

THE EFFECT OF DELAYED ETTRINGITE FORMATION AND ALKALI-SILICA REACTION ON CONCRETE MICROSTRUCTURE

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The simultaneous occurrence of the delayed ettringite formation mechanism and the alkali-silica reaction in concrete structures is not well understood. The studies have been carried out in order to produce more experimental data that would help to better clarify these problems. Three types of cements, with constant 1.2 % Na_2O_e and different SO_3 contents were prepared from industrial clinkers. The natural siliceous sand was used as an inert aggregate in the mortars, while opal was used as a reactive aggregate to produce the alkali-reactive specimens at a substitution ratio 6 % by total weight of aggregate. One series of samples was steam-cured at temperature of 90°C and the other was examined in accordance with ASTM C 227. The tests showed that the ettringite formation might increase the expansion due to the alkali-silica reaction as well as in the mortars cured at temperature of 38°C, as a result of the delayed ettringite formation on steam-curing of mortars. Irrespectively of the conditions of curing, the samples prepared from cement with very low SO_3 content always reveal the smallest expansion.

INTRODUCTION

Concrete is a building material that has many different applications, and in recent years its technology has experienced dynamic development. The standard for concrete structures [1] holds that concrete is presumed to retain its functional properties for at least 50-year service life. However, it is known that in some cases the premature failures, i.e. internal damages can develop in the concrete structures. That shows our understanding of the concrete chemistry to be incomplete. Concrete internal damages may be caused by the alkali-aggregate reaction and/or delayed ettringite formation.

Certain types of silica present in aggregate may react with hydroxide ions in the concrete pore solution and a sodium-potassium silica gel thus produced expands and causes the formation of internal stresses. As a consequence, the expansion of concrete takes place [2]. Delayed ettringite formation is a relatively rare and controversial phenomenon in concrete deterioration, strongly related to the practice of heat curing. Damage due to the delayed ettringite formation allegedly occurs when material is exposed to temperatures higher

than 70°C [3,4]. At this temperature range, ettringite is never formed during the early stage of hydration. In the presence of moisture, sulphates are released from the inner calcium silicate hydrate (C-S-H), formed during the heat treatment, and the formation of ettringite in matured, hardened concrete, months or years later than at normal temperatures, causing concrete damage is observed. These experimental results have been confirmed theoretically in the thermodynamic calculations performed by Damidot and Glasser [5]. The most important conclusion that can be drawn from these calculations is that alkalis play an important role in the stability of ettringite at high temperature. Concretes are usually exposed to the elevated temperatures at early ages, during the heat curing, to accelerate the early strength development. But there are also the other factors producing high temperature in concrete, among them high cement content, massiveness of concrete structure and hot weather conditions should be mentioned.

In numerous works the simultaneous occurrence of alkali-silica reaction and the delayed ettringite formation in the steam cured concrete has been reported, e.g. in those by Shayan and Quick [6, 7], as well as

Adams [8], Scrivener [9], and Shayan and Ivanusec [10]. It should be noted, however, that not all the authors relate the delayed ettringite formation to the products of the alkali-silica reaction [11]. Some works indicate that the earlier alkali-silica reaction leads to the generation of microcracks, which promote the onset of the delayed ettringite formation [12, 13]. In other words, it has been reported that the occurrence of microcracks due to the alkali – silica reaction in concrete is favourable for the subsequent expansion due to DEF [14].

As it is demonstrated by the foregoing cases, the determination of the role played by alkali-silica reaction and delayed ettringite formation in concrete deterioration has been an area of interest for many researchers. The objective of this contribution is to present some further data as the relative importance of alkali-silica reaction and delayed ettringite formation in concrete deterioration is concerned.

EXPERIMENTAL

Three types of laboratory made cements, from industrial clinkers, with low alkali and SO_3 content were used. Cement number one was produced from the ordinary Portland cement clinker (clinker "Portland 1") with gypsum only. Cement number two was produced using the same clinker with gypsum and potassium sulphate. The third cement was produced from the Portland cement clinker with no calcium aluminate (clinker "Portland 2") and no gypsum. The constant alkali level up to 1.2 % Na_2O_e was obtained by introducing the potassium hydroxide admixture. The chemical and phase composition (Bogue) of Portland cement clinkers is given in Table 1. The compositions of cements and sample notations are presented in Table 2. The $25 \times 25 \times 250$ mm mortar bars were produced from cements and quartz sand with 6 % opal, according to the ASTM C 227.

One series of mortar bars was steam-cured at temperature of 90°C for 8 hours (initial curing at an ambient temperature for 4 hours, the time during which the temperature was raised from ambient temperature - 4 hours, the time during which the temperature was lowered to an ambient temperature - 8 hours). The initial reading was taken after demoulding the specimens (after

steam-curing process). Subsequently, they were moist-cured at temperature of $38 \pm 2^\circ\text{C}$.

The second series of mortar bars was kept in the moulds for 48 ± 2 h and then removed from the moulds and measured for initial length. Afterwards the specimens were placed standing on one end over water in a storage container and the container was placed for 12 days in a chamber at temperature of $38 \pm 2.0^\circ\text{C}$. The 15-day measurement of the specimens' length was carried out after removing the container from the storage chamber and conditioning it at $23.0 \pm 2.0^\circ\text{C}$ for at least 16 h before opening the container. After every measurement the specimens were returned to the $38 \pm 2.0^\circ\text{C}$ storage and subsequent measurements at later ages were done following the same procedure.

The expansion of the bars was measured using a length comparator that conformed to the ASTM C 490-93a standard. Evaluations were made at the micro-structural level by SEM – EDAX equipment: JEOL JSM 35 CP scanning electron microscope equipped with an EDAX. The X-ray diffractometer (Philips PW-1040 A) using mono-chromatic $\text{CuK}\alpha$ radiation operating at a voltage of 30 kV and current of 15 mA was used in the phase composition examinations. The parameters of XRD measurements were as follows: scanning speed of $2^\circ 2\theta/\text{min.}$, step size of 0.02° ; the samples were analyzed in the range of $5 \div 60^\circ 2\theta$ to identify the mineral crystalline phases of mortars.

Table 2. Notation of mortar samples, composition of cements, contents of SO_3 and Na_2O_e , proportioning of mortar.

Notation of mortar	I	Ia	II
Types of clinker	Portland 1	Portland 1	Portland 2
Regulator of cement setting	6% gypsum	6% gypsum	–
Admixture	KOH	KOH+ K_2SO_4^*	KOH
SO_3 content (%)	2.29	3.76	0.19
Na_2O_e content (%)	1.2	1.2	1.2
Proportioning of mortar			
Cement	1 part	1 part	1 part
Aggregate	2.25 parts	2.25 parts	2.25 parts
w/c	0.47	0.47	0.47

* 2.11 %

Table 1. The chemical and phase composition (according to Bogue calculation) of Portland cement clinkers.

Clinker	Loss on ignition	Chemical composition (%)									
		SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	CaOw	Na_2O	K_2O	Na_2O_e
1	0.23	22.85	4.94	2.23	68.03	0.92	0.13	1.46	0.32	0.25	0.45
2	0.20	21.57	4.52	7.45	64.68	0.91	0.19	0.42	0.27	0.23	0.42
Clinker	Phase composition, (%) and specific surface of cement										
	C_3S	$\beta\text{C}_2\text{S}$	C_3A	C_4AF	$\text{S} (\text{cm}^2/\text{g})$						
1	61	20	9	7	3500						
2	54	21	0	23	3500						

RESULTS

Steam-cured mortars

The results relating to the measurements of linear changes of steam-cured mortar bars are shown in Figure 1. The curves shown in Figure 1 demonstrate that mortar I, made from Portland cement with an addition of standard gypsum, and mortar II, made from Portland cement 2, free of gypsum, did not reveal any expansion on curing over a period of 270 days, and only a very slight expansion was observed thereafter. However, the greatest expansion of 0.35 %, is found for the mortar Ia, made from cement with higher SO_3 content of (3.76 %), to which both gypsum and K_2SO_4 have been added (Table 2). The results of the observations under the scanning microscope after 150 days of hardening are shown in Figures 2 - 6.

In mortar Ia produced from cement with higher SO_3 content, there was ettringite, presented as the layers surrounding the surface of sand grains and filling the cracks in cement paste, including the cracks propagating from opal grains (Figure 3).

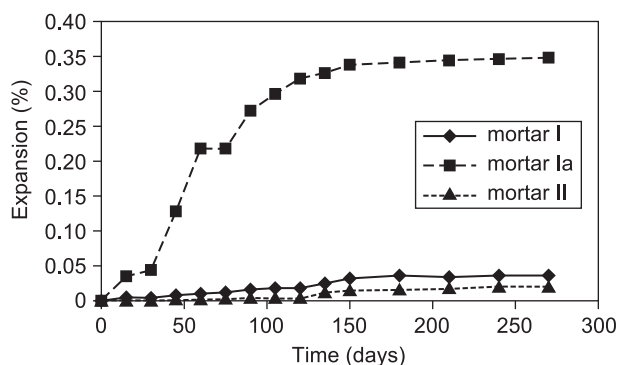


Figure 1. Expansion of steam-cured mortar bars with a reactive aggregate and cements with a varying content of sulphates (mortar II - 0.19% SO_3 , mortar I - 2.29 % SO_3 , mortar Ia - 3.76 % SO_3).

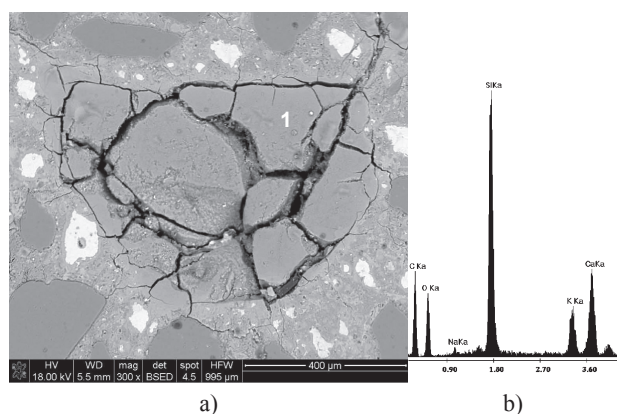


Figure 2. a) Opal grain; b) EDXA, alkali-silica gel shown in 1 (mortar Ia).

In mortars I and II, a potassium-calcium silica gel was present in the place of opal grains (Figures 4 and 6). Mortar I, produced from Portland cement 1, with a standard content of SO_3 , revealed the traces of ettringite in air cavities, partially filling them, as well as present in cement paste in the form of small concentrations in the vicinity of non-hydrated cement grains (Figure 5). This type of localization of ettringite in the mortar, as shown in Figure 5, is typical for this phase and can be found in many concrete samples that were not subjected to thermal processing [15]. A potassium-sodium-calcium silica gel is present in the area of the opal grains (Figure 6).

The observations of microstructure of mortars I and II show that the influence of potassium hydroxide has brought about the formation of potassium-calcium silica gel inside the opal grains, with increasing calcium content. At the same time, the mortar with very small SO_3 content (mortar II) and the mortar with standard gypsum content (mortar I) do not display any expansion. The results of the tests did not show the occurrence of ettringite in mortar II, made from cement with very small content of SO_3 . In mortar I, with standard content of SO_3 , ettringite was formed in small quantities, mainly in the air cavities, and did not influence the expansion. The mortars steam-cured at a temperature 90°C exhibit a significant expansion caused by the delayed ettringite formation, when produced from cement with high SO_3 content up to 4 %.

Mortar cured in accordance with ASTM C 227

The results of investigations relating to the expansion of mortar bars made from cements and quartz sand with the addition of opal according to the ASTM C 227 standard, including sample storage at 38°C and relative humidity $\geq 95\%$, are shown in Figure 7.

The bars of mortars, made from three different types of cement, displayed rapidly increasing expansion as early as 15 days after sample preparation (Figure 7). The expansion of the bars after 270 days varied within a range of 0.11% to 0.18%, despite the fact that the content of alkalis in all of the cements was identical (1.2%) and the addition of opal was also the same (6%). The greatest increase in the length of the samples was found in case of the mortar made from the cement with the highest sulphate content while the smallest one – in the mortar made from cement with the lowest content of SO_3 . It is noteworthy that an expansion of 0.1% (considered a threshold for non-reactive aggregate) was attained by the mortars after different time of maturing; mortar Ia – as early as 15 days, mortar I – after 30 days, and mortar II after 90 days. Observations of the microstructure were carried out and they are illustrated as SEM images in Figure 8.

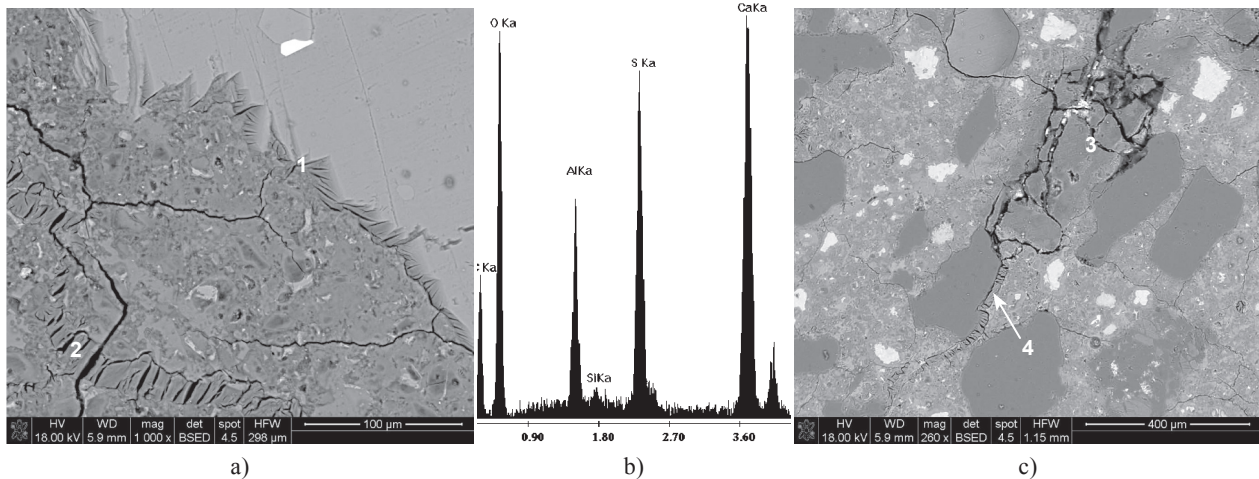


Figure 3. a) Ettringite on the surface of a sand grain (1) and in the cement paste (2); b) EDXA, ettringite shown in (1), (mortar Ia); c) opal grain (3) and ettringite filling in the crack (4) propagating from the sand grain in to the cement paste.

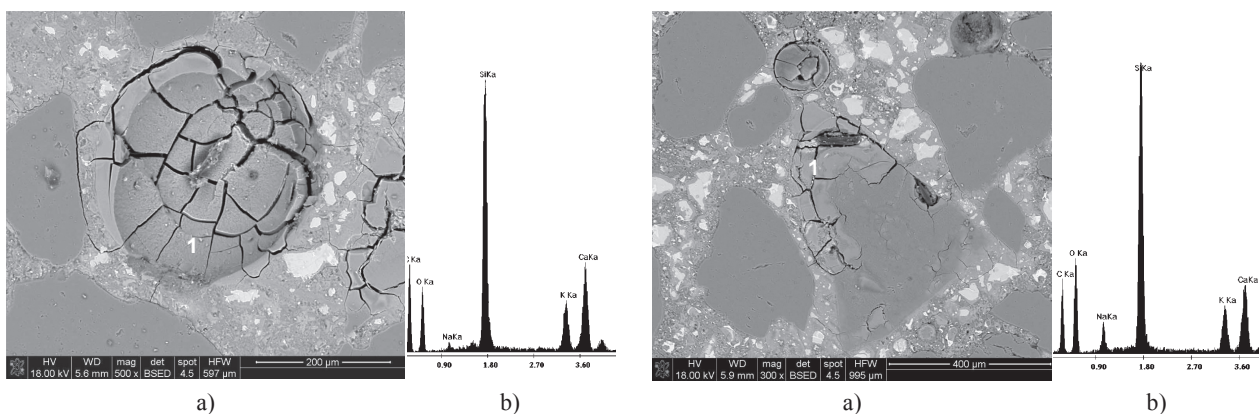


Figure 4. a) Potassium-calcium silica gel in the place of opal grains; b) EDXA, alkali-silica gel shown in 1 (mortar I).

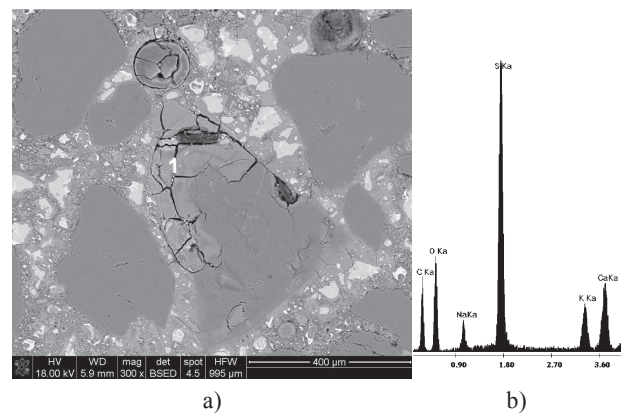


Figure 6. a) Opal grain (mortar II); b) EDXA, the alkali-silica gel shown in (1).

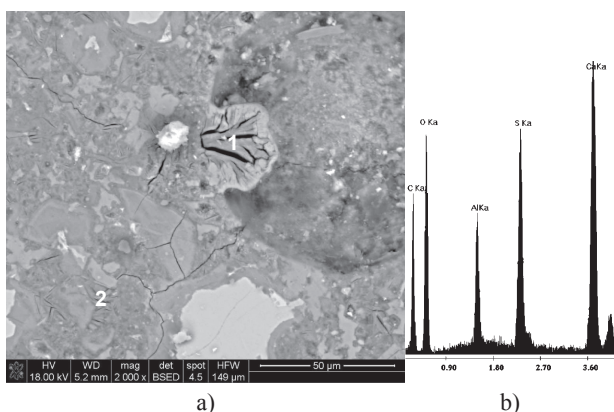


Figure 5. a) Massive ettringite in the air cavity (1) and its conglomerate in the cement paste (2); b) EDXA, ettringite shown in 1 (mortar I).

The results of the examinations of microstructure have shown that in all the mortars, the potassium-sodium-calcium silica gel occurs inside the opal grains. The considerable elongation of the mortar bars (exceeding the threshold expansion) points to the reactivity of the

aggregate, while the alkali-silica reaction is proved by the presence of potassium-sodium-calcium silica gel. The results of the tests indicate that the destruction of mortars stored at relative humidity $\geq 95\%$ and temperature of 38°C , is caused by the alkali-silica reaction. In none of the tested mortars (over a period of up to 150 days) we did not observe the presence of massive ettringite, characteristic for the delayed ettringite formation.

X-ray diffraction tests disclose the presence of ettringite in mortars I and Ia (Figure 9), thus, as one could presume, the delayed ettringite formation may increase the expansion of the mortars tested according to ASTM C 227.

DISCUSSION

Mortars I and II did not reveal any expansion during the storage after the steam curing, even at addition of opal and at significant potassium content, to give (with the small quantity of sodium from the clinker, approximately 0.3 %) the sodium equivalent at a level

of 1.2 %. In these mortars, the formation of potassium-calcium silica gel was observed. However, this gel was shown to have a high calcium content and low sodium content. In these two mortars the delayed ettringite formation did not occur.

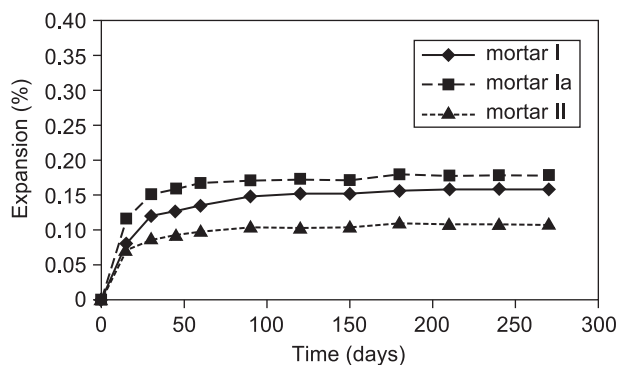


Figure 7. Expansion of mortar bars with a reactive aggregate and cements with a varying content of sulphates, cured in accordance with ASTM C227 (mortar II - 0.19 % SO_3 , mortar I - 2.29 % SO_3 , mortar Ia - 3.76 % SO_3).

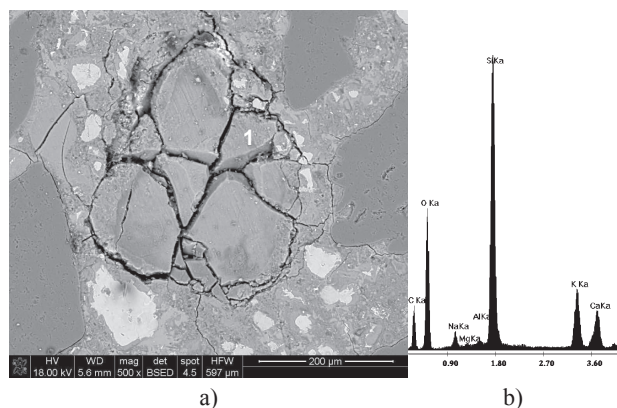


Figure 8. Opal grain; b) EDXA, alkali-silica gel shown in (1), (mortar Ia).

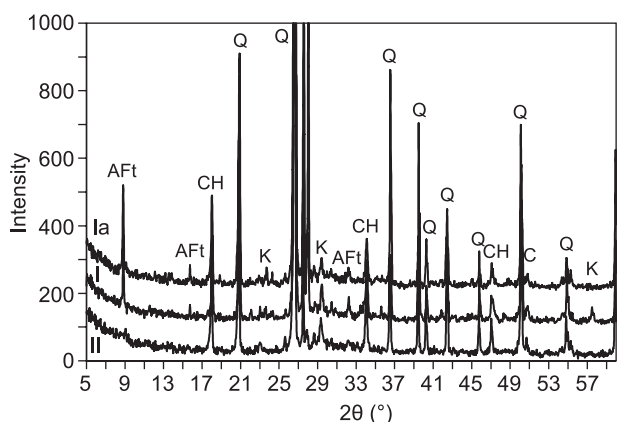


Figure 9. X-ray diffraction pattern of the cement mortars cured in accordance with ASTM C227, mortar II - 0.19 % SO_3 , mortar I - 2.29 % SO_3 , mortar Ia - 3.76 % SO_3 (AFt - ettringite, CH - portlandite, K - calcite, Q - quartz).

The microstructure of the steam-cured mortar Ia made from cement with higher SO_3 content, showing a considerable expansion, differs from the microstructure of the mortars discussed previously. In this mortar, the bands of delayed ettringite can be observed mostly in the paste-aggregate interfaces and in the paste too (Figure 3). Thus, we may assume that the alkali-silica reaction, which is promoted by steam curing, was the initiation of the cracks observed in this mortar. The ettringite fills these cracks or cavities in the aggregate-cement paste interface. A sodium-potassium-calcium silica gel was produced in the mortar Ia with a considerably higher content of sodium and a relatively lower content of calcium. This type of gel is characterised by significant expansion [16]. At the same time, the ettringite occurred in the mortar in forms indicating its delayed formation [17]. This corresponds to the results obtained by Kelham [18], who observed the highest expansion associated with the delayed ettringite formation in the 4% SO_3 containing mortar. In the case of mortar Ia, the content of SO_3 was 3.76 %.

It remains unexplained why, in spite of the addition of opal, no expansion was observed as a result of the alkali-silica reaction. Probably, the explanation may be found in the higher calcium content in the gel. We cannot exclude that the generation of microcracks caused by the delayed ettringite formation in mortar Ia facilitates the diffusion of sodium and causes the changes in the composition of silica gel, thereby producing a greater expansion.

The results of the tests may be provided with the following comments that put additional light on the unexpected lack of expansion in mortar I which, despite the addition of opal, did not expand after steam curing. Shayan and Ivanusec [10] showed that the measurement of the initial length of the samples was taken following a partial execution of the alkali-silica reaction in steam curing and, therefore, further measurements exhibit smaller increase in deformations, and the expansion observed during further storage reveals smaller values. We should assume that rapid expansion (approximately 0.15% as given by [10]) on steam curing is caused by alkali-silica reaction, the sulphate ions remain in the solution during this period and ettringite is not formed. It has not been explained why no expansion is noted in the experiments during the subsequent storage after curing of mortar with the reactive aggregate and cement free of sulphates or with a standard content of sulphates.

The results obtained point to the effect of SO_3 and alkalis content in cement on the stability of ettringite in the mortar subjected to steam curing. The mortar made from cement with a standard content of sulphates, as well as the mortar free of sulphates do not expand during the further storage that follows steam curing. Therefore, in the mortar with reactive aggregate, made from cement with high SO_3 content of (3.76%), the initial expansion during the period of 270 days was caused by the process

of delayed ettringite formation. This has been proved by the presence of ettringite on the surface of the aggregate grains.

There is a clear relation between the content of alkalis in cement and the stability of ettringite, as well as the rate of its formation during the steam curing, as it has been proved experimentally by Wieker [4], Damidot and Glasser [5, 20]. These authors analysed the phase balance in the $\text{CaO-Al}_2\text{O}_3\text{-CaSO}_4\text{-H}_2\text{O}$ system at temperatures of 25°C and 85°C on the basis of thermodynamic calculations. The calculations show that the minimal content of SO_4^{2-} in the liquid phase, determining the stability of ettringite, depends on the temperature and content of the sodium and potassium ions. The data from the work [21] also indicate that the higher content of alkalis leads to the increase of the sulphate ions content in the pore solution. Thus, the mortar Ia used in the experiment reveals three important features that are necessary for this process: reactive aggregate, as well as considerable concentration of alkalis and sulphates in the concrete pore solution.

Ong [22] observed a secondary ettringite in a mortar subjected to the steam curing; this ettringite, mainly filling the cracks, was generated as a result of the alkali-silica reaction. He suggests that the considerable expansion during and immediately after the steam curing is exclusively a consequence of the alkali-silica reaction, while the subsequent expansion at later age may be caused partially by the delayed ettringite formation. Shayan and Quick [13] are also of the opinion that the primary cause of concrete destruction is the alkali-silica reaction. Johansen et al. [23], however, pointed out that the stresses accompanying the alkali-silica reaction may cause cracks in the aggregate and surrounding cement paste, but not at the cement-aggregate interface. Diamond and Ong [24], in turn, found that high temperature curing of concrete elements may initiate the alkali-silica reaction in the case of certain reactive aggregates, while the subsequent reaction takes place during their further curing under moist conditions.

The results of our studies do not confirm that the alkali-silica reaction is the primary cause of the microstructure damage in the mortar prepared from free of SO_3 cement (mortar II) and in the mortar made from the cement containing a standard quantity of calcium sulphate; these mortars do not exhibit any expansion over a period of up to 270 days of curing. Mortar Ia, with an increased content of SO_3 , displayed the greatest expansion. The microstructure of this mortar is characterised by the occurrence of ettringite on the sand grains, which fills the cavities at the cement-aggregate interface. This is a characteristic feature of mortars displaying expansion that is connected with the delayed ettringite formation. In the case of the mortar with reactive aggregate and a large quantity of sulphates, the expansion may be caused by the pressure of the crystallisation of ettringite both in microcracks and in the cement paste.

The expansion of mortar bars made from three types of cement, as tested according to ASTM C 227, was diverse despite the fact that the content of alkalis in all of the cements was identical and totalled 1.2 %, and the opal addition to cement was also the same (6 %). It is also noteworthy that the expansion of all the mortar types even after 270 days exceeded 0.1 %, which is considered to be the threshold for non-reactive aggregate.

We must, however, underline that the expansion of the samples increased visibly as the content of SO_3 grew in the range between 2.29 % and 3.76 % (Figure 7). We cannot exclude that this dependence is the same as in the case of steam-cured mortars (Figure 1). X-ray diffraction tests disclose the presence of ettringite in mortars I and Ia (Figure 9), thus, as one could presume, the delayed ettringite formation may increase the expansion of the mortars tested according to ASTM C 227.

CONCLUSION

The results show that the formation of ettringite will augment the expansion of the mortars caused by the alkali-aggregate reaction. This increased expansion is also the consequence of the delayed ettringite formation in steam-cured mortars (Figure 1), and in mortars stored at temperature of 38°C (Figure 7). Irrespectively of the conditions of the processing or curing of mortars, the samples prepared from cement with a very small content of SO_3 always display the smallest expansion.

Further proof of the share of delayed ettringite in the expansion of mortars is the importance of SO_3 content. A comparison of mortar I (Table 2), which contains 2.29% SO_3 , with mortar Ia with a 3.76% content of SO_3 , points to the considerable role of delayed ettringite formation, for the maximum level of this expansion is connected with a content of approximately 4% of SO_3 [26].

A separate matter is the lack of expansion in mortars I and II containing opal, which were subjected to steam curing (Figure 1). This phenomenon cannot be explained on the basis of the tests. We may only note here that the behaviour of the mortars containing reactive aggregate is variously explained in the literature [10, 19].

However, mortars cured at a temperature of 38°C (Fig. 7) behave differently. The scale of the increasing expansion is maintained (mortars II, I and Ia) but the expansion of sample I is considerably greater than for the non-steam cured mortar sample and close to the expansion in sample Ia. In this case, the expansion connected with the opal reaction is predominant, while ettringite has a smaller importance. This is attested by a considerably greater expansion of mortar II (a comparison of Figures 1 and 7), which contains very little SO_3 . The formation of ettringite - whether delayed or connected with the recrystallisation of this phase - increases the expansion caused by the alkali-silica reaction.

It is still necessary to explain significant differences in the behaviour of the steam-cured opal containing mortars. This requires, therefore, further research to be carried out.

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