CONRETE TESTS FOR ASR ASSESSMENT: EFFECTS OF TESTING ENVIRONMENT ON PRECONDITIONS FOR AN ASR AND TRANSFERABILITY OF TEST RESULTS

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Abstract

On the basis of tests originally developed for testing aggregate's sensitivity to ASR in the last 20 years more and more test series have been performed to show the capability of cementitious materials to control a deleterious ASR. At the 12th ICAAR conference it was shown, that the test results might be misleading in the sense that a deleterious ASR does not occur although cracking and expansion is observed under field conditions [1].

The investigations presented here deal with the pre-conditions for an ASR during the induction phase and the behaviour of the reaction product in the damaging phase. It is shown that under test conditions both can be affected in a way that a deleterious ASR is less likely than under normal conditions. The type of cementitious material in combination with temperature and humidity determine the risk of getting wrong results .

Keywords: ASR-testing, cementitious materials, transferability, pore solution, gel properties

1 INTRODUCTION

1.1 Discrepancy of concrete behaviour stored outside and under laboratory conditions

Test methods for the assessment of aggregates' reactivity are known since several decades. Starting from mortar tests it is broadly accepted nowadays that concrete test methods like the ones according to RILEM AAR-3/4 give the most reliable results. Transferability to practice is guaranteed due to calibration against aggregates' behaviour in existing concrete structures. Reaction acceleration is reached by increased temperature and humidity as well as high concrete pore solution alkalinity.

In the past two decades some of the test procedures have been used for assessing the effectiveness of supplementary cementitious materials (SCM) to control a deleterious ASR. A calibration of these methods against practical behaviour is more difficult in this case. SCMs in cement and concrete are only used for about 25 years. Additionally the occurrence of ASR damages is most probably retarded compared to OPC concrete [1]. All in all long-term experience, in the sense that damages at concrete structures with pozzolana could be observed, is hardly available. On top of that results obtained by parallel testing of the same SCM containing concrete under laboratory and practical conditions unfortunately indicate, that the transferability of laboratory results might not be given [2, 3, 4, 5]. Expansion and cracking occurred at specimen stored outside, whereas those stored under accelerating conditions remained integer. The combination of opaline sandstone/flint and SCM appears to be specifically critical. This situation is not acceptable, particularly because a possible ASR in practice would not be detected by laboratory testing. Thus, there is a need to understand the reasons and interactions leading to the discrepancy and to consider the findings for future developments of more appropriate testing procedures, especially with regard to SCM usage in cement in order to control an ASR.

1.2 Investigation approach

There are two possible reasons for the discrepancy: Either expansion and cracking outside have been induced by other mechanisms than an ASR, or the storage conditions originally defined for testing aggregates' reactivity do prevent a deleterious ASR rather than supporting it when SCMs are used. The first option is rather unlikely, firstly because of the choice of standardized materials (limited free lime and brucite content) preventing damages from crystal formation and secondly due to a concrete composition (low w/c ratio) which, related to standard requirements (EN 206), should be resistant to freeze thaw attack even without artificial air entrained. Consequently the investigations

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presented here are focused on how laboratory testing conditions may lead to the avoidance of a potentially deleterious ASR, if SCMs are used.

A literature review on this revealed only qualitative indications. Quantitative data was hardly available. Nonetheless the following conclusions could be drawn:

1. Leaching as reported for several test methods can reduce the alkalinity of the pore solution in concrete specimen exposed to humid environment (above or under water) [6, 7, 8]
2. Possibly a simultaneous water uptake of specimen further reduces the alkalinity [1].
3. Reaction of SCMs can be more enhanced at higher temperature than an ASR resulting in a shift towards higher likelihood of damage-preventing interactions like decreasing alkalinity, refining pore size distribution and diminishing $Ca(OH)_2$ availability [9, 10, 11, 12].
4. Finally, due to an accelerated water uptake of the reaction product (gel) when stored under humid conditions and at elevated temperature its viscosity might be affected in a way, that the gel is flowing into the surrounding porous structure without creating damaging pressure [13, 14, 15].

In order to get a clearer picture these four items were addressed in an in-depth study. Therefore items 1 to 3 were dedicated to the induction period of a deleterious ASR. Here it was tested how the pre-conditions for an ASR are influenced by environmental conditions when SCMs are used (reaction kinetics, alkalinity of pore solution, precipitated $Ca(OH)_2$ in paste, pore structure). Item 4 is more related to the damaging period. Here in dependence of environmental conditions it was investigated which characteristics of ASR-gel influence the creation of structure damaging internal pressure leading to cracking and expansion.

2 MATERIALS AND METHODS

2.1 General

For the results presented hereafter related to the induction period concrete specimen and pure cement pastes of comparable w/c ratio have been produced. To see the different effects of humidity and raised temperature concrete specimens of the same batch were stored one time exposed to both, humidity and temperature, and one time shrink-wrapped in 0.1 mm plastic foil only to temperature (marked by suffix -K). Concretes were produced in a laboratory mixer. Total mixing time after addition of all materials was 2.5 minutes. Cement pastes were made according to EN 196-3.

In order to investigate the possible effect of ASR gel in a confined space in a porous media (see item 4) mortar bar prisms were produced comparable to EN 196-1. After demoulding they were stored under water at 20 °C for 21 d and for further 10 d at 65 % RH to let them dry out. Final preparation of the prisms according to FIGURE 1 was done 32 d after prism production. Comparable porosity of the 1.5 g water glass sample inside the holes was guaranteed by compacting it with a mini proctor hammer (200 g falling mass, 100 mm falling height) with a bottom end accommodated to the drilled hole.

2.2 Materials

2.2.1 Cementitious materials

The industrially produced cements and SCMs used in the trials are listed in TABLE 1. Main characteristics determined according to EN 196 test series are given there. All materials complied with the requirements of EN 197-1. CEM I (1.3) had a high alkali content of 1.29 % by mass providing a high initial potential for a deleterious ASR.

Three further cements were produced by mixing the cement CEM I (1.3) with fly ash V, ground slag (S) or silica fume (D), respectively. The mixes contained either 20 % fly ash/slag or 10 % silica fume. Thus, these cements could be designated as CEM II/A-V, CEM II/A-S and CEM II/A-D according to EN 197-1. In order to guarantee a homogeneous distribution of silica fume it was added as a slurry directly to the concrete mix.

2.2.2 Aggregate

For the concrete tests presented here exclusively inert limestone in the fractions 0/2, 2/8 and 8/16 mm with a density of 2.66 Mg/m³ has been used. The grading curve was composed to meet the requirements of a standard grading curve B/C 16 according to DIN 1045-2:2001-07. For the mortar prisms Rhine river sand 0/2 mm from the Düsseldorf region was chosen.

2.2.3 Chemicals

As model substances for ASR-gel five different industrially produced ground solid water glasses of comparable particle size distributions provided by Cognis Silicates have been used. The different compositions were chosen in order to assess the effect of different alkali c ontents in the reaction product e.g. resulting from alkali addition as prescribed in some test methods. The compositions of the water glasses are shown in TABLE 2. They were in the range of the values given in [16, 17].

2.3 Concrete and mortar composition

All concretes were produced with 500 kg/m³ cement and a w/c ratio of 0.55. This relatively unusual composition was necessary to achieve an adequate amount of pore solution to be squeezed out up to 91 d. Moreover this is in line with German regulations for testing effectiveness of newly developed LA cements and existing concretes showing discrepant behaviour.

The mortar composition was according to EN 196-1. Cement CEM I-NA was used to minimize an influence of alkalis diffusing from the mortar to the water glass.

2.4 Storage conditions and concrete specimen size

The concrete specimen (shrink-wrapped and unsealed) were stored after demoulding in three different ways: firstly according to RILEM AAR-4 (60 °C above water in a stainless steel container), secondly according to the German Alkali Guideline in a 40 °C fog chamber and thirdly according to a newly developed regime (FIGURE 2) reflecting an outside storage as performed at the research institute of the German Cement Works Association (VDZ). They are designated in the following by Ü60, NK40 and SAL. Either cubes of 100 mm side length (NK40, SAL) or prisms of dimensions 70 x 70 x 140 mm (Ü60) were used. Since the surface ratio to volume is comparable, a significant size effect on possible leaching can be neglected. Pure cement pastes have been filled in polystyrene tubes, moved for 24 h to avoid segregation and stored afterwards in accordance with the regimes described before.

Supplementary to the Ü60 storage prisms have been stored above water at 20 °C (Ü20) and 40 °C (Ü40). This allowed investigating the behaviour of specimen filled with different water glasses in dependence of temperature. For checking the influence of humidity conditions the specimen have been stored at 20 °C in a fog chamber (RH > 100 %), above water (RH ≈ 100 %) and above saturated solutions of KBr (RH ≈ 91 %) and BaCl (RH ≈ 82 %).

2.5 Methods for assessment and analysis

2.5.1 Induction period

Water uptake

To clarify the influence of water uptake in dependence of the storage and the cement type the unsealed specimens were weighed periodically. The determined water uptake (difference to weight after demoulding) was then used to calculate a dilution factor V_{PL} of the pore solution, which is the ratio of specific free water content to specific free water content plus water uptake. The specific free water was defined as the difference of total water and chemically bound water by CEM I phases calculated acc. to [18].

Leaching and pore solution alkalinity

In order to evaluate leaching either out of the water bath below the specimen or out of the water dropped from the specimen into a stainless basin (fog chamber) a sample was taken and the total water quantity was gravimetrically determined. Aiming at following the composition of concrete pore solution this was squeezed out according to the method introduced in [19]. Up to further treatment the samples were filtered (0.45 µm) and filled in a polypropylene tube Ar-filled to avoid carbonation. Both, aqueous solution and pore solution were analyzed for its alkali content by ionic chromatography. Calibration of the DIONEX DX 500 system for concentrations of 1 to 50 mg/l made it necessary to dilute the samples by a factor of 100 and 1000, respectively. In order to minimize inaccuracy by dilution the ion concentrations were determined at both dilution levels.

To assess the impact of leaching the potentially effective amount of K in the specimen (K_{pot}) were calculated by referring the difference between K introduced to the concrete specimen by the CEM I cement and the leached amount of K (accumulated product of determined K^+-concentration and collected water) to the amount introduced by the CEM I cement alone. Na was not considered for the calculation, because a significant pollution of spray water in the fog chamber had been detected in some pre-tests.

To evaluate the alkalinity of the pore solution (K^++Na^+) just the values determined were used.

Development of $Ca(OH)_2$

The pure cement pastes were used to follow $Ca(OH)_2$ development. At the time of interest hydration in the neat cement pastes was stopped by grinding the samples to analytical fineness (< 90 µm) in acetone, which was then removed by a vacuum pump. Afterwards the sample was rinsed three times with diethyl ether. The remaining filter cake was ground with a pestle. Finally the sample was analyzed by simultaneous TGA and DTA with a Bähr STA 501 device. Therefore an 80 mg sample was heated up to 1000 °C (heating rate 10 K/min) in a N_2-atmosphere. The temperature of 105 °C was kept for one hour to allow evaporation of remaining diethyl ether. The $Ca(OH)_2$ content in the sample was determined from the signal between 450 °C and 600 °C as described in [20]. Figures discussed are related to the ignited cement sample.

Pore size distribution

Samples for the determination of the pore size distribution were taken from the concrete specimen to be squeezed out. Coarse aggregate particles were manually eliminated and the remaining mortar portion was crushed and dried at 10^{-3} mbar above silica-gel for 24 h. Afterwards the pore size distribution was determined at 20 °C with a Fision Instruments Porosimeter 2000 WS by injection of mercury with an pressure up to 200 MPa. Thereby the pore opening diameter recovered was of inverse relation to the pressure applied. By the Washburn-equation and the assumption of cylindrical pores the pore radii can be calculated. The angle between mortar matrix and mercury was assumed to be 141.3° and the mercury surface tension to be 0.48 MPa.

All the aforementioned investigations were performed until a concrete age of 91 d.

2.5.2 Damaging period

While stored under different conditions the length change of the prisms was measured and expansion referred to the initial distance between the reference studs was calculated as mean value of the measurements at each side face up to an age of about 180 d. To eliminate the temperature effect those prisms stored at temperatures different from 20 °C were allowed to adopt for 4 hrs. In addition to that exudations and cracks were recorded.

3 RESULTS

3.1 Introduction period

3.1.1 Water uptake and leaching

With regard to water uptake no considerable differences could be seen in dependence of the cement type used. However, related to the different storages it became obvious, that the concrete pore solution is diluted down to about 90 % of its specific concentration by water uptake if stored in a fog chamber (first 7 d for storage SAL, continuously for storage NK40). During the following range of exposures described in FIGURE 2 for storage SAL the dilution was reduced (specimen dried out) and reached a value around 100 % of its original concentration after 90 d. For storage Ü60 it was observed, that right after the storage in the stainless steel containers the specimen show a slight tendency to dry out, which means an increased pore solution concentration based on water balance. From 28 d onwards the dilution factor was about 100 % and remained almost constant in the following.

Concerning the effect of leaching the situation is different. Here during all storages a significant decrease in the potentially effective K-content (K_{pot}) in the concrete specimens was observed (FIGURE 3), which means that leaching occurred. Even during the simulated outside storage about 15 % of the K introduced by the clinker portion disappeared after 90 d, a portion of roughly 5 % within the first 7 d. At the same time there were no considerable differences in dependence of the cement type. This changed for storage NK40 and Ü60. Whereas K_{pot} for the CEM II/A-D and CEM II/A-V was about the same range as mentioned before, after 91 d the loss was about 41 % (NK40) and 37 % (Ü60), respectively, for the concrete specimens made of CEM I cement. For the specimens with CEM II/A-S a loss of about 30 % could be assumed, if the temporary defect of the vaporizer is neglected (actually K_{pot} was 20 % less than initially introduced).

3.1.2 Alkali concentration of concrete pore solution

For concrete with CEM I (reference) it was observed that for storage SAL-K and NK40-K after 28 d the same level of alkalis in the pore solution was achieved (app. 700 mmol/l). Before, the alkali concentration was slightly smaller at lower temperature (SAL). For the specimens stored at 60 °C (Ü60-K) a constantly less alkali concentration was determined ($D \approx 50$ mmol/l).

For concrete produced with CEM II/A-S comparable alkali concentration evolutions were observed. Even the constantly less alkali content at 60 °C could not be confirmed. In contrast big differences became obvious, when cement with pozzolana was used (FIGURE 4). Compared to the simulated outside storage (SAL-K) a strongly decreased alkali concentration was noticed during storage NK40 and Ü60, i.e. with increased temperature. The maximum difference was about 200 and 350 mmol/l for the concretes with CEM II/A-V and CEM II/A-D, respectively. It occurred 28 d (CEM II/A-V) and 3 d to 7 d (CEM II/A-D) after concrete production. Finally, after 56 d and 28 d the alkali concentration of the pore solution reached a comparable level.

3.1.3 $Ca(OH)_2$-content in cement paste

The results are quite analogous to those with regard to the alkali concentration of the pore solution. Whereas the differences were just slight and dependent on temperature for CEM I paste at early ages (higher values at higher temperature) and absolutely comparable for CEM II/A-S pastes, considerable differences in $Ca(OH)_2$ content were detected, if cement with pozzolana was used. Again compared to the simulated outside storage, the $Ca(OH)_2$ content in the fly ash cement pastes (see FIGURE 4) was clearly lower after 28 d and 3 d, if stored according to storage NK40-K and Ü60-K, respectively. The maximum difference was about 1/4 (NK40) and 1/3 (Ü60) of the maximum value reached during storage SAL-K. For cement with silica fume the effects were even more pronounced. Already after three days the $Ca(OH)_2$ content was, compared to the values during storage SAL-K, about 25 % and 50 % less than under NK40-K and Ü60-K storage conditions. Finally it seems as if again comparable final levels are achieved. Whereas this was already the case for the CEM II/A-D pastes after 91 d (6 g/100g cement), for the CEM II/A-V cement paste this only can be an assumed.

3.1.4 Pore size distribution

In FIGURE 5 the characteristic values derived from the measured pore size distributions are shown. It can be seen that after 7 d storage the capillary pore volume > 0.01 μm (relevant for transport phenomena) was smaller the higher the temperature was (conserved storage). When CEM I (1,3) and CEM II/A-V were used, the biggest drop occurred between storage NK40-K and Ü60-K. The difference was about 4 to 5 % by volume. For the slag and silica fume containing cements the biggest drop is shown between the storages SAL-K and NK40-K with a maximum difference of more than 5 % for CEM II/A-D cement at the age of 7 d. In contrast to this the capillary pore volume after 91 d was almost of the same range. For CEM II/A-S and CEM II/A-D even a slight increase with higher temperature could have been detected.

The median radii, also plotted in FIGURE 5, support the observations above. The data indicate that the capillary pore system was much finer at higher storage temperatures after 7 d. The biggest refinement can be observed, when CEM II/A-V cement was used. Whereas for the temperature regime SAL-K adopted to practical conditions a median radius of about $45 \cdot 10^{-3}$ μm was detected, it was reduced to $35 \cdot 10^{-3}$ to $17 \cdot 10^{-3}$ μm for storage NK40-K and Ü60-K, respectively. In addition again after 91 d the situation was almost comparable for all temperature regimes with a median radius constantly below $20 \cdot 10^{-3}$ μm.

3.1.5 Reaction kinetics

On the basis of pore solution alkalinity of concrete with non-reactive limestone as aggregate and the $Ca(OH)_2$ content of cement paste the starting point of supplementary cementitious materials could be determined by comparison with OPC concrete and paste, respectively. Moreover for one quickly reactive aggregate (gravel with opaline sandstone and flint) and for one slowly reactive aggregate (chippings with silicified limestone) the reaction start in OPC concrete could be estimated (more detailed information on this can be found in [1]).

In FIGURE 6 the latter ones are compared to the previous ones. It can be seen that, related to the reaction start of the fast reactive aggregate, the difference to the reaction start of the SCMs was less the higher the storage temperature was. Whereas during storage SAL-K and NK40-K the reaction started after an ASR it began in parallel during storage Ü60-K. Compared to the slow reactive aggregate the situation was different. Slag and silica fume reacted always earlier than the ASR began. Fly ash started to react in parallel with an ASR with the late reactive aggregate during storage SAL-K and earlier during storage NK40-K and Ü60-K, i.e. at higher temperature.

3.2 Damaging period – expansion of mortar prisms

The maximum expansion achieved by the prisms prepared with water glass is shown in FIGURE 7. The relation to different parameters is plotted in a way that the specific property of the water glass to take up water is increasing from the left to the right side.

The graph on the left of FIGURE 7 shows, that with increasing relative humidity in the air the maximum expansion had the tendency to increase linearly. In contrast it can be derived from the middle and the right graph, that the maximum expansion was linearly decreasing, when the temperature or the alkali content of the water glass had been raised. In parallel the occurrence of exudations was increasing for these cases. Whereas at low temperature/alkali content only seldom distinct and solid exudations became visible on the prisms surface, the prisms at higher temperature or incorporation of high alkali water glass looked like impregnated with water glasses in extreme cases. In addition it is interesting to see in these cases that significant differences between prisms filled with different water glasses or exposed to different temperatures became smaller the higher the storage temperature/alkali content in the water glass had been. Finally the maximum expansions achieved were quite in the same low range but still keeping the order.

4 DISCUSSION

The availability of a certain amount of reactive silica, a concrete pore solution alkalinity sufficient to induce an ASR and moist conditions to allow appropriate swelling of the gel are broadly accepted as major pre-conditions for a deleterious ASR. Moreover a supporting influence of $Ca(OH)_2$ usually available in excess around aggregates was shown. Finally differences in the paste structure influencing the resistance to any transport mechanism are likely to impact the damage. Keeping this in mind the results presented show clearly, that the pre-conditions for an ASR during the storage according to the German Alkali-Guideline (NK40) and RILEM AAR-4 (Ü60) are not necessarily as severe as assumed, if compared to more practical conditions (SAL-K).

Extremely humid conditions like in a fog chamber can firstly lead to a dilution of the pore solution by water uptake of the specimen especially at early ages (independent of cement type). Vacuum pressure created by cement hydration can be seen as a reason for that. Secondly and on top of that under such conditions intensive leaching occurs. A loss of 20 % of its initial soluble alkali content after 28 d and more than 30 % after 90 d is possible. Concrete made of OPC or cement with slag (20 %) seems to be particularly vulnerable. Storage above water appears to be more sufficient in this respect for early ages up to 28 d (Ü60). But for later ages, when the water balance is achieved in the storage containers, leaching reaches comparable ranges (30 %). This is in line with observations in [8]. Considerable water uptake should not occur during this storage.

Related to the exposition to elevated temperatures concrete specimen containing pozzolana are especially prone to show differences. Due to an accelerated reaction and thereby activated capacity to bind alkalis these are rapidly extracted from the pore solution. Correspondingly also the $Ca(OH)_2$ is remarkably reduced. Although shown for pure paste this should also count for the transition zone. Finally the pore structure in the concrete is different at early ages with considerably less capillary pore volume relevant for transport of alkalis and water to the reaction site at higher temperature. All in all it must be concluded, that these are phenomena, which could explain the discrepancy highlighted in the introduction. Against the original target they reduce the likelihood that an ASR occurs under "accelerating" storage conditions.

Besides the refined pore structure hindering a water uptake of the gel the phenomena described before especially affect the preconditions for an ASR (induction period). Whether they become really relevant, depends also on the velocity of reaction between alkalis and reactive aggregate. FIGURE 6 gives an idea about the relations depending on the type of reactive aggregate. For slow reacting aggregates the usage of silica fume would certainly work in this respect, even under practical conditions it reacts quicker than the aggregate and its beneficial way of action could inhibit an ASR. But fly ash is already critical. Its reaction starts parallel to the reaction of the aggregate. Thus, if an ASR shall be avoided with fly ash in combination with this late reactive aggregate, it can probably only be guaranteed by dilution of the pore solution (no water binding by fly ash, reduced alkali release compared to cement clinker). If it comes to a fast reactive aggregate it is in anyway doubtful that an ASR can be avoided by alkali binding. The ASR always starts before or in parallel to the pozzolanic reaction. How it comes, that concretes with these aggregates show no significant expansion during the laboratory trials, can be derived from the prism tests related to the damaging period.

The idea behind these investigations was that a damaging internal pressure is created, if the reaction product (here the water glass) increasing its volume by water uptake is not able to penetrate into the surrounding porous media [14]. Thus the viscosity of the reaction product plays also an essential role. Increased indirect (via mortar) water availability obviously leads to a bigger volume increase and results in damaging pressure. Although the viscosity decreases with increasing water content the reaction product is not that sufficiently deformable to enter the paste structure. This changes as soon as due to an increased temperature or increased alkali content the specific viscosity of the reaction product decreases. With increasing tendency the reaction product might then penetrate into the porous structure, so that the damaging pressure and thereby the deterioration decreases. A decreasing maximum expansion of test specimen at higher temperature as reported in the literature is an indication of this effect. In extreme cases, e.g. if a quickly reactive porous aggregate in combination with pozzolana/slag is tested, even the expansion can totally disappear. Here on top of the impact on viscosity of the gel it can be expected, that due to less available $Ca(OH)_2$ in the transition zone the characteristic reaction rim preventing in addition the gel penetrating into the surrounding paste is less solid.

5 CONCLUSIONS

The investigations and results presented before clearly have confirmed the qualitatively identified phenomena to explain the misleading behaviour of concrete specimen under laboratory conditions by quantitative data. Humid conditions and high temperature can decrease the likelihood for an ASR compared to more realistic conditions by various factors. Their impact especially on the alkalinity of the pore solution as well as on the behaviour of the reaction product – the gel – determine whether an ASR takes place and, even when it has taken place, whether a deterioration of the structure occurs.

In order to raise the reliability of test methods the phenomena described in the text have to be considered for the future. Appropriate storage scenarios must be developed especially for testing the effectiveness of cementitious materials. One appropriate measure would be to store the concrete under ambient temperature to allow a normal development of pore solution composition and paste structure for a certain time. Then also the conditions for an ASR would be reflected correctly, because it can be assumed, that it takes place within 28 d to 56 d, when maximum alkalinity in the pore solution usually is achieved. In order to accelerate a possible damaging process afterwards storage under wet conditions seems to be a good option (leaching is of minor interest, when an ASR has already occurred). Increased temperature still would bear the risk of too fluent gel. But probably this has to be kept in order to get an acceptable testing duration. More sensitive limits for evaluation or a structured approach for the evaluation of exudations could be worth thinking of in this respect.

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7 REFERENCES

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[1] Bokern, J.: Betonprüfungen zur Beurteilung einer AKR: Auswirkung der Prüfbedingungen auf die Voraussetzungen für eine AKR und die Übertragbarkeit der Prüfergebnisse. Phd-thesis in preparation, 2007.

[2] Bokern, J.; Siebel, E.: Alkali-Silica Reaction in Germany – Transfer of Laboratory Results to Practice. In: Proceedings of the 12th International Conference on Alkali-Aggregate Reaction in Concrete, Vol. 1, 15-19 October 2004, Beijing, China, S. 490-498.

[3] Fournier, B.; Nkinamubanzi, P.-C.; Chevrier, R.: Comparative field and laboratory investigations on the use of supplementary cementing materials to control alkali-silica reaction in concrete. In: Proceedings of the 12th International Conference on Alkali-Aggregate Reaction in Concrete, Vol. 1, 15-19 October 2004, Beijing, China, S. 528-537.

[4] Oberholster, R.E.: Alkali-aggregate reaction in South Africa: Some recent developments in research. In: Proceedings 8th International Conference on Alkali-Aggregate Reaction, Kyoto, Japan, 1989, S. 77-82.

[5] Siebel, E.; Sylla, H.-M.; Bokern, J.: Einfluss von puzzolanischen Betonzusatzstoffen auf die Vermeidung einer schädigenden Alkali-Kieselsäure-Reaktion. In: Tagungsbericht 14. Internationale Baustofftagung, F.A. Finger-Institut für Baustoffkunde (Hrsg.), Weimar, 2000, Band 1, S.959-967.

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[6] Bakker, R.F.M: The influence of test specimen dimensions on the expansion of alkali reactive aggregate in concrete. In: Alkalis in concrete – Research and Practice, Proceedings 6th International Conference, Copenhagen, 1983, S. 369-375.

[7] Siebel, E.; Reschke, T.: Alkali-Reaktion mit Zuschlägen aus dem südlichen Bereich der neuen Bundesländer – Untersuchungen an Laborbetonen. In: beton 46 (1996), Heft 12, S. 740-744 und beton 47 (1997), Heft 1, S. 26-32.

[8] Thomas, M.D.A.; Fournier, B.; Folliard, K.; Ideker, J; Shehata, M.: Test methods for evaluating preventive measures for controlling expansion due to alkali-silica reaction in concrete. In: Cement and Concrete Research 36 (2006), S. 1842-1856.

[9] Barneyback, R.S.: Alkali-silica reactions in Portland cement concrete. Purdue University, Dissertation, 1983.

[10] Hobbs, D.W.: Deleterious alkali-silica reactivity in the laboratory and under field conditions. In: Magazine of Concrete Research 45 (1993), Heft 163, Juni, S. 103-112.

[11] Battagin, A.F.: Influence of degree of hydration of slag on slag cements. In: 9th International Conference on the Chemistry of Cement, Vol. III – Communication Papers – Theme II, National Council for Cement and Building Materials, New Delhi, 1992, S. 166-172.

[12] Sybertz, F.: Beurteilung der Wirksamkeit von Steinkohlenflugaschen als Betonzusatzstoff. Deutscher Ausschuss für Stahlbeton (Hrsg.), Heft 434, Beuth, Berlin, 1995.

[13] Vivian H.E.: Studies in cement-aggregate reaction: part VII. In: Journal of the Council for Scientific and Industrial Research 20 (1947), S. 585-594.

[14] Vivian H.E.: Studies in cement-aggregate reaction: XVII, Some effects of temperature on mortar expansion. In: Australian Journal of Applied Science 2 (1951), Heft 1, S. 114-122.

[15] Nilsson, L.-O.: Moisture effects on the alkali-silica reaction. In: Alkalis in Concrete – Research and Practice, Proceedings 6th International Conference, Copenhagen, 1983, S. 201-208.

[16] Idorn, G.M.: Studies of Disintegrated Concrete –Part I. The Danish National Institute of Building Research, Copenhagen (1961) – Series: Progress Report N2.

[17] Krogh, H.: Examination of synthetic alkali-silica gels. In: Symposium on Alkali-Aggregate Reaction Preventive Measures, Reykjavik, August 1975, S. 131-163.

[18] Catharin, P.: Hydratationswärme und Festigkeitsentwicklung. In: Zement und Beton 22 (1977), Nr. 4, S. 148-159.

[19] Longuet, P. ; Burglen, L. ; Zelwer, A. : La phase liquide du ciment hydraté. In : Revue des Matériaux de Construction 676 (1973), S. 35-41.

[20] Marsh, B. K., Day, R. L.: Pozzolanic and cementitous reactions of fly ash in blended cement pastes. Cement and Concrete Research 18 (1988), Nr. 2, 301-310.

TABLE1: Specific properties of cements, slag (S), fly ash (V) and silica fume (D) determined according to EN 196 test series (glassy contents determined by XRD)

Specific value		Unit	CEM I (1.3)	CEM I-NA	S	V	D 1)
Physical properties							
Density		g/cm³	3.07	3.12	2.91	2.25	2.07
Fineness (Blaine)		cm²/g	3875	3355	3820	3890	200000 2)
Water demand		m.-%	30.5	27.0	-	-	-
Initial setting		min	240	165	-	-	-
Compressive strength	2d	MPa	25.8	20.1	-	-	-
	28d		44.9	60.7	-	-	-
Chemical properties and glassy content							
Oxides (LOI included)	SO_3	m.-%	3.31	3.13	0.09	0.40	0,13
	SiO_2		20.07	22.49	34.78	47.07	97,45
	Al_2O_3		5.47	3.68	12.04	30.51	0,17
	Fe_2O_3		3.08	1.36	0.32	8.23	0,07
	CaO		60.91	65.23	42.53	4.46	0,01
	MgO		2.83	0.78	7.60	1.33	0,19
	K_2O		1.55	0.51	0.43	1.63	0,39
	Na_2O		0.27	0.20	0.32	0.60	0,12
	$Na_2O_{\text{Äq}}$		1.29	0.54	0.60	1.67	0,38
LOI		m.-%	2.00	2.21	0.24	3.47	1.26
Glassy content		%	-	-	> 99	68	-

1) dried residue of the slurry used 2) according to BET, manufacturer information

TABLE 2: Chemical composition of ground water glasses (manufacturer information)

Designation	SiO_2	Na_2O	K_2O	molar ratio	mass ratio
	m.-%			SiO_2/M_2O	
Na (4.13)	79.6	19.9	-	4.13	4.00
Na (3.36)	76.5	23.5	-	3.36	3.25
Na (2.12)	66.9	32.6	-	2.12	2.05
K (4.15)	72.3	-	27.2	4.15	2.65
K (3.37)	68.0	-	31.5	3.37	2.15

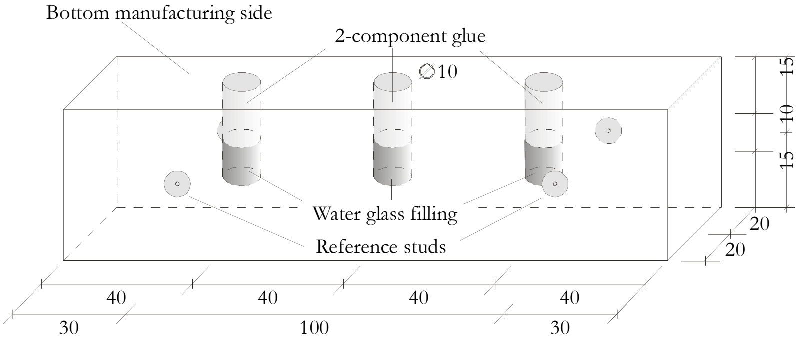

Figure 1: Preparation of mortar prisms for simulation of ASR-gel effect in a porous matrix (dimensions in mm).

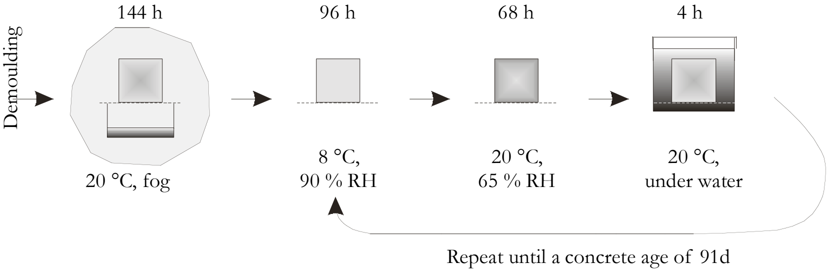

Figure 2: Order of different expositions during storage SAL.

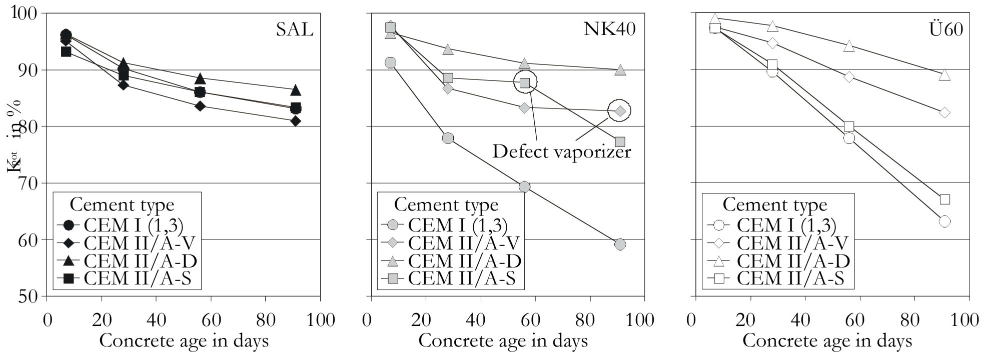

Figure 3: Remaining potentially effective K-content K_{pot} in concrete specimen produced with different cement types ($c = 500$ kg/m³, $w/c = 0.55$, limestone aggregate, $A/V_{specimen} = 0.60$).

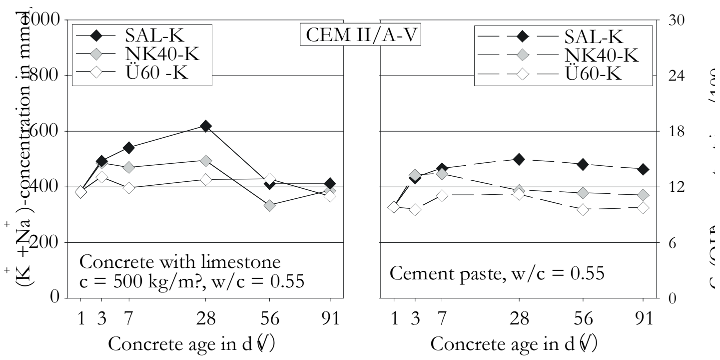

Figure 4: Alkali-concentration of concrete pore solution (left side) and Ca(OH)$_2$ content in cement paste in dependence of the applied temperature regime (conserved storage).

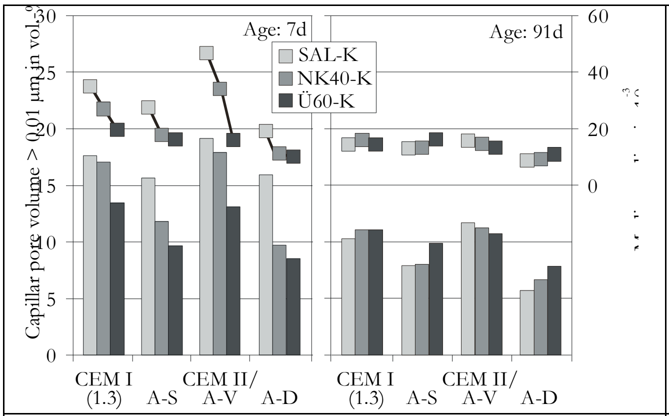

Figure 5: Capillar pore volume and median radius of the pore size distribution, both of the fine mortar portion of concrete at the age of 7d and 91d in dependence of the temperature regime.

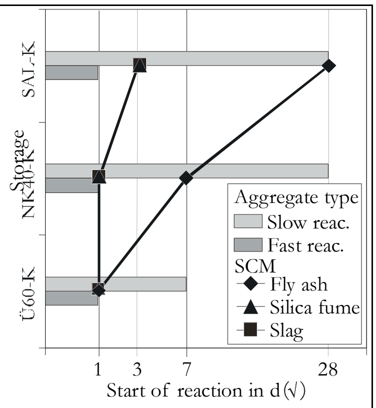

Figure 6: Start of reaction of different SCMs compared to the start of an ASR in OPC concrete.

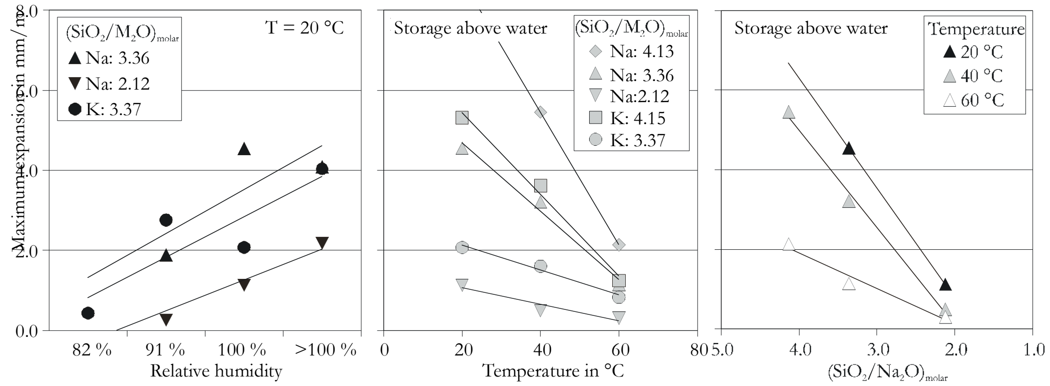

Figure 7: Maximum expansion of mortar prisms filled with ground solid water glass, dependent on relative humidity at 20 °C (left), on temperature (middle) and on the alkali content (right) of the water glass during storage above water.