THE EFFECT OF THE ALKALINITY OF THE PORE SOLUTION ON ASR

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Abstract

Results of a study are presented, showing a correlation between the damage of concrete specimens with different aggregates caused by alkali-silica reaction (ASR), estimated by length change measurements, and the alkalinity of the corresponding pore solutions.

The results show that for concretes with different Ordinary Portland Cements (OPCs) under otherwise identical conditions (aggregate, cement content, water-cement ratio) the damage of the concrete specimens depends on the alkalinity of the pore solution of the corresponding hardened cement paste.

The experiments with latent-hydraulic and pozzolanic materials show that their contribution to the avoidance of ASR is mainly based on the decrease of the alkalinity of the pore solution due to their specific hydration reactions and products, but other factors also seem to influence the degree of reaction.

Keywords: alkali-silica reaction, pore solution, alkalinity, expansion, supplementary cementing materials

1 INTRODUCTION

A deleterious alkali-silica reaction (ASR) can only occur in a concrete, if the aggregates contain alkali reactive constituents, if the pore solution is highly alkaline and if a sufficient amount of moisture is available. To avoid damages caused by ASR, at least one of these three factors has to be eliminated.

Consequently, an ASR will be prevented by lowering the pH-value of the pore solution under a critical level where the dissolution of alkali reactive constituents of the aggregates is reduced or even inhibited. Low levels of alkalinity can be achieved either by the use of OPCs with low alkali contents [1][2], or the use of blended cements with latent-hydraulic or pozzolanic constituents [2][3].

Since the solubilities of different alkali reactive constituents differ [4], it has to be assumed that the critical pH-values for aggregates containing these constituents in various amounts do not coincide. If the critical pH-value for an aggregate is known, adequate cements could be chosen to design concrete mixtures that are not susceptible to damage due to ASR.

Most national regulations regarding ASR stipulate a limited set of low-alkali cements when potentially reactive aggregates are used for certain structures, regardless of the differences in the reactivity of different aggregates. An approach based on critical pH-values could allow a more flexible combination of potentially reactive aggregates with cements without an increase of the risk of a deleterious ASR.

To evaluate the influence of the alkalinity of the pore solution on the course of an ASR in concretes with different alkali-reactive aggregates, several series of experiments were conducted. Concrete specimens were prepared in two series of mixtures. The concrete specimens were stored at 40 °C over water and their length changes were observed for one year. Simultaneously, cement pastes were prepared with all cements used in the concrete mixtures. The pore solutions were extracted by pressure from the hardened cement pastes and analysed for alkalinity. The hydroxide ion concentrations of the pore solutions in these samples can be interpreted as the highest alkalinity the aggregates are potentially exposed to in the concretes.

In the first series of concrete mixtures, an OPC with a low alkali-content (0.55 wt.% Na_2O_{equ}) was used as cement. Its alkali content was varied by adding different amounts of NaOH and KOH to the mixing water. The molar NaOH/KOH-ratio used was the same as in the cement itself. The resulting cement pastes had alkali contents of 0.70, 0.80, 0.90 and 1.00 wt.% Na_2O_{equ}, calculated for the dry cement. For comparison, an OPC with an original alkali content of 1.00 wt.% Na_2O_{equ} was

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also used. In this series, solely the influence of the alkalinity of the pore solutions on ASR was investigated.

In the second series of concrete mixtures, a high-alkali OPC (1.26 wt.% Na_2O_{equ}) and several blended cements with different amounts of ground granulated blast furnace slag (GGBFS) and fly ash (FA) were used as cements. The OPC served as a basis for the blended cements, which were mixed in the laboratory. In this series it was investigated, if the supplementary cementing materials (SCM) only have an effect on ASR by influencing the alkalinity of the pore solution, or if the behaviour of these concretes is different from concretes with OPC and a comparable alkalinity of the pore solution.

In both series, the cement content was varied for certain mix designs to comprise the influence of this factor.

2 MATERIALS AND METHODS

2.1 Materials

Aggregates

Four different German alkali-reactive aggregates were used as coarse aggregates (2-16 mm) in the concrete mixtures. They are listed in Table 1. These aggregates represent the rock types that have to be monitored in terms of their alkali-reactivity according to the German guidelines for the avoidance of a deleterious ASR [5]. All aggregates were provided by the respective producer in large batches of several hundred kg each and were reduced to the required amounts by sampling and splitting.

A natural quartz sand was used as a fine aggregate (0-2 mm) in all mixtures.

Cements

According to the experimental designs described in section 1, three different OPCs (CEM I 32,5 R according to [6]) and five different blended cements were used. One OPC (Z1a00) was modified by adding different amounts of NaOH and KOH to the mixing water. The blended cements were made in the laboratory by mixing different amounts of GGBFS and FA with different amounts of the OPC Z3a00. Homogeneity was achieved by mixing several kg of the materials for about 15 min in a laboratory scale Eirich mixer. The different cements are listed in Table 2.

The GGBFS fulfils the requirements of [6]. The fly ash fulfils the requirements of [7] and hence is equivalent to a Class F fly ash acc. to [8].

Admixtures

Some concrete mixtures showed inappropriate workability. In these cases a superplasticizer with a low alkali-content (0.5 wt.% Na_2O_{equ}) was added in amounts of about 1 wt.% referring to the cement.

2.2 Cement paste preparation and methods of examination

Mix designs

All cements listed in Table 2 were used to produce cement paste specimens with a water-cement ratio of 0.45.

Cement paste production

After mixing the cements with water or NaOH/KOH solutions at 20 ± 2 °C, the cement pastes were immediately filled air bubble-free into tightly closable 300 ml PE bottles. In order to avoid segregation the bottles were kept in motion for 24 h in a drum mixer. Afterwards the bottles were stored at 40 °C.

Storage and extraction of pore solutions

After different hydration times (7, 28, 90, 180 and 365 d) one bottle of each mixture was cut open and the cylindrical cement stone specimens were mounted into a press. Under a pressure of up to 260 MPa the pore solution was extracted, filtrated with a 0.45 μm filter and captured in PP tubes. The tubes were flushed with Argon and immediately tightly closed.

Pore solution analysis

The pore solution samples were analysed for their alkalinity. The pH-values were determined with 0.1 M hydrochloric acid in a titrator (Mettler Toledo DL55) with a pH electrode (DG111).

2.3 Concrete preparation and methods of examination

Mix designs

For the two experimental series described in section 1, concrete mixtures were produced with a water-cement ratio of 0.43 and a cement content of 600 kg/m³. For some cement-aggregate combinations, concretes with lower cement contents (350 and 450 kg/m³) were produced. An AB 16 grading curve was chosen for the aggregates (natural sand: 0-2 mm 40 wt.%; reactive aggregate: 2-4 mm 20 wt.%, 4-8 mm 20 wt.%, 8-16 mm 20 wt.%). The mix designs are listed in Table 3.

Concrete production

After the mixing of each concrete mix, two concrete prisms with dimensions of 4 cm by 4 cm by 16 cm were cast and compacted in steel moulds at 20 ± 2 °C. Gage studs were cast in the two front faces of each concrete prism to facilitate length change measurements. After 24 h of storage at 20 ± 2 °C and ≥ 95 % relative humidity, the concrete prisms were demoulded. The prisms were stored according to [9], i. e. upright in lidded containers over but without contact with water and at 40 °C.

Storage and length change measurements

24 h after the beginning of the storage an initial length measurement was performed at 40 °C. Further length measurements were performed 7, 28, 90, 180, 270 and 365 d after demoulding. Length changes were calculated for each concrete mixture as a mean value for the two concrete prisms in mm/m. The measurements were performed with an electric digital length comparator.

3 RESULTS

3.1 Pore solution composition

The analysis of pore solutions extracted from the investigated cement stones over a hydration time of one year yielded a range of hydroxide ion concentrations between 268 and 963 mM (Figures 1 and 2). The highest concentrations were measured in the OPC with 1.26 wt.% Na_2O_{equ} (Z3a00), the lowest concentrations in the blended cement with 40 wt.% Z3 and 60 wt.% GGBFS (Z3a16).

The three OPCs as well as the four cements with the low-alkali OPC Z1a00 and additions of NaOH and KOH (Table 2, first series) mostly reach constant levels of alkalinity after hydration times of 7 to 28 d. Only the analysis of the pore solution of Z3 after a hydration time of 90 d yielded a value significantly higher than the values of younger or older samples. The higher the Na_2O_{equ} of these cements is, the higher are the hydroxide ion concentrations found in the pore solutions. The concentrations in the pore solutions of the cement stones of cement Z1e00, i. e. OPC Z1 with a Na_2O_{equ} of 1.00 wt.% through addition of NaOH and KOH, are very similar to those in the cement stone of OPC Z2 with a Na_2O_{equ} of 1.00 wt.%.

Like the samples with OPCs, the pore solutions of the cement stones with different portions of Z3a00 and GGBFS (Table 2, second series) show relatively constant levels of alkalinity over the monitored period. The pH values decrease with increasing amounts of GGBFS in the blended cements. The hydroxide ion concentrations in the pore solution of the samples with 30 wt.%, 40 wt.% and 60 wt.% of GGBFS in the cement are reduced by about 230 mmol/l, 340 mmol/l and 520 mmol/l, respectively, compared to the results from pure Z3.

The hydroxide ion concentrations of cement stones with different portions of Z3a00 and FA (Table 2, second series) decrease about 100 mmol/l over the first 180 d of hydration, especially between the ages of 7 and 28 d. Between the ages of 180 and 365 d, there are only minor changes in the hydroxide ion concentrations of the pore solutions. After a hydration time of 365 d, the hydroxide ion concentrations in the pore solution of the samples with 20 wt.% and 30 wt.% are reduced by approx. 300 mmol/l and 400 mmol/l, respectively, compared to the results from pure Z3.

3.2 Length change measurements

The length change measurements carried out on concrete prisms with four different alkali reactive aggregates yielded expansions of up to 2.05 mm/m after one year of storage at 40 °C over water. For all four aggregates the highest expansions were measured in the concrete mixtures with the high-alkali OPC Z3a00.

In the first series of concrete mixtures with GW1 (cf. Table 3) only the specimens with the cements Z1e00 and Z2a00, both with alkali contents of 1.00 wt.% Na_2O_{equ}, show significant expansions of 0.46 and 0.75 mm/m after one year, respectively (Figure 3). The expansions start between 180 and 270 d of storage and seem to keep rising after one year. The length changes of the specimens of mixtures with lower alkali contents do not exceed 0.29 mm/m after one year

(Z1d00/GW1/x). In the second series of concrete mixtures with GW1 (cf. Table 3) the specimens with the cement Z3 show high expansions of 1.65 mm/m after one year of storage and seem to keep rising (Figure 4). The expansions of the concrete prisms with GGBFS and FA containing cements do not exceed 0.26 mm/m.

In the first series of concrete mixtures with GW2 (cf. Table 3) the specimens with the cements Z1e00 and Z2a00 show high expansions of 1.82 and 1.45 mm/m, respectively, after one year of storage (Figure 5). The expansions start between 90 and 180 d of storage and are considerably higher in the specimens of the mixture with Z1e00 at the beginning, although the alkali content of both cements, calculated for the dry cement, is identical. The expansion values converge after one year but seem to keep rising in both cases. The prism lengths of the mixtures Z1a00/GW2/x and Z1c00/GW2/x deviate 0.02 and 0.26 mm/m from the initial lengths after one year. In the second series of concrete mixtures with GW2 (cf. Table 3) the mixture Z3a00/GW2/x shows the largest observed expansions of 2.05 mm/m after one year (Figure 6). But also the mixture with 30 wt.% GGBFS in the cement (Z3a13) shows significant expansions of 0.79 mm/m. The length change measurements on specimens of the mixtures with 40 wt.% GGBFS and with 30 wt.% FA in the cement yielded lower expansions of 0.35 and 0.26 mm/m, respectively.

The results for the concrete mixtures with QP are similar to those of the mixtures with GW1. In the first series (cf. Table 3) only the mixtures with the alkali richest cements Z1e00 and Z2a00 show elevated expansions of 0.40 and 0.37 mm/m after one year, respectively (Figure 7). The expansion starts after 180 to 270 d of storage. The length changes of the other specimens of this series of mixtures do not exceed 0.19 mm/m. From the second series of concrete mixtures (cf. Table 3) only the specimens of the mixture with the cement Z3a00 show high expansions (0.63 mm/m) after one year of storage. The expansion starts after about 180 d of storage. The concrete prisms with blended cements do not show expansions exceeding 0.12 mm/m.

Like the concretes with the aggregates GW1 and QP, also the concretes with OR do not show expansions as high as the concretes with GW2. In the first series of mixtures, the concretes with the cements Z1d00, Z1e00 and Z2a00 show expansions from 0.33 to 0.64 mm/m after one year. The expansions start after different times between 90 and 270 d of storage. The concrete prisms with cements with lower alkali contents (Z1a00, Z1b00, Z1c00) do not show expansions exceeding 0.19 mm/m. In the second series of concrete mixtures the expansions of the specimens with the cement Z3a00 show high expansions of 0.83 mm/m after one year. The expansions start after about 180 d of storage. The concrete prisms with blended cements do not show expansions exceeding 0.30 mm/m. Between 270 and 365 d of storage the expansions of all concrete prisms with OR decline or stop.

All concretes produced with cement contents lower than 600 kg/m^3 (cf. Table 3) did not show significant expansions (not shown in figures).

4 DISCUSSION

4.1 Pore solution composition

The hydroxide ion concentrations in the pore solutions of the different cement stone samples made with OPC show a clear dependency on the alkali content of the cement (Figure 11). This is in accordance with the literature (e. g. [1], [2]). The results also confirm that adding sodium and potassium hydroxide solutions to the mixing water of an OPC leads to hydroxide ion concentrations in the pore solution that follow the trend of the other OPCs when plotted against the alkali content calculated for the dry cement (Figure 11). Therefore the method can be used for a controlled increase of the hydroxide concentrations in pore solutions.

The hydroxide ion concentrations in the pore solutions of cement stones with different amounts of GGBFS deviate from the trend of the OPCs. The alkalinity of the pore solutions is lower than in the pore solution of a Portland cement stone with a comparable alkali content. The deviation increases with increasing contents of GGBFS in the cement (Figure 11). This can be explained by the observation that the contribution of alkali ions to the pore solution by GGBFS is significantly lower than by Portland cement clinker. Additionally, the amount of released alkali ions is almost independent of the alkali content of the GGBFS [2].

After a hydration time of one year, the alkalinity of the pore solutions in cement stones made with blended cements with FA is lower than that of the corresponding OPC (Z3a00) although the fly ash is richer in alkalis (Figure 11). The products of the pozzolanic reaction bind more alkali ions than are released by the fly ash [2].

The stabilisation of the hydroxide ion concentrations in the pore solutions of the OPCs, the OPCs with NaOH and KOH and the blended cements with GGBFS after a hydration time of 28 d coincide with the findings of other researchers (e. g. [2], [10]). The delayed stabilization of the concentrations in the cement stones of blended cements with FA can be explained with the slow progress of the pozzolanic reaction.

4.2 Influences on the expansion of concrete prisms

The length change measurements and the comparison with the results of the pore solution analysis and the mixture compositions show different factors controlling the expansion of the concrete prisms.

The alkalinity of the pore solution plays an important role for the occurrence of a deleterious ASR. In all cases, expansions attributable to a deleterious ASR only occur when a "critical" pH-value is exceeded (Figures 12 to 15). This critical pH-value is different for the four aggregates. It lies at about 13.6 for the greywacke GW2, between 13.6 and 13.7 for the greywacke GW1, and at about 13.7 for the rhyolite QP and the crushed gravel OR. For all four aggregates, the expansion of the concrete prisms generally increases with the alkalinity of the pore solution. The different critical pH-values reflect the different alkali reactivity of the aggregates. The concretes with greywacke GW2 not only show the lowest critical pH-value, they also have the highest expansion rates and the reaction starts earlier than in concretes with the other aggregates.

Another main factor controlling the expansion of the concrete prisms besides the alkalinity of the pore solution and the reactivity of the aggregates is the cement content of the concrete. The high cement content of 600 kg/m^3 used for the main experimental program provoked expansions that were not observed in the concrete prisms with the same cements and aggregates but lower cement contents that are more commonly used in concrete structures (cf. Table 3). Therefore, the critical pH-values resulting from the experiments with high cement contents probably contain a factor of safety when mixtures with lower cement contents are considered.

Another factor influencing the length change behaviour of the concrete prisms is the composition of the cement. The concretes with the blended cement Z3a13 show lower expansions after one year than the concretes with the Portland cement Z2a00, although the pore solutions of both cements have similar hydroxide ion concentrations. (Figures 12 to 15). This is possibly attributable to a lower porosity in the cement stone of the blended cement, which impedes the transport of the reactants (water, hydroxide ions, alkali ions).

But also some of the OPSs altered by the addition of NaOH and KOH (Z1d00 and Z1e00) in combination with the aggregates GW1, QP and OR lead to lower expansions than those observed in concretes with the OPC Z2a00 although the hydroxide ion concentrations of the corresponding pore solutions are similar. It is known that the addition of alkali hydroxides concrete mixtures affects not only the hydroxide ion concentration of the pore solution, but also the concentrations of the alkali ions and sulphate ions [11]. This might affect the course of the ASR and the expansion behaviour of the concrete.

It should be considered that the results and interpretations presented here are based on experiments with a relatively short duration time of one year. This could lead to wrong (too low) critical pH-values for aggregates reacting slowly. Some aggregates might start to react only after longer storage times in concretes with lower alkalinities. On the other hand, the extreme testing conditions (high temperature, high humidity, high cement contents in the concretes) could also lead to wrong (too high) critical pH-values. For a correct assessment of the significance of the found critical pH-values, the results have to be calibrated with long term field site tests.

5 CONCLUSIONS

- The hydroxide ion concentrations of the pore solutions in cement stones made with OPCs and with OPCs altered by the addition of NaOH and KOH depend on the alkali content of the OPC (calculated for the dry cement). The hydroxide ion concentration can be effectively reduced by the use of blended cements with GGBFS or FA. The alkalinity of the pore solutions of the cement stones produced with these cements is significantly lower than in cement stones made with OPCs with similar alkali contents.
- The expansion behaviour of concrete prisms stored at 40 °C over water for one year mainly depends on the reactivity of the aggregate and on the alkalinity of the pore solution. For each aggregate a critical pH-value could be defined for a cement content of 600 kg/m^3. Concretes with pH-values of the pore solution remaining below the critical value of the aggregate did not show significant expansions. When the same aggregates were combined with cements that led to alkalinities exceeding the critical pH-value, the concrete prisms showed expansions attributable to an ASR.

- Concrete prisms with lower cement contents more commonly used in concrete structures did not show significant expansions. Therefore, the critical pH-values found for a high cement content probably contain a factor of safety when mixtures with lower cement contents are considered.
- Blended cements with GGBFS seem to have ASR-reducing effects beneath the reduction of the alkalinity of the corresponding pore solutions, possibly caused by the lower porosity of the cement stone.
- The results of this study have to be calibrated with long time field site testing.

6 ACKNOWLEDGEMENTS

The investigations were founded by the Federal Ministry of Economics and Technology through the "Otto von Guericke" Federation of Industrial Research Associations (AiF Research Project No. 14242 N).

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TABLE 1: Aggregates used in the study.

Aggregate name	*Aggregate type*
GW1	greywacke
GW2	greywacke
QP	rhyolite
OR	crushed gravel from the Upper Rhine Valley with silicified limestone

TABLE 2: Cements used in the study.

Series of experiments	*Cement name*	*Cement composition*	*Na_2O_{equ} (wt.% referring to the dry cement)*	*Cement type according to [6]*
First series	Z1a00	Z1	0.55	CEM I
	Z1b00	Z1 + *KOH + *NaOH	0.70	CEM I
	Z1c00	Z1 + *KOH + *NaOH	0.80	CEM I
	Z1d00	Z1 + *KOH + *NaOH	0.90	CEM I
	Z1e00	Z1 + *KOH + *NaOH	1.00	CEM I
	Z2a00	Z2	1.00	CEM I
Second series	Z3a00	Z3	1.26	CEM I
	Z3a13	70 wt.% Z3 + 30 wt.% GGBFS	1.03	CEM II/B-S
	Z3a14	60 wt.% Z3 + 40 wt.% GGBFS	0.95	CEM III/A
	Z3a16	40 wt.% Z3 + 60 wt.% GGBFS	0.80	CEM III/A
	Z3a22	80 wt.% Z3 + 20 wt.% FA	1.61	CEM II/A-V
	Z3a23	70 wt.% Z3 + 30 wt.% FA	1.78	CEM II/B-V

* : added to the mixing water

TABLE 3: Mix designs for concretes.

Series of experiments	*Cement name*	*Coarse aggregate (2-16 mm)*			
		GW1	*GW2*	*QP*	*OR*
First series	Z1a00	x; z1; z2	x	x; z1; z2	x; z1*; z2
	Z1b00	x		x	x
	Z1c00	x; z1; z2	x	x; z1; z2	x; z1; z2
	Z1d00	x		x	x
	Z1e00	x; z1*; z2	x	x; z1*; z2	x; z1*; z2
	Z2a00	x	x	x	x
Second series	Z3a00	x	x	x	x
	Z3a13	x; z1; z2	x	x; z1; z2	x; z1; z2
	Z3a14	x; z1; z2	x	x; z1; z2	x; z1; z2
	Z3a16	x		x	x
	Z3a22	x		x	x
	Z3a23	x; z1; z2	x	x; z1; z2	x; z1; z2

x : cement content = 600 kg/m³; z1 : cement content = 350 kg/m³;
z2 : cement content = 450 kg/m³; * : addition of superplasticizer

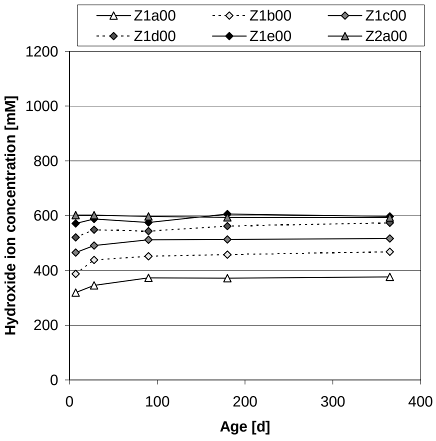

Figure 1: Hydroxide ion concentrations in the pore solution of cement stone samples (cf. Table 1).

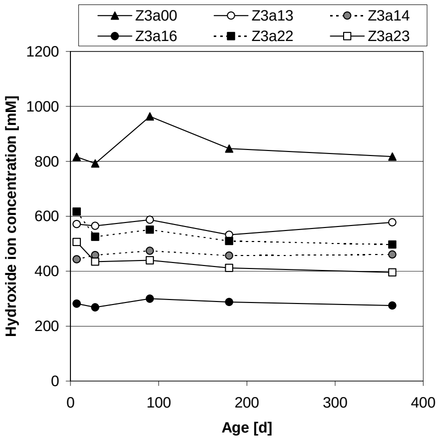

Figure 2: Hydroxide ion concentrations in the pore solution of cement stone samples (cf. Table 1).

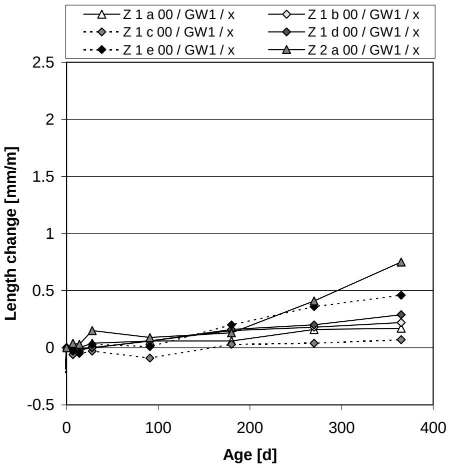

Figure 3: Length changes of concrete prisms with aggregate GW1 stored at 40 °C over water; sample designation: cement/aggregate/cement content (cf. Tables 1 to 3).

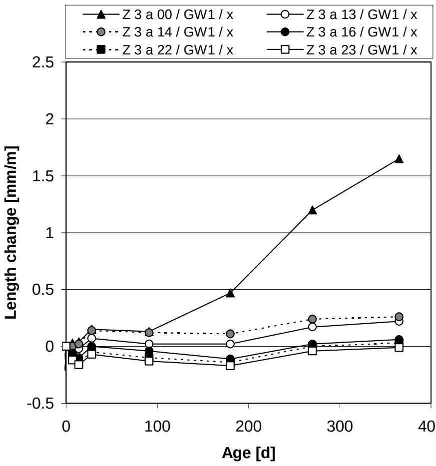

Figure 4: Length changes of concrete prisms with aggregate GW1 stored at 40 °C over water; sample designation: cement/aggregate/cement content (cf. Tables 1 to 3).

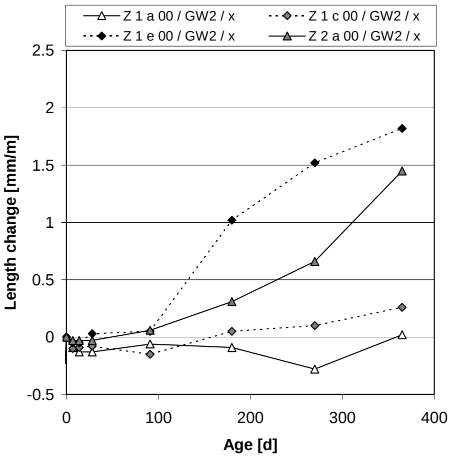

Figure 5: Length changes of concrete prisms with aggregate GW2 stored at 40 °C over water; sample designation: cement/aggregate/cement content (cf. Tables 1 to 3).

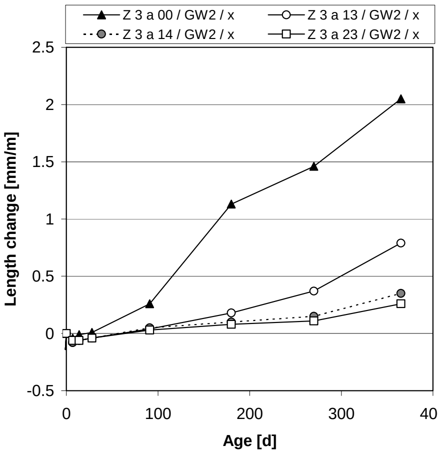

Figure 6: Length changes of concrete prisms with aggregate GW2 stored at 40 °C over water; sample designation: cement/aggregate/cement content (cf. Tables 1 to 3).

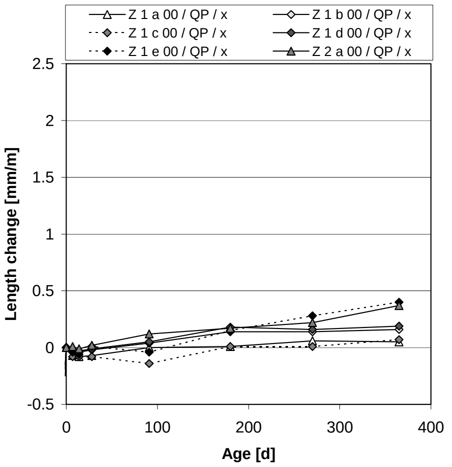

Figure 7: Length changes of concrete prisms with aggregate QP stored at 40 °C over water; sample designation: cement/aggregate/cement content (cf. Tables 1 to 3).

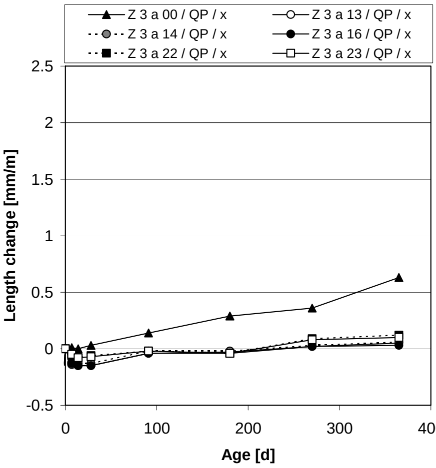

Figure 8: Length changes of concrete prisms with aggregate QP stored at 40 °C over water; sample designation: cement/aggregate/cement content (cf. Tables 1 to 3).

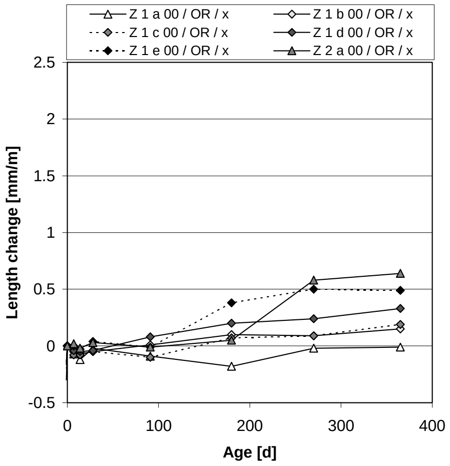

Figure 9: Length changes of concrete prisms with aggregate OR stored at 40 °C over water; sample designation: cement/aggregate/cement content (cf. Tables 1 to 3).

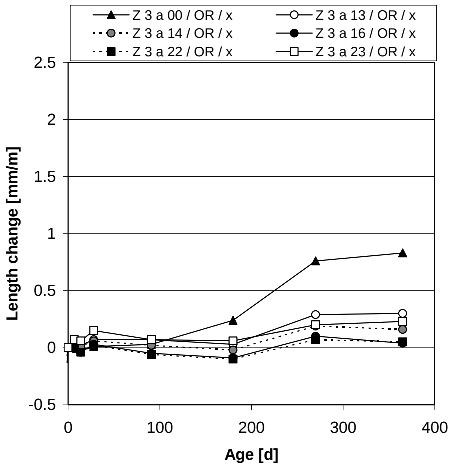

Figure 10: Length changes of concrete prisms with aggregate OR stored at 40 °C over water; sample designation: cement/aggregate/cement content (cf. Tables 1 to 3).

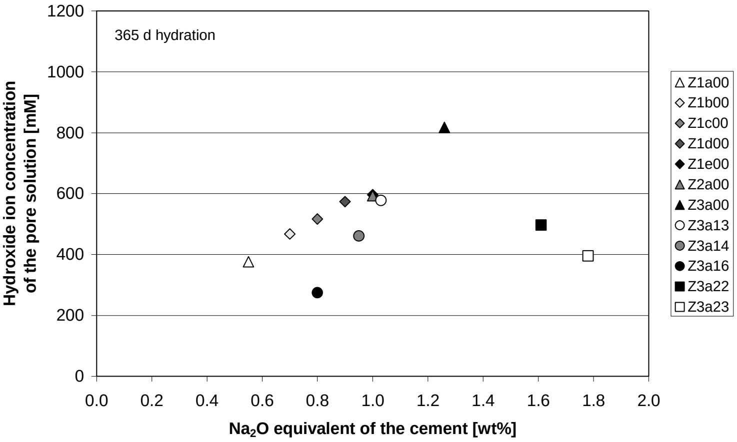

Figure 11: Hydroxide ion concentrations in the pore solution of cement stone samples vs. the Na_2O_{equ} of the corresponding cement (cf. Table 1).

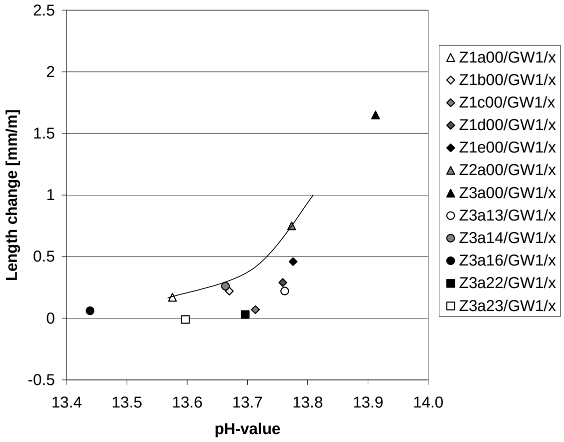

Figure 12: Length change of concrete prisms with GW1 vs. pH-value of the corresponding pore solution after 365 d of hydration.

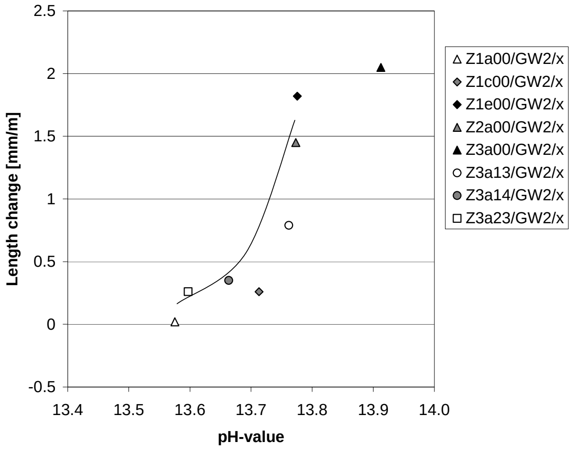

Figure 13: Length change of concrete prisms with GW2 vs. pH-value of the corresponding pore solution after 365 d of hydration.

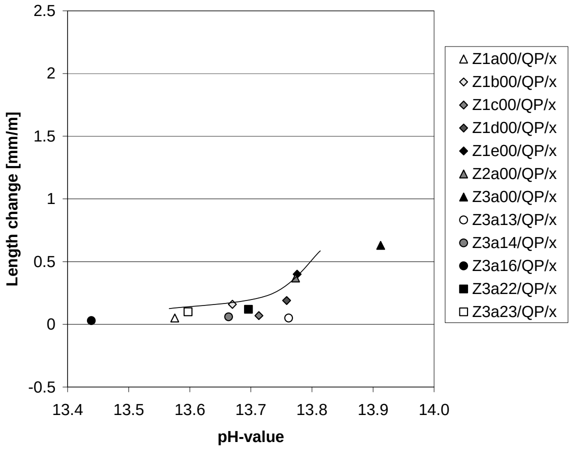

Figure 14: Length change of concrete prisms with QP vs. pH-value of the corresponding pore solution after 365 d of hydration.

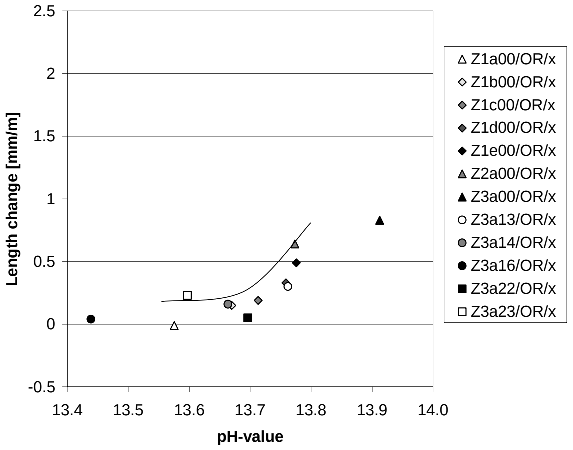

Figure 15: Length change of concrete prisms with OR vs. pH-value of the corresponding pore solution after 365 d of hydration.