L potassium hydroxide. The mixtures of grains and 1M KOH solution were stored in tightly sealed PE-bottles at 40°C till 100 days [12].

Corresponding to the CPT with 4 wt.% SCM related to the coarse aggregate content, 3.6 g MK and FA, respectively, were also stored in 1M KOH to determine their dissolution behaviour. Afterwards, the aggregates and SCMs were tested together in the same procedure, e.g. 90 g aggregate and 3.6 g SCM in 900 g KOH. After 4, 14, 28, 56, 90 till 300 days, respectively, approximately 3 ml of solution were taken from the liquid phase, filtered through a 0.2 µm membrane filter and the  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  concentrations were determined by an ICP-OES spectrometer.

## 3 RESULTS

### 3.1 Concrete prism test with different aggregates

For the solubility experiments, two different types of aggregates were chosen: a greywacke G9 and a rhyolite R2. The results of their corresponding expansion of concrete prism test are shown in Figure 1. It is obvious that the greywacke is clearly more reactive than aggregate R2 and causes a very high expansion of 2.25 mm/m after 200 days storing in the fog chamber. R2 is also reactive because the expansion exceeds 0.6 mm/m after 280 days of storage, but the expansion process is slower than with G9 and the expansion is only 0.8 mm/m at 280 days but proceeds. This was the starting point for the following solubility experiments in order to investigate how the SCMs act on the alkali sensitivity of the mentioned aggregate types. For the effect of SCM addition, the first 100 days seem to be important. That is the reason why this period was deeper investigated.

### 3.2 Solubility of the aggregates and the SCMs in KOH separately

Figure 2 shows the concentration courses of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  in the alkaline solution of the pure aggregates G9 and R2, respectively, and the SCMs metakaolin and fly ash till 250 days (Figures 3 and 4). As it can be seen in Figure 2, the aggregate G9 provides mainly  $\text{SiO}_2$  to the alkaline solution. After 250 days at 40°C, the concentration is about 1,350 mg/L. In contrast, the  $\text{Al}_2\text{O}_3$  concentration reaches only values of maximal 6 mg/L after 250 days. From the curve progression results, that the dissolution process will continue over a long period. The reaction of the aggregate R2 is slower than that of G9, related to the  $\text{SiO}_2$  concentration, but at every time, there is more dissolved  $\text{Al}_2\text{O}_3$  in the solution.

The dissolution of SCMs proceeds considerably faster, shown in Figures 3 and 4. Although the suspensions contain only 4 wt.% SCM, related to the mass of the pure aggregates (Figure 2), the concentration level of  $\text{SiO}_2$  lays in the region of about 800 till 1,200 mg/L. Furthermore, substantial

amounts of alumina are dissolved from the SCMs. Meanwhile the highest SiO<sub>2</sub> concentrations are comparable (900 mg/L for MK, 1,200 mg/L for FA), over the whole storage time, MK provides more Al<sub>2</sub>O<sub>3</sub> than SiO<sub>2</sub> to the solution whereas from the fly ash, only less than the half Al<sub>2</sub>O<sub>3</sub> is dissolved. Calculating the corresponding molar Si to Al ratio for the two SCMs, a value Si/Al < 1 for MK results what means that an excess of Al in solution exists. From the Si/Al value of 2.4 it can be seen that FA has a SiO<sub>2</sub> excess itself (see Figure 3 and 4). This seems to be the main difference between the two SCMs investigated.

### 3.3 Solubility of mixtures of aggregate and SCM in KOH

The effects of SCMs on the dissolution process of the aggregates are shown in Figures 5 and 6, as an example for G9 with 4 wt.% MK and FA. Till 100 days, the dissolution process is mainly influenced by the SCM. This is primarily caused by the already mentioned faster reaction of the SCMs with the KOH solution. But there is no adequate contribution of the aggregate to the concentration run, because the calculated concentrations provided by the aggregate and the SCM differ from the experimental results. In the case of G9 with MK, the SiO<sub>2</sub> concentration seems to be identical till 100 days, but the aluminium concentration is reduced. In the mixture of G9 and 4 wt.% FA, both the SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> concentrations are lower than the calculated sum from the aggregate and the SCM. These statements also apply to the mixtures of R2. Figures 7 and 8 show the changes in the molar Si/Al ratio of the corresponding aggregate by the use of additional SCMs. In both cases, the Si/Al ratio is decreased to values of 3 till 5 by FA and 1 by MK. Because G9 gives a much higher Si/Al ratio in solution than R2 the effect of the two SCMs is considerably higher on this aggregate type.

### 3.4 Influence of calcium hydroxide on the solubility of SCMs in KOH

From the previous described results, it can be seen that the solubility experiments in pure KOH solution are able to provide information about the alkali sensitivity of the aggregate and SCMs as well as their interaction. The next step in adapting the dissolution parameters to the conditions in real concrete is the investigation of the influence of calcium ions. Like in pore solutions, the Ca<sup>2+</sup> concentration corresponds to the saturation level and enough solid calcium hydroxide is available for further reactions. Under these conditions, the SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> concentrations are dramatically changed how it results from Figures 9 and 10. In the liquid phase of the system MK/KOH/Ca(OH)<sub>2</sub>, there is observed a maximum of dissolved SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> at 28 days. At 96 days the Al content of the solution is halved compared with the Ca free system. The SiO<sub>2</sub> concentration is reduced to a tenth of that in the Ca free solution. The effects for the system FA/KOH/Ca(OH)<sub>2</sub> are still stronger, reduction to 1/20 of the SiO<sub>2</sub> concentration and 1/4 for Al<sub>2</sub>O<sub>3</sub>. Comparing the remaining dissolved species of the two different SCMs, the Al concentration in the solution with MK is still relatively high, whereas most of the dissolved SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> coming from FA are not yet available for the interaction with the aggregate.

### 3.5 Expansion of concrete with SCMs

In a final step, the influence of MK and FA on the expansion of concrete with 4 wt.% SCM related to the aggregate content was tested. The results are shown in Figures 11 and 12. In concretes made with aggregate G9 (Figure 11), a strong reduction of the expansion from 2.25 mm/m at 200 days to 0.5 mm/m and 0.19 mm/m, respectively, is caused by both SCMs. Especially, the sharp increase of the expansion beginning at about 50 days can be prevented. But it seems that the limit of 0.6 mm/m will be exceeded with 4 wt.% FA later. Concerning the absolute values of the expansion, MK is more effective than FA at the same dosage. This sequence also applies to the concrete prism test with aggregate R2, depicted in Figure 12. But the reduction related to the initial value of the pure aggregate concrete is much smaller, e.g. from 0.66 mm/m without SCM to 0.32 mm/m with 4 wt.% FA and 0.21 mm/m with MK. So the effect of the particular SCM depends not only on its own reactivity, but also on the aggregate type.

## 4 DISCUSSION

### 4.1 Solubility of the aggregates in KOH and concrete expansion

The main result of the solubility experiments of the pure aggregates and the SCM is the fact that at 40°C the aggregate dissolves very slowly whereas the SCMs have a higher dissolution rate resulting in higher concentrations of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> at a given time. Furthermore, there is a big difference in soluble alumina content. In order to compare these absolute values of concentrations, it is useful to calculate the molar Si to Al ratio (Si/Al (molar)) because this value can characterize the situation in the liquid phase more detailed. According to [10], in alkaline solutions containing

dissolved  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  from aggregates, the formation of alkali aluminosilicates proceeds which do not contribute to swelling processes during ASR. They are innocuous and only the “excess-  $\text{SiO}_2$ ” is available for ASR. Using this approach, the preferred Si/Al ratio in alkali aluminosilicates is 1, also shown in [13]. Hence, the higher the Si/Al ratio, the more  $\text{SiO}_2$  is available for ASR. For the aggregate G9 used here, this means that nearly the whole  $\text{SiO}_2$  dissolved can form ASR gels in corresponding concrete specimens because the very low  $\text{Al}_2\text{O}_3$  concentration causes a high Si/Al ratio shown in Figure 7. This fact should be the reason for the very high expansion potential of this aggregate in concrete. For the aggregate R2, the Si/Al ratio is much lower (about 1/10 of G9), connected with a lower expansion in the concrete prism test (see Figure 1). These results confirm the relationship between dissolution behaviour of aggregates and concrete expansion.

#### **4.2 Solubility of the SCMs in KOH**

The same procedure is also applicable for the SCMs. So it is possible to evaluate the reactivity and, especially, the contribution of aluminium to the dissolution process. Thus it appears that MK has the lowest Si/Al ratio, much lower than that of FA, what means that MK should have a better influence on the solubility data and finally the concrete expansion of G9 than FA. The values of Si/Al < 1 for MK also indicate that there is free aluminium available in the solution for blocking  $\text{SiO}_2$  from the aggregate in mixtures of aggregate and SCM depicted in 3.2. Furthermore, the investigations show that not only the absolute values of the concentrations are important but also the ratio of dissolved  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ .

#### **4.3 Solubility of mixtures of aggregate and SCM in KOH**

The results show that the dissolution process of mixtures of aggregates and SCM at 40°C is mainly determined by the SCM, because it is more reactive in alkaline solution than the aggregate. But it is essential that the concentrations in solution are not the sum of the both constituents determined separately before. All concentrations are changed to lower values whereby the molar Si/Al ratio decreases compared with the dissolution process of the pure aggregate, but somewhat increases compared with the SCM. This fact should be a consequence of an interaction between the aggregate and the SCM via the KOH solution. By trend, the mixtures with more reactive aggregate G9 undergo stronger changes in the Si/Al ratio than the less reactive one (R2). Furthermore, the SCM with a higher content of soluble aluminium (MK) appears stronger than FA.

#### **4.4 Influence of calcium hydroxide on the solubility of SCMs in KOH**

As the described results show, the reactivity of aggregates and SCMs and their interaction can be recorded by solubility measurements in 1M KOH solutions. If calcium ions are available in this process, insoluble compounds are formed like CSH or CAH phases. Consequently, the concentrations of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  decrease. The extent of this competitive reaction is not yet predictable. Our first results show that MK provides even under  $\text{Ca}^{2+}$  influence more aluminium to the solution than FA. These investigations are continued, also in the system aggregate/SCM/KOH/ $\text{Ca}(\text{OH})_2$  and in corresponding hardened cement pastes.

#### **4.5 Expansion of concrete with SCMs**

The solubility data of two different aggregates with two aluminium proving SCMs are a suitable mean for interpreting the expansion behaviour. Comparing the changes of the Si/Al ratio during dissolution (see Figures 7 and 8) with the expansion data of the corresponding concrete prisms (see Figure 11 and 12), big similarities are obvious. The molar Si/Al ratio in alkaline solution of aggregate G9 is stronger reduced by SCMs than R2, because itself has a lower ratio than G9. Till 150 days of storage in alkaline solution, the addition of FA gives a value of Si/Al = 3-5 and MK of 1 for both aggregates. The same tendencies can be seen in concrete expansion test. Although the aggregates without SCM show very different expansion, at 150 days the situation is comparable: expansion 0.3 – 0.5 mm/m with FA addition and 0.2 mm/m with MK. Further investigation is needed for the evaluation the long term performance of these SCMs in order to find the ideal combination of SCM and the particular aggregate.

## **5 CONCLUSIONS**

The combination of concrete expansion test and dissolution experiments of two alkali sensitive aggregates and two aluminium containing SCMs have shown that

- aggregates which provide relatively high concentrations of aluminium compared with the corresponding SiO<sub>2</sub> concentration to KOH solutions are less expansive in concrete prism test than Al poor aggregates.
- a useful measure is the calculated molar Si/Al ratio which characterises the formation of aluminosilicates in the alkaline solution. MK with a ratio Si/Al < 1 has an aluminium excess and consequently, a higher potential for preventing ASR than FA.
- the effectiveness of SCMs can be deviated from their ability to lower the Si/Al ratio in alkaline solutions of the pure aggregates.
- calcium ions which are always present in pore solutions lower the capacity of the SCMs because competitive reactions proceed, also consuming dissolved aluminium.
- the optimal combination of aggregate and SCM depends on the dissolution behaviour of both components.

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TABLE 1: Mineralogical composition of aggregates G9 and R2 [wt.%].

Mineral	G9		R2	
	fraction 2/8 mm	fraction 8/16 mm	fraction 2/8 mm	fraction 8/16 mm
Biotite	3	2	10	8
Chlorite	13	11	-	-
Microcline	-	-	12	9
Plagioclase	7	7	29	31
Quartz	52	51	25	26
Calcite	15	16	-	-
Muscovite	10	13	-	-
Sanidine	-	-	25	27

TABLE 2: Chemical composition of SCMs MK and FA [wt.%].

SCM	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	SO <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>
FA	55.0	24.3	7.4	1.3	4.1	1.4	0.9	1.8	0.7	0.6
MK	55.0	41.0	< 1.4	< 3.0	< 0.1	< 0.1	< 0.05	< 0.4	< 0.05	< 0.2

TABLE 3: Concrete composition

	cement	water	SCM	sand 0/2 mm	gravel 2/8 mm	gravel 8/16 mm	K <sub>2</sub> SO <sub>4</sub>
	kg/m <sup>3</sup>	kg/m <sup>3</sup>	kg/m <sup>3</sup>	kg/m <sup>3</sup>	kg/m <sup>3</sup>	kg/m <sup>3</sup>	g
G9	400	180	0	529	730	554	2676
G9 with SCM	349	180	51	529	730	554	4210
R2	400	180	0	529	703	532	2676
R2 with SCM	351	180	49	529	703	532	4151

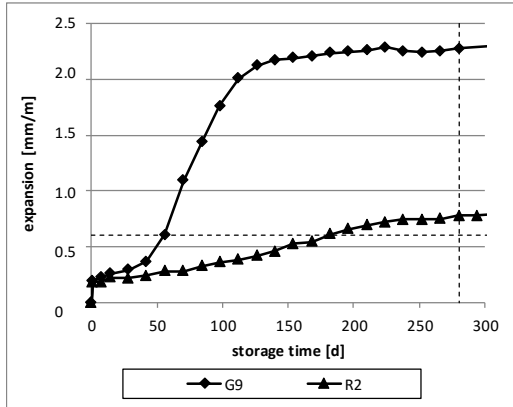


FIGURE 1: Expansions of concrete prisms with aggregates G9 and R2 according to German alkali Guideline versus storage time in the fog chamber at 40°C.

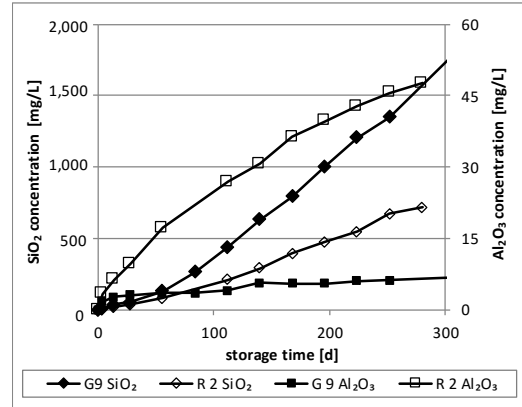


FIGURE 2: Dissolution of grain fraction 2/8 mm of aggregates G9 and R2 in 1 M KOH at 40°C versus storage time.

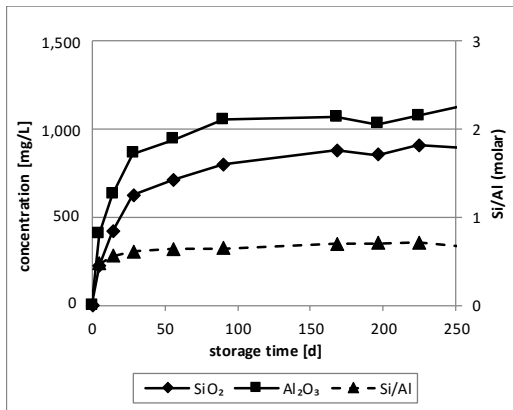


FIGURE 3: Solubility of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> and molar Si/Al-ratio of SCM MK 4 wt.% in 1M KOH at 40°C versus storage time.

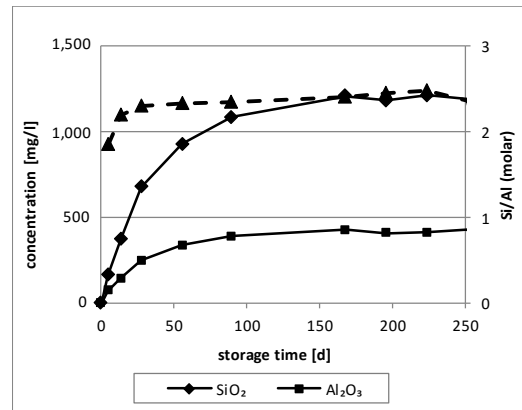


FIGURE 4: Solubility of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> and molar Si/Al-ratio of SCM FA 4 wt.% in 1M KOH at 40°C versus storage time.

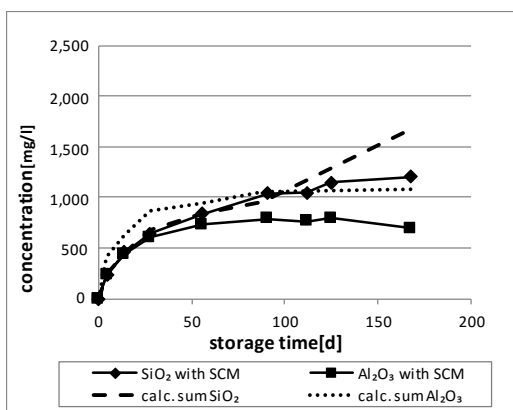


FIGURE 5: Solubility of SiO<sub>2</sub> resp. Al<sub>2</sub>O<sub>3</sub> of the mixture of aggregate G9 and SCM MK 4 wt.% and the calculated concentration of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> in 1M KOH at 40°C versus storage time.

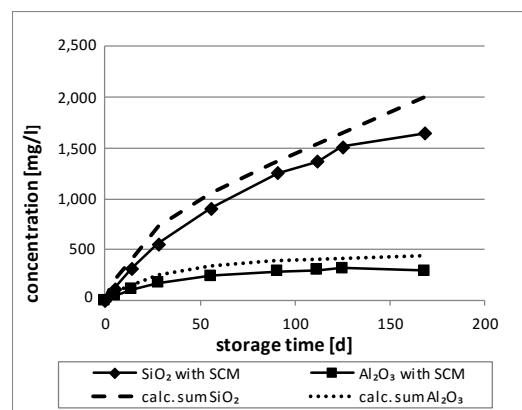


FIGURE 6: Solubility of SiO<sub>2</sub> resp. Al<sub>2</sub>O<sub>3</sub> of the mixture of aggregate G9 and SCM FA 4 wt.% and the calculated concentration of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> in 1M KOH at 40°C versus storage time.

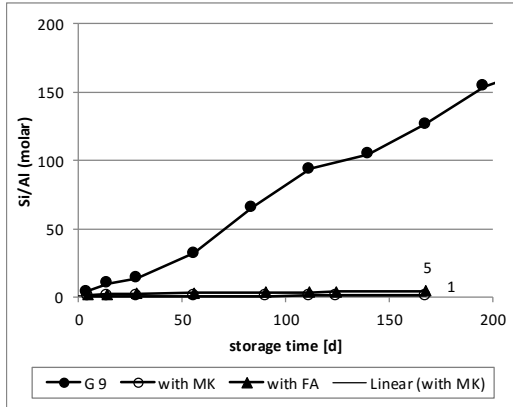


FIGURE 7: Comparison of molar Si/Al-ratio of the aggregate **G9** with and without SCM in 1M KOH at 40°C versus storage time.

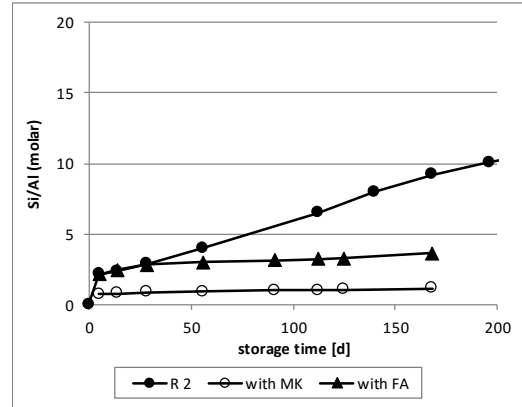


FIGURE 8: Comparison of molar Si/Al-ratio of the aggregate **R2** with and without SCM in 1M KOH at 40°C versus storage time.

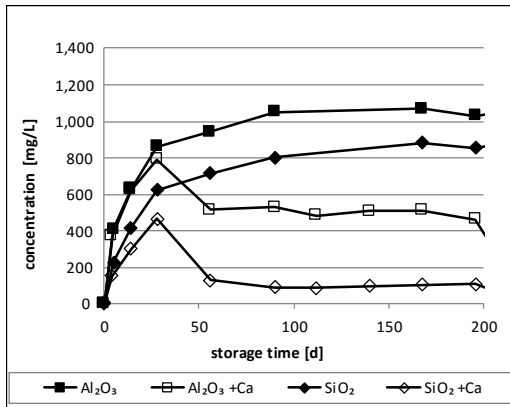


FIGURE 9: Influence of calcium hydroxide on the solubility of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> of the **MK 4 wt.%** in 1M KOH at 40°C versus storage time.

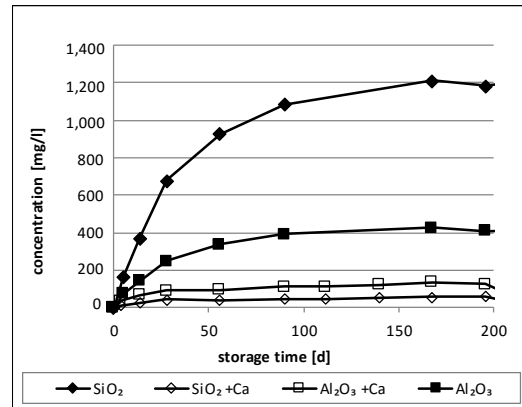


FIGURE 10: Influence of calcium hydroxide on the solubility of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> of the **FA 4 wt.%** in 1M KOH at 40°C versus storage time.

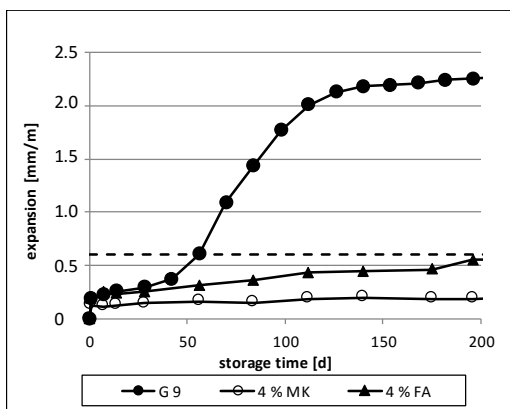


FIGURE 11: Influence of SCM **MK 4 wt.%** and **FA 4 wt.%** of the expansion of concrete prisms with aggregate **G9**.

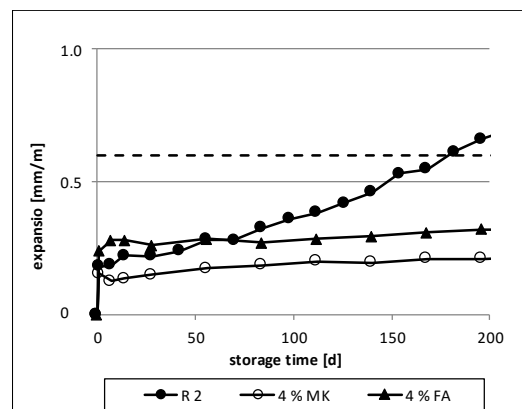


FIGURE 12: Influence of SCM **MK 4 wt. %** and **FA 4 wt.%** of the expansion of concrete prisms with aggregate **R2**.