Influence of Superplasticizers on C_3A Hydration and Ettringite Growth in Cement Paste

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Abstract

Cement-superplasticizer incompatibilities can be a serious problem manufacture of concrete. The presented work describes one possible mechanism of cement-superplasticizer incompatibility.

Combination of scanning electron microscopy, quantitative X-ray phase analysis, isothermal heat conduction calorimetry, analysis of cement pastes pore solution and viscometric measurements enabled us to demonstrate the effect of microstructural variation on the fluidity of cement pastes. Hence it was possible to show that during the first hour of hydration the ettringite crystal growth is governed by the degree of supersaturation of the cement pore solution. Long prismatic ettringite was found to be a good indicator that the set regulator proportion is to low to regulate C_3A hydration sufficiently.

Furthermore the presence of superplasticizers hinders the dissolution of set regulator. For polycarboxylate type superplasticizers, this can lead to increased C_3A hydration, if the cement has a low content of soluble sulphate. The consequence of increased degree of C_3A hydration is the loss of fluidity of cement pastes.

Strong adsorbing superplasticizers like polycondensates of naphthalene sulphonate are able to retard set regulator (bassanite) dissolution as well as the C_3A hydration. That is the reason why those superplasticizers do not disturb the set regulation of the investigated cement.

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1 Introduction

The fluidity of fresh cement paste is defined by various factors: water to cement ratio, presence of superplasticizers and specific surface area as well as chemical composition of the cement. The most vigorous improvement of fluidity is achieved by addition of superplasticizers. This is mainly attributed to the improved dispersion of cement particles by superplasticizer molecules. Due to the wide variety of cements and superplasticizers and the limited understanding of their mode of interaction, incompatibilities between cement and superplasticizers are hard to predict.

One possible reason for cement–superplasticizer incompatibility is suspected to be the influence of superplasticizers on cement hydration. Several authors investigated the influence of superplasticizers on C_3A hydration and morphology of ettringite crystals [1-9]. They found that C_3A hydration is retarded by polymers of sulphonated melamine and naphthalene formaldehyde superplasticizers [10]. This has been correlated with the preferred adsorption of superplasticizers on interstitial clinker phases [4, 5]. Ettringite precipitation experiments revealed that adsorption of superplasticizers on growing crystals leads to crystallization of smaller ettringite crystals as compared to crystals precipitated in the absence of superplasticizers [2, 7-9].

The influence of superplasticizers on C_3A and set regulator hydration has been considered in the present study. For the induction period of cement hydration we determined the isothermal heat release of hydration, degree of C_3A hydration (quantitative X-ray phase analysis), supersaturation of cement pore solution (aqueous phase) and the microstructure of cement pastes (Environmental SEM). Obtained results were compared with fluidity of the cement paste measured with a viscometer. On this way it was possible to show the influence of superplasticizers on C_3A hydration and ettringite crystallization. Furthermore, it was possible to correlate changes in cement microstructure with undesired loss of fluidity of cement paste. Effects of the degree of supersaturation on the ettringite morphology have also been investigated. Investigations in this study display causes and effects of one cement-superplasticizer incompatibility.

2 Materials and methods

2.1 Materials
Cement of type CEM I 42.5 R (as in European Standard ENV 197-1:92) and clinker of this cement were investigated. Chemical composition of the studied cement clinker was determined to be as follows (wt %): 65.8 CaO, 21.7 SiO_2, 6.8 Al_2O_3, 3.1 Fe_2O_3, 1.1 MgO, 0.24 K_2O, 0.1 Na_2O, 0.7 SO_3, 0.7 CaO_{free}. Specific surface area and set regulator proportions of cement are shown in Table 1.

Set regulators were added to release different quantities of calcium and sulphate ions to the cement pore solution at the initial stage of hydration

(induction period). Therefore set regulators with different solubility, dissolution rates and or quantities were added. Because bassanite possesses the highest solubility and dissolution rate the set regulator consisting of 4 %[**] bassanite releases the highest quantities of calcium and sulphate ions into the cement paste pore solution. In contrast anhydrite has a lower solubility and a markedly decreased dissolution rate. Compositions of investigated cements are listed in Table 1. Set regulators with high specific surface area, i.e. 700 m^2/kg (Blaine), were added after grinding to the clinker. A homogenous particle distribution was achieved by shaking the cements for two hours.

	Specific surface (Blaine) [m^2/kg]	Set regulator [%]	Abbreviation
CEM I 42.5 R	300	anhydrite/gypsum 2.6 / 1.9	CEM I
Laboratory cements	300	bassanite/anhydrite 2 / 1	c3-SR1
		anhydrite 3	c3-SR2
	400	bassanite/anhydrite 2 / 1	c4-SR1
		bassanite/anhydrite 3 / 1	c4-SR3
		bassanite 4	c4-SR4

Table 1 – Specific surface and set regulator (SR) content of the cements.

Three superplasticizers with the following differences in structure have been used for investigations: two are polycarboxylate based (PC1 and PC2); the third is a polycondensate of sulphonated naphthalene formaldehyde (SNF). PC1 and PC2 consist of the same backbone molecule (polymethacrylic acid) with methoxy-polyethylene glycol (MPEG) side chains. The molar ratio of carboxyl groups to MPEG for PC1 is 3.5 and for PC2 is 1.6. MPEG molecular weight of PC1 is higher than in PC2. Superplasticizers are neutralized by addition of sodium hydroxide.

All samples were mixed at a w/c of 0.4. Superplasticizers were added to the mixing water as solutions with 30 % solid content (2 % superplasticizer solution referring to the amount of cement). Samples were stored in sealed plastic containers.

2.2 Methods

2.2.1 Isothermal heat conduction calorimetry
Heat release during hydration was measured with an isothermal heat conduction calorimeter (DCA Type3, Tonitechnik, Germany). To produce a homogeneous paste, samples were stirred while injecting the water into the reaction vessel containing the cement powder.

[**] All given as weight percent.

2.2.2 Scanning Electron Microscopy (SEM)

Observation of early cement hydration was carried out with an Environmental SEM equipped with a Field Emission Gun (ESEM-FEG XL30, Philips, Netherlands). The operational parameters for the "Environmental" mode were the following: water vapor in the chamber was set to 10.7-12.5 mbar and sample temperature to 12 °C. According to pressure and temperature, the humidity was kept between 75 and 90 % for the area of the cooled sample. Micrographs were taken at 25 keV acceleration voltage.

It has been shown that under high vacuum conditions structure detoriation (shrinkage, formation of bubbles) of ettringite due to electron bombardment is significantly higher than in the low pressure, humid environment of the ESEM [15]. Therefore the application of ESEM is crucial for investigation of hydrate phases like ettringite.

In the ESEM it is necessary to avoid drying artifacts of alkali sulfates, which could crystallize even by slight surface evaporation. Two adjustments to address this problem were made: 1) the residual pore solution of the paste was removed with filter paper, 2) the sample was inserted into the microscope chamber and a few millimeters of the sample surface, that might contain salts evaporated in the chamber atmosphere, were removed by scratching with a micromanipulator.

2.2.3 X-ray diffraction

Degree of C_3A hydration was determined for the first hour of cement hydration. Pastes were mixed by hand. The hydration was stopped by adding 2-propanol to the paste. For drying, samples were kept for 15 minutes at 30 °C in a drying chamber. All samples were ground to a grain size smaller than 45 µm. Measurements have been performed with an X-ray powder diffractometer (D-5000, Siemens, Germany). The CuKα (λ = 1.5418 Å) radiation was generated at 40 mA and 40 kV. Data have been collected over a Bragg angle range of 8-60° (2 θ), using an angular step size of 0.05° and 5 s counting time.

X-ray diffraction intensities were used to quantify the C_3A content of the samples. Because intensities can show a relatively large standard deviation by different measurements, each sample was distributed on five sample holders and measured. Standard deviation was determined. Quantification was done by Rietveld analysis with software TOPAS (Bruker AXS, Germany). Because only short hydration periods were investigated, it has been assumed that the samples contain a small amount of amorphous phases; therefore no standard for quantification of the amorphous phase was added.

2.2.4 Analysis of pore solution (aqueous phase) of cement pastes

Extraction of the solution was accomplished by centrifugation for 10 minutes at 15.550 times g. Additionally, the solution was filtered through a syringe filter (pore diameter: 0.45 µm). Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES, Optima 3000, Perkin-Elmer, USA) was used to measure Ca, Na, K, S, Al, Mg, Si and Fe

concentrations in the diluted liquid phase of cement. The pH was measured with a glass electrode. Ion concentrations were used to calculate activities and ion activity products as described elsewhere [11]. Ion activity product for ettringite ($IAP_{ettringite}$) was calculated with the following equation 1:

$$IAP_{ettringite} = \{Ca^{2+}\}^6 * \{Al(OH)_4^-\}^2 * \{SO_4^{2-}\}^3 * \{OH^-\}^4 \qquad (1)$$

The calculated ion activity products were compared with the solubility product (K_s) for ettringite as given by Warren [14]. Equation 2 was applied for the calculation of supersaturation indices (SI):

$$SI = \log (IAP / K_s) \qquad\qquad\qquad (2).$$

2.2.5 Viscometric testing

Fluidity of the cement pastes was determined with a viscometer that allows measuring the resistant torque of the mixer while the vessel containing cement paste rotates (Viskomat NT, Schleibinger Geraete GmbH, Germany). Generally in ideal liquids, fluidity is the reciprocal of viscosity. Since cement suspensions possess a yield strength and a non linear flow behavior, the fluidity of cement pastes depends on yield strength and viscosity. By the applied viscometric measurement it is not possible to discriminate yield strength and viscosity. Therefore in the following the term fluidity is used to describe the consistency of pastes. Measurements were carried out at 20 °C. Cement pastes were prepared according to a defined procedure: 1. cement was added to water containing superplasticizer in a Hobart mixer, 2. after 10 minutes the paste was inserted into the Viskomat NT and the measurement was started, 3. the torque resistance was measured for 120 minutes at constant rotation speed (60 rpm) of the vessel.

3 Results and Discussion

3.1 General aspects of C$_3$A hydration

Ettringite, gypsum, syngenite and portlandite are the main hydration products during the first 3-4 hours of cement hydration. For cements with low set regulator to C$_3$A ratio, AFm phases are possible hydration products too [12]. Because present investigations have been carried out with one low alkali clinker, syngenite formation can be regarded as negligible. Therefore, the influence of superplasticizers on C$_3$A and set regulator hydration as well as ettringite, AFm and gypsum formation after short reaction periods could be studied.

3.1. 1 Heat release of pastes without addition of superplasticizers

Figure 1 shows heat release of clinker and cement for up to 1 hour hydration. Obviously, for one type of set regulator (SR1), the heat release is influenced by the specific surface area of clinker, i.e. the quantity of C$_3$A available for hydration. By comparing the heat release of c4-SR1 with c3-SR1 (Fig.1) the direct correlation between specific surface area of cement and heat release is demonstrated. Thereby, the increased heat release at higher specific surface area is caused by the fact that more C$_3$A becomes

available for reaction. However, this known fact is not validated if the heat release of c3 is compared with that of c4-SR1. The reason is that the variation of the set regulator also influences the quantity of C_3A consumption.
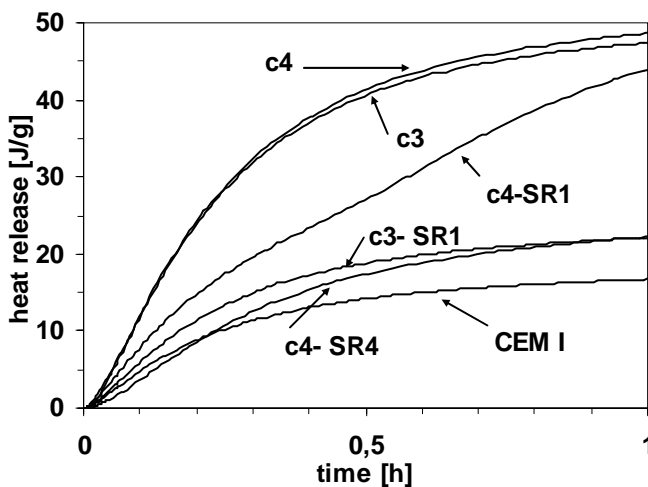

Figure 1 Heat release detected over the first hour of cement hydration in dependence of specific surface of clinker (300 resp. 400 m^2 / kg) and set regulators. *arrow indicates accuracy of measurement.

It is known that ettringite formation is mainly controlled by the available quantities of C_3A and calcium sulfate. Therefore, theoretically in the clinker with increased surface and 4 % bassanite (c4-SR4) the largest ettringite quantity can crystallize. Ettringite is supposed to generate more formation heat than any common AFm phase [13]. If the amount of released hydration heat is governed by ettringite formation, the cement paste c4-SR4 should release the maximum heat quantity. But results show that less heat is released than in the samples without set regulator. Therefore, it is concluded that the heat release is significantly influenced by the quantity of C_3A hydrated. The assumption that effectively less C_3A reacts if sufficient set regulator is added to the cement was verified by quantitative X-ray phase analysis.

3.2 Degree of C_3A hydration in cement pastes (quantitative X-ray phase analysis)

Results of quantitative (Rietveld-refined) X-ray phase analysis on cement pastes hydrated for one hour are shown in Figure 2. The content of 11 % C_3A in the unhydrated clinker is relatively high. Furthermore this cement is known to develop undesired workability loss when applied in combination with superplasticizers.

After a period of 1 hour hydration, the C_3A content in the cement containing 4 % bassanite (c4-SR4) is higher than in the sample without set regulator (c4). For the cements hydrated in the presence of PC-type superplasticizers and set regulator SR1 (2 % bassanite and 1 % anhydrite), surprisingly, the C_3A consumption was in a similar range as the sample hydrated without set regulator.

The results confirm the assumption arising form calorimetry; the degree of C_3A consumption depends on the set regulator. The reason for the in-

creased degree of C_3A hydration in the presence of PC-type superplasticizers will be discussed in the following.
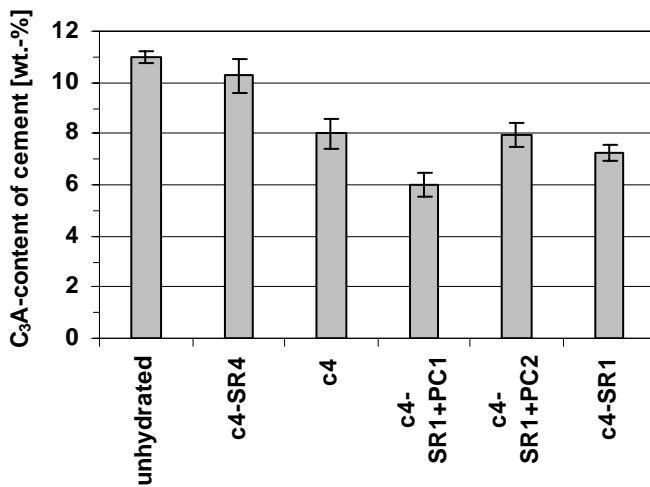

Figure 2: C_3A content of unhydrated and 1 hour hydrated cements (XRD-Rietveld). Error bars show standard deviation calculated by 5 measurements.

3. 3 The influence of superplasticizers on the heat release of cements and the hydration of bassanite

Results of heat conduction calorimetry of cement pastes containing superplasticizers are shown in Figure 3 (compare with Fig. 1). Obviously, during the first hour of hydration the PC based superplasticizers enlarge the heat release compared to SNF and reference sample. Similar trends were measured for the clinker with 400 m^2/kg specific surface (data not shown).
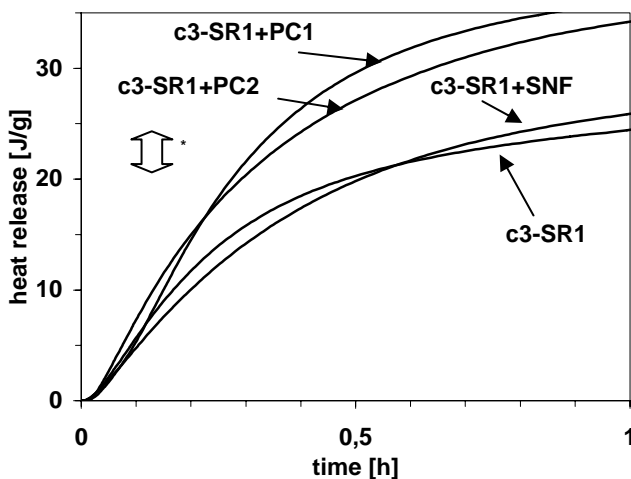

*Figure 3: Heat release over the first hour of cement hydration in dependence of superplasticizer addition. *Arrow indicates accuracy of measurement.*

These findings correlate very well with results of quantitative X-ray diffraction, which showed that due to addition of PC-type superplasticizers the degree of C_3A hydration increases.

In order to evaluate if the increased degree of C_3A hydration is caused by interaction of superplasticizers and set regulator, the heat release of bassanite hydration with and without addition of superplasticizers was determined. Results in Figure 4 clearly show that the bassanite hydration

is retarded by superplasticizers. Consequently less calcium and sulphate ions are supplied into the pore solution.
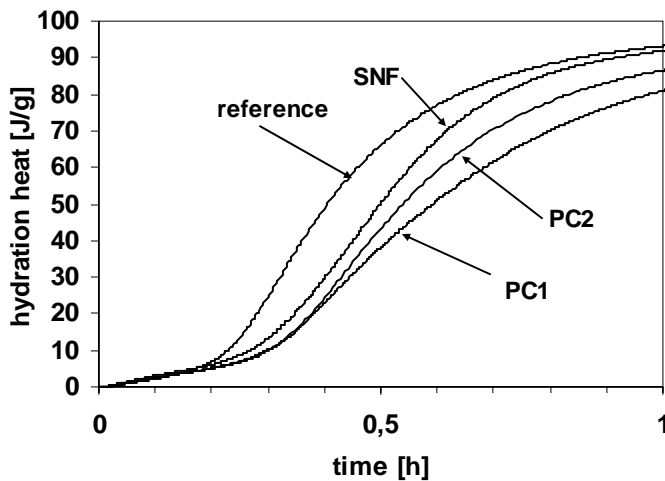

Figure 4: Heat release of bassanite hydration in dependence of superplasticizers addition.

*Table 2: Ca and S concentration in cement pastes pore solution*

Cement paste	Ca	S
	[mmol/l]	
ref (c3-SR1)	36	21
+SNF	26	36
+PC1	10	1
+PC2	21	7

For PC-type superplasticizers this thesis is supported by analysis of cement pastes pore solution (Table 2). For the SNF-type super-plasticizers the increased sulphur concen-tration results from sulfonate of unadsorbed superplasticizers (approx. 16 mmol/l). There-fore it is concluded that contrary to polycar-boxylates, SNF-type superplasticizers do not significantly influence the sulfate concentration in cement pastes pore solution.

3. 4 Microstructure and hydration of cement pastes
Microstructural changes (morphology of hydration products) of cement pastes were imaged by ESEM after 10 min hydration. Figures 5a & 5b show two morphologies of ettringite crystals: short prismatic (Fig. 5a) as
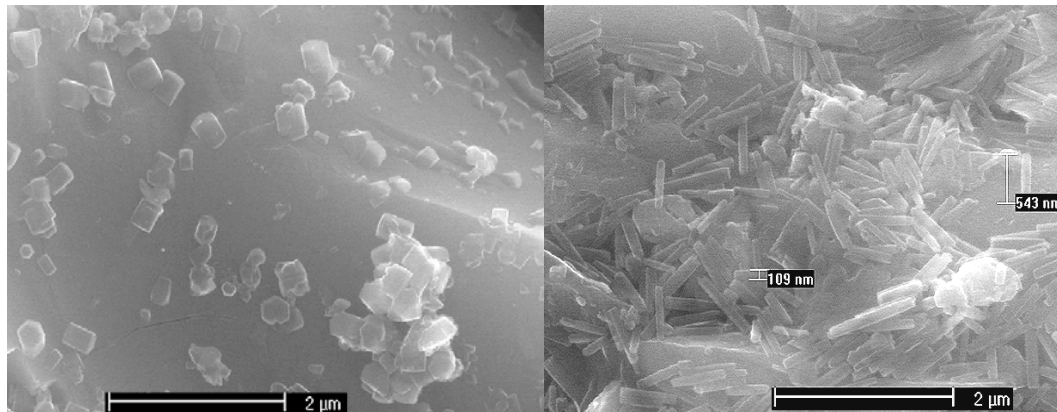
a) Short prismatic ettringite (CEM I)   b) Long prismatic ettringite (c3-SR1+PC1)

Figure 5: Habitus of ettringite crystals in cement pastes after 10 min of hydration (imaged in ESEM-FEG wet mode).

well as long prismatic (Fig. 5b) types. Ettringite aspect ratios (length vs. thickness) for all investigated pastes are shown in Table 3. Obviously, without addition of superplasticizers, primary ettringite crystals develop variable aspect ratios in dependence of set regulator composition and specific surface of the clinker (i.e. the quantity of C_3A available for hydration). The aspect ratio of ettringite crystals decrease as the amount and / or solubility of set regulator increases.

Table 3: Aspect ratio and saturation index of ettringite

Sample	Aspect ratio	$SI_{(ettringite)}$
CEM I	2	10
c3	-	6.1
c3-SR1	2	11.5
c4-SR1	3.5	10.4
c3-SR2	8-12	7.9
c3-SR1+ PC1	5-6	6.9
c3-SR1+ PC2	3-5	7.4
c3-SR1+ SNF	2.5	10.7

Also it is shown that the SNF-type superplasticizer generates ettringite of the same aspect ratio as observed in the reference sample without super-plasticizer (c3-SR1). In contrast the investigated PC type superplasticizers increase the ettringite aspect ratio.

Based on the assumption that the ettringite growth is governed by the availability of calcium, sulphate and aluminate ions, supersaturation of cement pastes pore solution with respect to ettringite was calculated from measured ion concentrations. Results in Table 3 show that by decreasing the amount of soluble calcium sulphate and / or increasing the quantity of C_3A available for hydration, the degree of supersaturation for ettringite decreases. Similarly, compared to the reference cement, addition of PC-type superplasticizer decreases the degree of ettringite supersaturation. By contrast the addition of SNF has a minor influence on the ettringite supersaturation.

Figure 6 displays the ettringite supersaturation indices in dependence of crystal aspect ratios. Obviously, low aspect ratio correlates with high supersaturation indices, and high aspect ratio correlates with low supersaturation indices. It is concluded that a saturation degree above 10 is necessary for growth of (hk0) and (00l) crystal faces at similar rates. In this case ettringite with low aspect ratio (short prismatic habitus) is formed. If the saturation index is below 9 growth on (00l) faces is favored and habitus of crystal can be described as long prismatic.

From this data we conclude that without addition of PC based superplasticizers to c3-SR1, the supersaturation for ettringite is sufficiently high to grow crystals on (hk0) and (00l) planes at similar rates. Whereas favored growth on the (00l) planes and lower supersaturation indices were determined by addition of PC based superplasticizers.

SNF-type superplasticizers retard bassanite hydration in a similar way as PC-type superplasticizers. One should therefore expect long prismatic ettringite growth and increased C_3A consumption for SNF sample too. This is contrary to our actual observations and previous studies (9, 10). These showed that due to their strong adsorbance, polycondensate based superplasticizers retard the C_3A hydration. To the contrary, most of PC

superplasticizers are lesser adsorbing on the cement surface. Referring to the ettringite morphology in the SNF sample, we find that hindrance of bassanite hydration is accompanied by retarded C_3A hydration and therefore, C_3A hydration is sufficiently regulated.
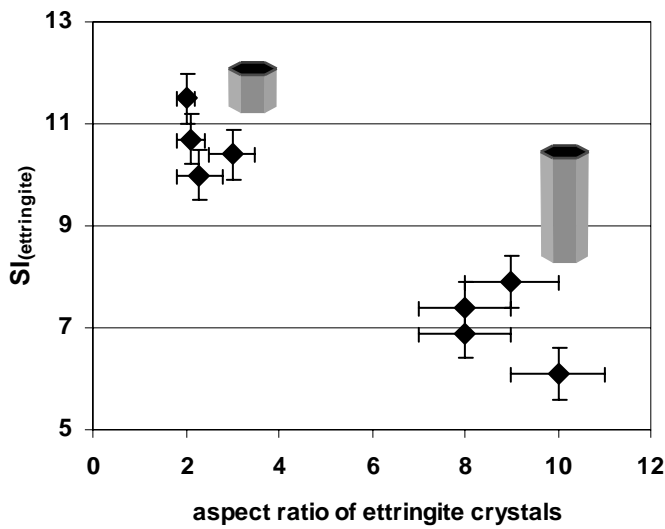

Figure 6: Saturation index (SI) of cement pastes pore solution in dependence of ettringite aspect ratio.

These findings show that dissolution behavior of calcium sulfate and C_3A control ettringite morphology.

3.5 Fluidity of cement pastes
Fluidity of the paste is a macroscopic parameter that is influenced by increased C_3A hydration. Without continuous stirring, all of the investigated cement pastes containing PC type superplasticizers showed a dramatic fluidity loss. Accompanying the fluidity loss an increased the hydration heat was mesured (Fig. 3). This indicates that flash set is the cause of fluidity loss.
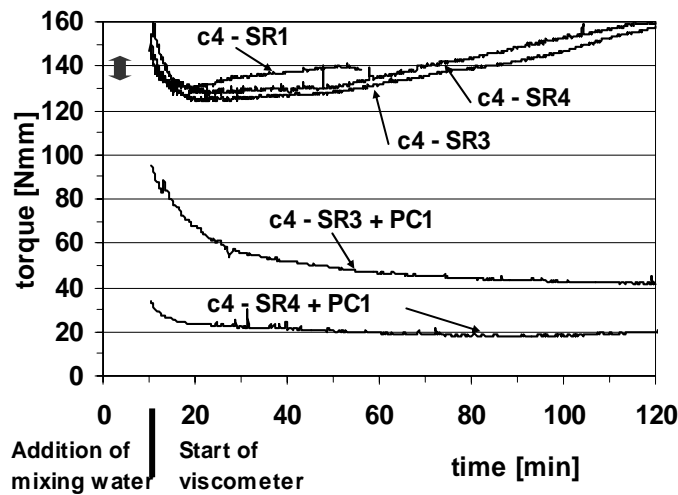

Figure 7: Fluidity (expressed as torque) of cement pastes in dependence of hydration time, set regulator and addition of PC-type superplasticizer. Double-headed arrow indicates standard deviation of measurement.

Viscometric measurements of the cement pastes (Fig. 7) reveal, that the fluidity of cement pastes containing PC-type superplasticizer is increased, if the bassanite quantity increases. In the cement pastes without addition

of PC-type superplasticizers the fluidity is not increased by increasing the bassanite quantity.

These data indicate that if cements with low set regulator to C_3A ratio are combined with certain polycarboxylate type superplasticizers, undesired flash set or loss in fluidity can occur. In order to exclude this incompatibility, it is important to determine if the quantity and solubility of the set regulator is sufficient to regulate the C_3A hydration in the presence of the superplasticizer.

4 Discussion and Conclusions

Previous findings [1, 6-9] indicated that crystal growth of ettringite is specifically influenced by superplasticizers and retarders. The authors showed that the crystal size of ettringite is smaller when admixtures are present. Adsorption of phosphonate, sulphonate and sulphate groups of admixtures are said to decrease the growth rate of ettringite at (00l) faces whereas polycarboxylate type superplasticizers are said to reduce the growth rates of the (00l) and the (hk0) faces of crystals. Clear indications for a preferred adsorption on (00l) ettringite crystal planes could only be found for phosphonates and sulphonates [6, 7].

Our findings demonstrate that in the absence of organic admixtures, ettringite crystal growth is governed by the degree of ettringite supersaturation of the cement pore solution. In the presence of organic admixtures, one has to consider at least two factors that influence the crystal growth; one is the specific adsorption of organic molecules on crystal faces and the other is the composition of the pore solution which is expressed by supersaturation index.

The present investigation has purposely been carried out with cement containing a low level of set regulator. This feature was chosen to unravel the mechanism of one particular cement-superplasticizer incompatibility. In that case, the hindrance of set regulator dissolution by superplasticizers has a significant influence on the ettringite crystallization. If cements with excess set regulator or high alkali content are chosen, the ettringite growth might be governed more significantly by the specific adsorption of superplasticizers on crystal faces.

We have shown that superplasticizers specifically influence C_3A and bassanite hydration. Investigated PC type superplasticizers mainly retard the bassanite hydration. In this way, for cements with general low set regulator content, PC type superplasticizers can cause an undersulphated cement paste. The consequence is increased C_3A hydration and the occurrence of long prismatic ettringite. Undesired loss of fluidity, or in extreme cases, flash set is the result of the superplasticizer influence on cement hydration.

The described cement–superplasticizer incompatibility can be prevented if the set regulator to C_3A ratio of the cement and the solubility of the set regulator is well adjusted.

The study showed that for a complete understanding of superplasticizers, not only dispersion mechanisms and forces have to be considered, but also that the influence of superplasticizers on cement hydration governs the plasticizing ability of superplasticizers.

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