

INFLUENCE OF AGGREGATE SIZE ON THE DAMAGE POTENTIAL OF ALKALI-SILICA REACTION

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Abstract

The susceptibility of different aggregate types used in concrete to ASR is strongly associated with their petrography. The aim of this study is to assess the influence of the aggregate size on the ASR potential, in particular applied to the sand fractions in the size range of 0.125-4 mm. The investigation concept is based on the hypothesis that at a specific particle size reactive aggregates could change their characteristic influence in mitigating ASR instead of promoting it.

For the study of the size dependent behaviour mortars are prepared, considering aggregates of different reactivity, including very reactive, slow-late and quasi non-reactive types. The analysis is based on a stepwise substitution concept, in which mortars with a constant overall grain size distribution are compared, while only one size fraction of the non-reactive base aggregate is substituted by a reactive aggregate type of this specific size fraction. For the expansion experiments an accelerated mortar bar test (80°C, immersed in NaOH solution) was applied. The results show that the slow-late reactive greywacke yielded a strong reduction of the expansion below an aggregate size in the range of 0.5-1 mm. On the other side with the very reactive borosilicate glass, the expansions increased steadily with decreasing size. To validate if the acquired expansions were caused by ASR, thin sections were analysed. Mainly, higher expansion values were consistent with extensively more amounts of ASR indicators.

Keywords: alkali-silica reaction, aggregate size, mortar, expansion, microscopy

1 INTRODUCTION

The alkali-silica reaction (ASR) is a harmful expansion process which is responsible for a substantial part of durability problems with cementitious materials. Three components are required to trigger ASR: alkali-sensitive aggregates, alkalis available in the pore solution and moisture. When sufficiently present, the alkali-sensitive siliceous aggregates can be partially dissolved to form a first gel-like hygroscopic alkali-silica reaction product (ASRP). This ASRP needs additional space when it is formed. Furthermore it is able to swell in the presence of humidity, both processes generating an expansion pressure inside the concrete [1-4].

There are numerous factors related to the materials used for the concrete, climatic conditions or the practical use of the structure, which influence ASR. The most important factor is the aggregate, which is the major component in concrete (about 70 % by volume). Its sensitivity regarding ASR depends on its type and origin and it was comprehensively investigated in previous studies. These studies showed that the presence of amorphous, poor crystalline (incl. deformations of lattice) or microcrystalline silica will promote this reaction [5-6].

Beyond that, geometrical factors like aggregate size, pore structure of the grains and also the specimen size are also important [7]. The current publication focuses on the influence of the aggregate grain size on the ASR potential. In a first step, this size effect was investigated for three aggregate types of different alkali sensitivities. The experiments were based on an accelerated mortar bar expansion test and a subsequent petrographic examination. The concept is based on the stepwise substitution of only one single aggregate size fraction by a reactive aggregate type, while the other four aggregate size fractions are composed of the non-reactive aggregate.

Typical aggregates in concrete vary in their grain sizes from fine particles of 0.1 µm (or even less) up to coarse particles with a maximum size of 32 mm (sometimes also 63 mm or more). As a general trend it was described in literature that the problem of ASR-expansion mainly increases with decreasing aggregate sizes [2, 8]. As an example, Hobbs [2] showed this within an aggregate size range of 0.15-13 mm. The expansion of reactive opaline silica aggregates increased significantly as the

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reactive particle size was reduced. By testing reactive chloritic schist, Dunant and Scrivener [9] showed that expansions within the coarse aggregates (4-8 mm and 8-16 mm) followed this general trend, but for small aggregates (0-2 mm and 2-4 mm) the effect reverses.

Such contradictory results discussed in literature [2, 7-14], in particular with regard to the indefinite influence of the small reactive aggregate sizes in the sand fraction, are also the reason why in some testing guidelines for classifying the ASR-sensitivity of aggregate proveniences, the influence of the sand fraction is excluded, by prescribing the use of a non-reactive sand of known origin for the tests [15].

Based on these premises, the aim of ongoing work presented in this study is to investigate systematically, how significant the size of reactive aggregates in the sand fraction (< 4 mm) reveals related to ASR. The authors put forward the hypothesis that there is probably a certain particle size in the area between 0.125-4 mm, where reactive aggregates change their characteristic influence. While the general trend followed by bigger particles means that the contribution to ASR-potential of concrete increases with decreasing particle size, below a certain limiting value this trend inverts. This limit value in aggregate size is expected to be located at different sizes, depending on the reactivity of the type of aggregate.

The hypothesis defined above is supported by the long established knowledge related to the reactivity of pozzolans [16-17]. Strict extrapolation to “aggregates” of very small size (below 100 μm) should allow a reaction of incorporated reactive siliceous material similar to a pozzolanic reaction. This reaction leads to the formation of C-(A)-S-H phases instead of the typical gel-like ASRP and it is well known to mitigate ASR [18-19] instead of promoting it. The better availability of Ca in case of reaction of smaller grains containing reactive silica may be one explanation for the reaction to C-S-H-like products instead of forming expansive ASRP.

It is not possible to distinguish whether C-S-H in hardened cement paste is completely originating from binder reaction, or whether additional phases were formed due to fine aggregate reaction. Other crystalline reaction products resulting from ASRP are easier to prove and mentioned in literature. They are identified as types of zeolites, which are closely related to the layered framework structure of the ASRP as investigated by [20-21].

Only few studies dedicated to the specific effect of aggregate size below 4 mm are discussed in literature. The results were obtained by using different test methods with different special conditions, some of them difficult to compare with more common test procedures of ASTM or RILEM. Also the influence of different specimen sizes, as investigated systematically by Gao et al. [7], makes it difficult to extract general relationships. Some studies indicate that also fine sized reactive aggregate follow the general trend that the expansion increases steadily if the reactive aggregate size is reduced. For example analyses by [10-11] support this assumption in the size range of 0.15-5 mm. The 20 mm x 20 mm x 60 mm specimens of the experiments by Zhang et al. [10] contained reactive quartz glass as aggregate with a w/c of 0.3. They were cured at 100°C for 4 h in steam, autoclaved at 150°C for 6 h in a solution containing 0.2 M KOH and before the length change was measured to calculate expansion. Kuroda et al. [11] analysed 40 mm x 40 mm x 160 mm mortar bars made with non-reactive fine aggregates and reactive bronzite andesite aggregates with a w/c of 0.5 in an autoclave apparatus for 7 days immersed in different NaOH solutions.

In contrast to that, expansion measurements performed by [7, 12-14] obtained limit values of particle size, below which the expansion potential decreases significantly. Lenzner and Ludwig [12] investigated mortar bars of 40 mm x 40 mm x 160 mm with reactive opaline sandstone aggregates. The decline of expansions started below the aggregate size of 0.5 mm. This inversion occurs at the experiments of Multon et al. [13] below a particle size of 0.315 mm. In this study the 20 mm x 20 mm x 160 mm specimens contained a non-reactive marble and a reactive siliceous limestone of only one single size varying in six fractions between 0 and 2.5 mm with a w/c of 0.5 as well as 0.6 and were stored for 500 days at 60°C and 95% relative humidity. Another limiting value for this inversion determined by Ramyar et al. [14] was a size of 0.25 mm. This latter experiment was based on the ASTM C 1260 [22] mortar bar test method with a natural all-in reactive aggregate. The mortar composition was systematically varied, following an aggregate substitution concept, which was applied also in the current work.

2 MATERIALS AND METHODS

2.1 Approach to aggregate size variations

To study the aggregate size dependency, this work focuses on five aggregate size fractions within the sand and fine gravel range of 0.125-4 mm. All mortar samples were produced by using the standard grading curve of [15] as listed in Table 1. This grading curve was used to keep close relation

to real mortar composition and standard testing procedures, although the proportion of the size fraction varies. For an improved interpretation further investigations will additionally include mortars with an invariable proportion of each size fraction (20%).

While using all five aggregate size fractions in each mortar sample, a substitution of single fractions by different reactive aggregate types is the strategy to obtain the relevant variations within the resulting ASR-expansion. This substitution concept is an easy applicable approach to analyse an alkali-reactive aggregate type of a specific size fraction in a mix with a non-reactive aggregate, called “base-aggregate” below. As shown in Table 1, this base-aggregate type (B) is always present in four of the five aggregate size fractions of each mortar sample. The fifth aggregate size fraction is replaced by the reactive aggregate type under investigation (A).

For visualisation, a simplified schema of this mix pattern and the stepwise substitution of the aggregate size fractions are shown in Figure 1. It is easy to imagine how strong the number of reactive aggregates and the total aggregate surface increase with decreasing size, while the volume of aggregates stays constant. In detail, if the aggregate size is divided by two, the number of grains increases by the factor eight, and the total aggregate surface by the factor two..

2.2 Materials and substitution mixtures

Three aggregate types of significant different reactivity were chosen to analyse the aggregate size dependency. As the base-aggregate type, quasi non-reactive quartz sand (QS) was selected. These QS fractions were provided by the Euroquarz GmbH from the Upper Lusatia region in Germany. Additionally two different alkali-sensitive aggregate types were chosen: a slow-late reactive greywacke (GW) from the Sauerland region in Germany and a very reactive borosilicate glass (BG) provided by the Technical Glass Factory of Ilmenau in Germany. This BG is often used in ASR research projects as a reference material, comparable in its reactivity to very alkali-sensitive opaline sandstone [23].

The aggregates of GW and BG had to be crushed and sieved to obtain the required aggregate size fractions of the standard grading curve mentioned above. By using a jaw crusher the aggregates were first ground and then sieved automatically. Afterwards the separated aggregate size fractions were sieved manually under water to remove adhering fine particles from the surface. Finally, the aggregate size fractions were dried in a heating cabinet at 100°C.

The used cement was the alkali-rich standard German test cement (class CEM I 32.5 R) stipulated according to the German ASR-guideline [15] for classifying aggregates concerning their alkali sensitivity. The Na₂O-equivalent of 1.09 wt% was boosted according to the guideline to 1.30 wt% by the addition of potassium sulphate to the mixing water. The complete mortar formula, as proposed in the guideline [15] is shown in Table 2.

The Mortar prisms (40 mm x 40 mm x 160 mm) for the current study were produced according to this formula. Keeping in mind the results of Gao et al. [7] showing that expansion reveals different in samples of other size, this specimens size has the advantage to be not too small, while being used in the most common standard test procedures. The prisms were made with gauge studs at both fronts, which is necessary to measure the length change developments. After production, all specimens were stored for 24 h above water and then demoulded.

The three aggregate types were first tested in mortars without employing the substitution concept. In the following, these test series are called the pure QS, GW or BG specimens. Afterwards six substitution mixtures were produced based on the substitution concept, three substitution mixtures for both reactive aggregate types under investigation (GW and BG). These nine different variations of the aggregate types used in the mortar mixtures are listed in Table 3.

2.3 Methods for assessment and analysis

The procedure of the ASR investigation concerning the aggregate size effect was based on an expansion test for mortar samples to determine the length changes caused by ASR. Subsequently petrographic analyses of selected specimens were performed by using optical microscopy.

2.3.1 Expansion test method

The standard German accelerated mortar bar test [15] was used for this work. One test series consists of three mortar prisms. After demoulding the specimens were stored in metal containers, in which they were fully immersed in cold distilled water, and then put in a heating cabinet for 24 h to heat them up to 80°C. Subsequently the length of the specimens were determined (zero measurement). Afterwards the prisms were placed in a 1 M NaOH solution (pH=14) at 80°C for 13 days. The specimens rested on small bars made of polyvinyl chloride, to ensure that a maximum

specimen surface is in contact with the solution. The amount of solution (either H₂O or NaOH) for three prisms in one metal container was 3.5 litres.

These conditions cause a rapid induction of the deleterious ASR and, due to the high temperature during the aging time, speed up the reaction rate of ASR leading to a very viscous ASRP [24]. The lengths of the specimens were measured approximately daily to obtain possible length changes induced by ASR. When fluid loss happened due to evaporation, distilled water was added to keep the specimens fully immersed. This test method is largely similar to the ASTM C 1260 [22] and RILEM AAR-2 [25]. The only differences are the specimen dimensions and the slightly varied grading curve.

2.3.2 Thin section preparation and microscopy

After completed expansion testing, one specimen of each mortar prisms series was chosen for the thin section preparation. Normally ASR damaged samples show a weak cohesion. Therefore the preparation process is based on the solidification of the sample by using a specific resin. In addition, this prevents the sample from further deterioration during the preparation steps. After drying at moderate temperatures and a vacuum-pressure impregnation the resin penetrates the whole sample and after curing it obtains the sample material in a fixed state. In this way the ASRP, which is very sensitive to leaching and drying, was conserved in the resin with as small as possible alteration. A fluorescent colored pigment was added to the resin, which allowed discerning the finer resin-filled areas in detail by its color and by irradiating with UV light. The whole preparation process was performed completely anhydrous (use of petroleum) to prevent changes of the ASRP.

The images were acquired employing an optical microscope (Zeiss Axioskop 40) using transmitted illumination in plane polarised light and crossed polarised light, or UV light respectively.

3 RESULTS AND DISCUSSION

3.1 Expansion

3.1.1 Expansion of pure specimens with QS, GW and BG

To determine and to compare the general reactivity of the three selected aggregate types QS, GW and BG, pure specimens were investigated, containing always the same aggregate type in all size fractions. The expansion results of these three series are shown in Figure 2.

The specimens made with QS showed the weakest reactivity development, as expected. During and at the end of the ASR-provoking storage in the heating cabinets, these samples showed a constant and slow expansion rise. They reached a final expansion value of 1.4 mm/m after 13 days of immersion in the hot alkali solution. Evaluating this on the basis of the criteria proposed in the different guidelines [15, 22, 25], this means that QS is an appropriate base aggregate for reference. Probably due to the high internal alkali supply the final expansion slightly exceeds the lower limit of 1 mm/m, but it keeps a safe distance to the limit of 2 mm/m, above which a clear indication of potentially deleterious expansion is given.

In contrast to that, the expansions of the specimens made with GW occurred significantly faster. After 5 days of immersion in the hot alkali solution GW showed expansions nearly 3 times higher than QS. The increase of the expansion declined slightly during the second half of the test. The final expansion value of GW was 2.8 mm/m. This clearly indicates that GW is to be characterized as a reactive aggregate of the slow-late type.

BG provided a special case, and the results have to be discussed against the background that this specific accelerated test used in this research is principally not designed to fit for very reactive aggregates. The pure BG mortar prisms reacted extremely rapid and reached 1.2 mm/m already after one day, which is 85% of its final expansion value. This extremely intensive early reaction has to be interpreted as clear evidence for a high ASR susceptibility, as expected. It has to be pointed out that the final expansion value of 1.4 mm/m was identical to the quasi non-reactive QS. This visualizes why the 13day expansion criterion of the used accelerated test is not at all meaningful for assessment of very reactive aggregates. This is known, and therefore fully considered in the guidelines [15, 22, 25]. Also from other literature it is notified that for rapidly reacting aggregates the phenomenon called "pessimism effect" occurs. If a critical content of a reactive aggregate is reached, the highest expansion respectively the most damages occur. The expansion decreases not only, as expectable, by decreasing this aggregate content, but also by increasing it [26-27]. A plausible explanation for this effect is that an aggregate reacting extremely intensive, this creates enough space where products of further reaction can precipitate without forcing expansion, because a major part of the aggregate grains are dissolved.

These results lead to the conclusion that the selected aggregates are appropriate for the current study. The individual aggregate types showed the necessary distinct differences in the magnitude and in the characteristics of their reactivity. The expansion raise with QS was significantly weaker than that with GW and BG. Therefore, the use of QS as the base-aggregate type is suitable for analysing the reactive aggregate size fractions with the described substitution concept.

3.1.2 Expansion of specimens with one substituted size fraction

The expansion results of the specimens prepared with four aggregate size fractions of quasi non-reactive QS and one aggregate size fraction of GW are given in Figure 3. All specimens showed a constant expansion development over the entire testing period. By reducing the aggregate size fraction of GW starting with 2-4 mm, the expansions increased until the maximum expansions were reached for the aggregate size fraction of 0.5-1 mm. The final expansion value for this aggregate size was 2.4 mm/m. This value is above the upper assessment limit of 2 mm/m, and only 12% below the pure GW mortar specimen. The further reduction of the aggregate size of GW leads to smaller expansions. The least reactive aggregate size fraction was the smallest one of 0.125-0.25 mm with a final expansion value of 1.1 mm/m, which was hardly above the lower evaluation limit of 1 mm/m, and even 20% lower than the pure QS mortar specimen. These results support the hypothesis of the authors that there could be a characteristic size of aggregate, below which the characteristic dependence of ASR susceptibility from size changes.

In analogy, Figure 4 shows the expansion results of the substitution mixes with BG. These specimens behaved significantly different than the substitution mixes of QS with GW. By reducing the aggregate size of BG, the expansion values increased steadily. Thus, the smallest aggregate size reached the highest final expansion value of 6.8 mm/m, which is very high, also compared to the upper evaluation limit of 2 mm/m. With 2.9 mm/m even the weakest expansions of the biggest BG aggregate size fraction were still higher than this limit, and also than the expansion of the most reactive GW size fraction.

Figure 5 presents a direct comparison of all final expansions of each GW and BG substitution mixture after the final storing time of 13 days. These data considering three size fractions significantly support the difference in the size dependent expansion, as discussed above. Comparing the expansions of the pure BG specimen mentioned above (Fig. 2) with the samples incorporating only single size fractions of BG (Fig. 4) emphasizes the validity of the pessimum effect, mentioned in 3.1.1. In detail, BG was present in the substitution mixtures with only 10-25 wt%, but showed significantly larger expansions (nearly up to 500% more) than the pure BG (100 wt% BG).

3.2 Microstructure

The pure QS and the pure GW specimen, as well as all three substitution mixtures of QS with GW showed slightly ASR-induced structural changes. ASRP was mainly deposited as circular rims inside the pores (for example Figure 6a), but none completely with ASRP filled pores were present. In a few cases the ASRP was observed in cracks starting from GW aggregates and penetrating through the cement paste (as an example Figure 6b). Almost all aggregates (QS and GW) showed no signs of dissolution effects.

The pure BG specimen as well as all three substitution mixtures of QS with BG showed substantial deleterious features in their structure. The ASRP was extensively located inside the specimens. Many pores were filled partly, and also some of them completely, by the ASRP (for example Figure 7a). In addition, the numbers of cracks were much higher than the specimens containing GW. Widespread cracks penetrated from the BG aggregates through the cement paste. The affected BG aggregates were partly or even fully dissolved (for example Figure 7b).

In all specimens the ASRP was detected as primary cause of the expansions and structural damages. It showed characteristic shrinkage cracks, which are typically caused by the drying during the sample preparation. Fortunately, no signs of ettringite were found, which often occurs along with ASR. With the absence of ettringite, a potential other cause of the expansions can be excluded.

These petrographic results are completely consistent with the expansion development of the mortar prisms. Generally the more expansions occurred, the higher the extent of the ASR-induced structural changes. Also the special case of the pure specimen with BG, where the high reactivity did not match to the expansion development, can be explained with these microscopic observations. The images reveal plenty of available space, created by dissolving processes in the aggregate grains. This offers more than enough room to incorporate the ASRP formed.

4 CONCLUSIONS

The present study aimed to investigate the effect of different aggregate sizes on ASR expansion. The basic assumption was that at a certain particle size in the range of 0.125-4 mm, reactive aggregates could change their characteristic influence in mitigating ASR instead of promoting it. Three aggregate types of different reactivity were selected and separated in five aggregate size fractions. A stepwise substitution concept was performed, based on the mixture with quasi non-reactive sand. For the experiments an accelerated expansion test at a temperature of 80°C for mortar bars of 40 mm x 40 mm x 160 mm was applied. After expansion, petrographic thin sections were analysed, to identify ASR indicators as the primary cause of the expansions. The following conclusions were obtained:

1. The three selected aggregates proved to have the desired differences in ASR susceptibility for the performance of this research. They showed quasi non-reactive properties (QS), slow-late properties (GW) or intensive reaction (BG), when only one type of aggregate was present in the mortars.
2. The substitution concept for preparing the mortar mixtures turned out as an appropriate approach to analyse and compare the contribution of different aggregate size fractions to ASR expansion.
 - The influence of the high reactive BG aggregate size fractions followed the generally expected trend: the smaller the aggregate size, the higher the expansion due to ASR.
 - The slow-late reactive GW followed this general trend only above a size fraction of 0.5-1 mm. Smaller size fractions led to a decrease of the acquired expansions. These results support the hypothesis of the authors that below a specific size, a reactive aggregate mitigates the ASR risk, instead of promoting it.
3. By petrographic analysis, reaction products (ASRP) were observed in all mortars. Consistent to the higher expansion values, BG showed more amounts of the ASRP and cracks in comparison to GW. Additionally, its fast and intensive reaction is obviously associated with the appearance of partial dissolved aggregates. In case of the specimen with pure BG in all size fractions the dissolution was actually so high, that it is easy to imagine that enough space is available for any ASRP to be formed. This explains the result that further reaction does not result in further expansion in case of high amounts of very reactive aggregate.

Based on these results, the aggregate size exerts a significant influence on the damage potential of ASR. By reducing the aggregate size, both mitigation as well as promoting effects were documented, depending on the chosen aggregate type. Further research on other types of reactive aggregates, the missing intermediate size fractions, additional finer size fractions and other proportions is considered, to gain a better generalization. Fundamental researches of the reaction mechanism as well as the time dependent and locally varying chemical composition of the ASRP, and their distribution in the microstructure are expected to provide supporting findings. Further studies will also include dissolution tests to analyse the solubility properties related to SiO₂, CaO, NaOH and KOH.

5 REFERENCES

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TABLE 1: Standard Grading Curve [14] and the visualization of the substitution concept.

[mm]	[g]	[wt%]	stepwise substitution of only one single aggregate size fraction by a reactive aggregate type		
			A	B	B
0.125–0.25	168.75	15	A	B	B
0.25–0.5	281.25	25	B	B	B
0.5–1	281.25	25	B	A	B
1–2	281.25	25	B	B	B
2–4	112.50	10	B	B	A

A: reactive aggregate type under investigation B: base-aggregate type

TABLE 2: Formula for the production of mortar prisms with high internal alkali supply for accelerated ASR tests (three prisms of 40 mm x 40 mm x 160 mm) [14].

	[g]	[wt%]
CEM I 32,5 R	500	26.84
K ₂ SO ₄	2.75	0.15
Water	235	12.62
Aggregates	1125	60.39
Sum	1862.75	100
w/c = 0.47	a/c = 2.25	

TABLE 3: Variation of the aggregate types used in the mortar mixtures.

[mm]	pure			substitution mixtures					
	1	2	3	4	5	6	7	8	9
0.125–0.25	QS	GW	BG	GW	QS	QS	BG	QS	QS
0.25–0.5	QS	GW	BG	QS	QS	QS	QS	QS	QS
0.5–1	QS	GW	BG	QS	GW	QS	QS	BG	QS
1–2	QS	GW	BG	QS	QS	QS	QS	QS	QS
2–4	QS	GW	BG	QS	QS	GW	QS	QS	BG

QS: quartz sand GW: greywacke BG: borosilicate glass

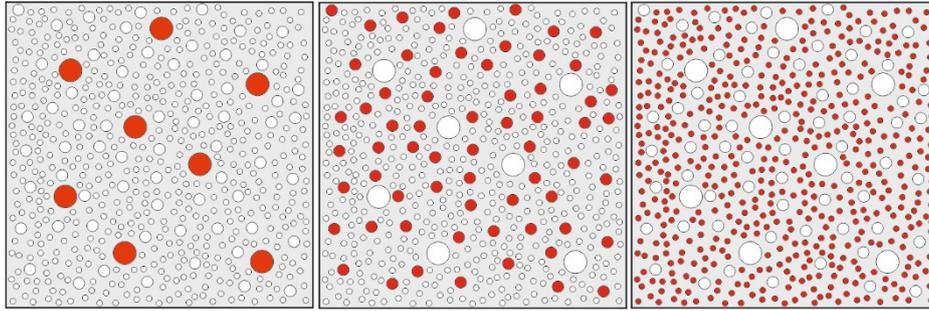


FIGURE 1: Schema of the simplified mix pattern with a stepwise substitution of single aggregate size fractions by using two aggregate types. White dots indicate the non-reactive base-aggregate type and red dots the reactive aggregate type under investigation.

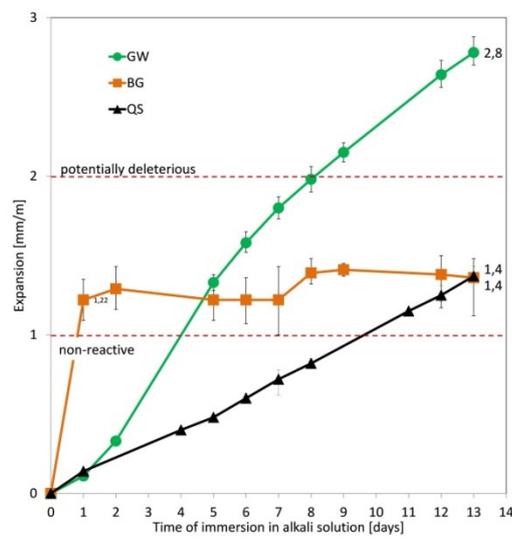


FIGURE 2: Results of the standard German accelerated mortar bar test of pure mortar specimens with QS, GW and BG.

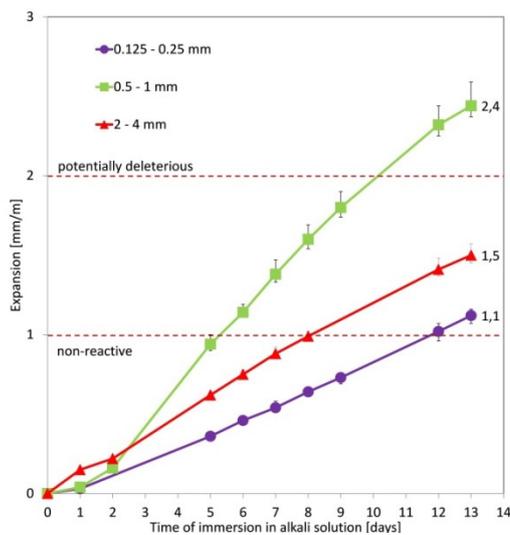


FIGURE 3: Results of the standard German accelerated mortar bar test of the GW mortar substitution mixtures 4, 6 and 8.

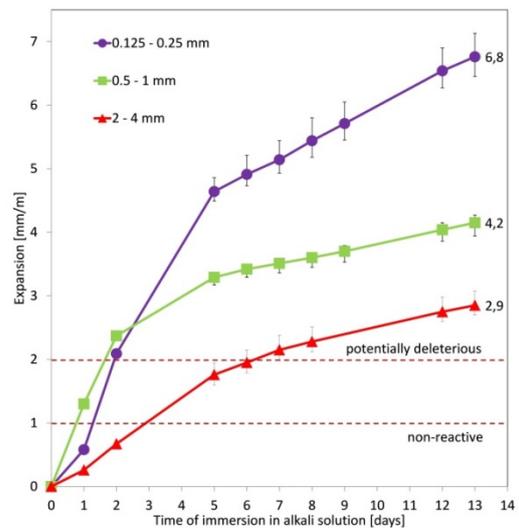


FIGURE 4: Results of the standard German accelerated mortar bar test of the BG mortar substitution mixtures 9, 11 and 13.

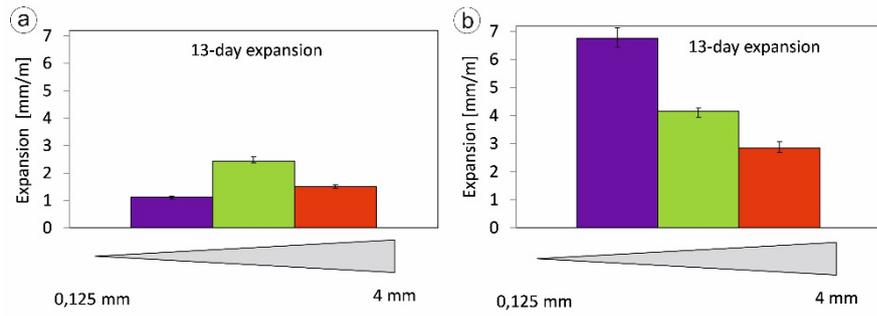


FIGURE 5: Comparison of the ASR-expansions of all GW mortar substitution mixtures 4 to 8 (a) and all BG mortar substitution mixtures 9 to 13 (b).

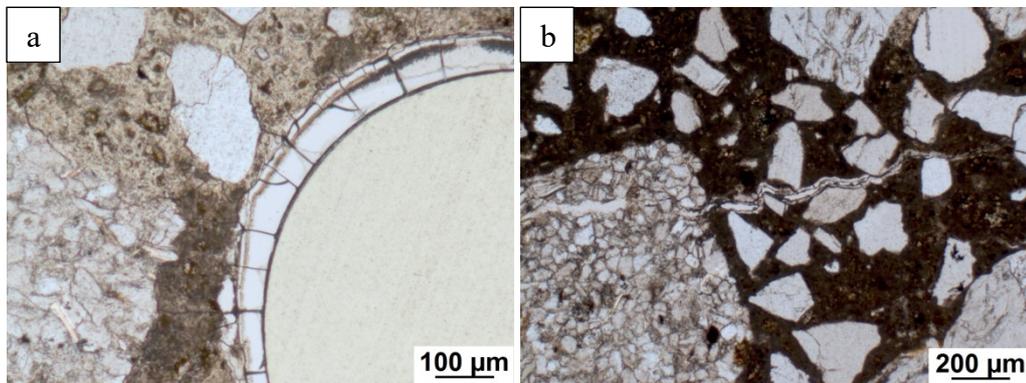


FIGURE 6: Microscopic images of the ASRP in the GW substitution mixtures (plane-polarised light). (a) The ASRP was mostly deposited as circular rims at the pores wall. (b) Rarely cracks were partly filled up with the ASRP and penetrated from the GW aggregates through the cement paste.

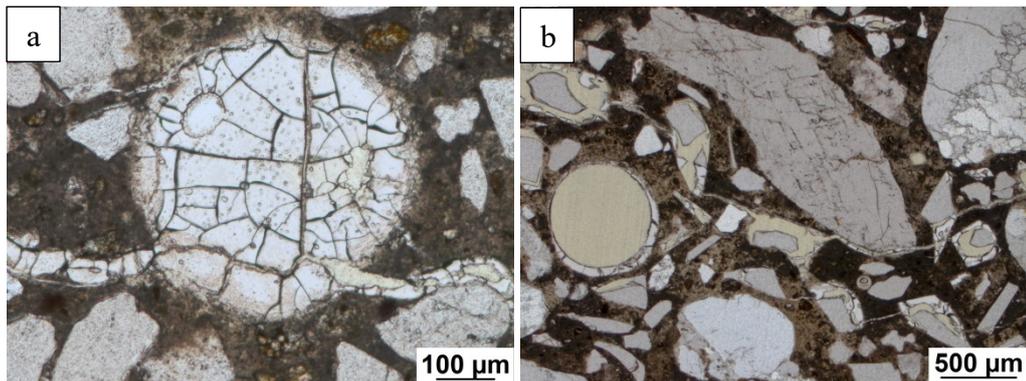


FIGURE 7: Microscopic images of the ASRP in the BG substitution mixtures (plane-polarised light). (a) Completely with ASRP filled pore as a common feature for all specimens containing BG. (b) Extensive ASR affected aggregates with abundant dissolved BG aggregates and several cracks, which are partly filled with the ASRP.