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Some factors affecting delayed ettringite formation in heat-cured mortars

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Abstract

Although more than 10 years of studies on delayed ettringite formation (DEF) have led to consensus in numerous areas of past disagreements, some questions remain experimental work is needed to complete the knowledge of this pathology. Following this objective, this paper studies the influence of pre-existing microcracking, wetting/drying cycles and the type of sulfated addition on DEF in steam cured mortars. The mortar specimens were prepared using an Ordinary Portland Cement and two types of sulfate were added to the mixtures: calcium sulfate ($CaSO_4$) or sodium sulfate (Na_2SO_4). The results confirm the well-known effect of temperature: no expansion was observed in any of the mixtures cured at room temperature. Moreover, no expansion was observed after 800 days for the reference mortar or for the mortar containing calcium sulfate but all the specimens of heat-cured mortars containing sodium sulfate expanded markedly after about 50 days whatever the supplementary treatments applied (thermal shrinkage or wetting/drying cycles). These results show the significant role played by alkalis in the occurrence of delayed ettringite. The supplementary treatments intended to cause prelimiray microcracking of the specimens did not promote expansion but contributed to a slight acceleration of the reaction. The ultimate values of expansion were similar to those obtained with sound mortars.

Keywords: Delayed Ettringite Formation (DEF); Alkalis; Expansion; Microcracking; Microstructure

1. Introduction

Heat-induced internal sulfate attack (still commonly referred as Delayed Ettringite Formation or DEF) of cement-based materials is a relatively recent pathology compared to other pathologies like alkali–silica reaction (ASR). During more than 10 years, numerous studies have been carried out by some of the most eminent researchers world-wide. Four years ago, a RILEM workshop on Internal Sulfate Attack and Delayed Ettringite Formation was held in order to debate the advances made in this area [1]. The proceedings of this workshop are concluded by the communication of Skalny [2] in which the author discusses the points of agreement and disagreement related to Internal Sulfate Attack.

Based on the conclusions of this workshop and other references [1–7], it is possible to summarize that the conditions necessary for the appearance of DEF are:

- an excessive rise of temperature during the casting of the concrete, related to the high heat of hydration of large masses of concrete or to the steam curing of concrete, especially in the case of precast products. This high temperature (>60–70 °C) inhibits the formation of non-expansive preliminary ettringite;
- the presence of sulfates, essentially coming from the cement (added as set retarder or present inside the particles of clinker) and, to a lesser extent, from the aggregate or the mixing water. These sulfates can react with the aluminates of the cement in presence of water to form delayed expansive ettringite;
- a moist environment: all the field disorders have appeared in areas exposed to water (bridges, dams, rail ties, …).

Some authors quote other factors that are important or even indispensable:

- pre-existing microcracking, which could be the consequence of thermal shrinkage or other pathology, such as ASR [5–9];
- wetting/drying cycles [9–11];
- a high alkali content in the concrete: alkalis play an important role in the stability of ettringite at high temperature [12–17].

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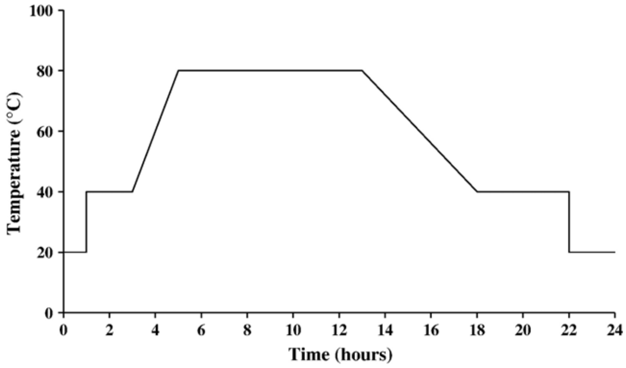

Fig. 1. Temperature cycle imposed on the heat-cured specimens.

To complete the knowledge of this pathology, the influence of pre-existing microcracking, wetting/drying cycles and the type of sulfated addition on DEF in steam cured mortars is studied in the present paper.

2. Experiments

2.1. Materials

The mortar specimens were prepared using an Ordinary Portland Cement (CEM I 52,5 R type according to the NF EN 197-1 standard) commonly used in the precast industry (64.3% CaO, 19.2% SiO_2, 5.1% Al_2O_3, 3.4% SO_3, 2.9% MgO, 2.5% Fe_2O_3, 0.7% K_2O and 0.3% Na_2O). This cement is comparable to the ASTM Type III cement that is, according to the literature, the cement the most susceptible to DEF [8,18,19]. The Blaine surface area of the cement was equal to 4200 cm^2/g.

The siliceous aggregate was a French sand meeting standard NF EN 196-1. Such sand was chosen because the expansion is faster with siliceous aggregate that with limestone sand [20–22].

Two types of sulfate were added to the mixtures: calcium sulfate or sodium sulfate. The calcium sulfate was a natural anhydrite ($CaSO_4$), ground to fineness similar to that of the cement. The sodium sulfate was a pure commercial product and its fineness was also comparable to that of the cement. The addition of sodium sulfate increased the amount of sulfate and, at the same time, the alkali concentration of the pore solution.

Three mixtures were tested: a reference mortar (C) and two compositions (A and S) in which 3% (by weight of cement) of $CaSO_4$ and 3.1% of Na_2SO_4 respectively were added to the mixture. So, the sulfate contents of the mixtures A and S were equal (5% total SO_3). The water/cement ratio was 3 and the sand/cement ratio was 0.5 in all the mixtures.

Mortars were prepared according to the standard NF EN 196-1. The mortar prisms were cast in 40*40*160 mm^3 molds equipped with embedded length measurement studs.

2.2. Procedures

2.2.1. Heat treatment

After casting, half of the mortar specimens were cured at room temperature (20 °C) and at 100% Relative Humidity and half were cured according to the heat treatment presented in Fig. 1 ($T_{max} = 80$ °C for 8 h). This heat treatment is representative of the treatments used in some precast industries.

The mortar specimens were cured in their metallic molds, tightly covered by a metallic plate to prevent the evaporation of water during the heat treatment. The specimens were placed in a drying oven equipped with a temperature controller. Thermocouples were placed in the control specimen to verify the temperatures reached during the treatment. After cooling, the prisms were demolded and stored at 20 °C in sealed bags until further treatment was applied.

2.2.2. Pre-existing microcracks induced by thermal shrinkage

The effects of the pre-existence of microcracks on the development of delayed ettringite were studied on microcracked specimens. Half the 7-day-old specimens (heat-cured or not) were precracked by placing in an oven at 40 °C for 24 h.

2.2.3. Curing conditions and wetting–drying cycles

Exposure to a moist environment is one of the conditions necessary for the occurrence of delayed ettringite. Some studies have also shown that wetting–drying cycles accelerate the reaction [9–11].

To test these two parameters, 14 days after demolding, half the specimens were stored in lime-saturated water at 20 °C, while the other half underwent nine wetting–drying cycles (5 days in water at 20 °C followed by 2 days in a drying oven at 40 °C). In both cases, the storage water was not renewed during the tests.

2.2.4. Nomenclature of the specimens

The following nomenclature will be used to facilitate discussion of the results:

- the specimens containing sodium sulfate are called S, those containing calcium sulfate (Anhydrite) are called A and those containing only cement are called C;
- the heat-cured specimens are called T, the others (cured at 20 °C) are called NT;
- the microcracked specimens are called P, the sound specimens are called NP;
- the specimens stored in water are called H, the specimens that underwent wetting–drying cycles are called HS.

There were three types of binder and eight specimens for each binder. Table 1 identifies the 24 specimens.

Table 1
Nomenclature of the specimens

C series	A series	S series
C-NT-NP-H	A-NT-NP-H	S-NT-NP-H
C-NT-NP-HS	A-NT-NP-HS	S-NT-NP-HS
C-NT-P-H	A-NT-P-H	S-NT-P-H
C-NT-P-HS	A-NT-P-HS	S-NT-P-HS
C-T-NP-H	A-T-NP-H	S-T-NP-H
C-T-NP-HS	A-T-NP-HS	S-T-NP-HS
C-T-P-H	A-T-P-H	S-T-P-H
C-T-N-HS	A-T-N-HS	S-T-N-HS

2.2.5. *Test procedures*

Compressive and flexural strength were measured, according to the standard NF EN 196-1, on 28 day old specimens. The accuracy of the length comparator was within 0.001 mm. The measurements on the specimens subjected to the wetting–drying cycles were carried out after each immersion period and after each drying period. In parallel, the weight of each specimen was measured with an accuracy of 0.01 g.

Microscopic examinations were carried out on fresh fractures or polished sections of mortar specimens using a Scanning Electron Microscope (SEM) (JEOL 6380 LV equipped with a backscattered electron (BSE) detector and an energy dispersive X-Ray analyzer (Röntec)).

3. Results

3.1. *Flexural and compressive strength*

The flexural and compressive strengths at 28 days of heat-cured mortars and mortars cured at room temperature are presented in Table 2.

The strength of the mortars cured at room temperature was comparable even though the addition of sodium sulfate seemed to decrease the compressive strength (about ten percent).

The thermal treatment led to a significant decrease of the mechanical strength at 28 days for each mortar and more significantly for the reference mortar and mortar S. This decrease is commonly observed when such strong thermal treatments are applied.

The particular behavior of mortars S could be explained by the accelerator effect of the alkalis on the setting and hardening of cement-based materials, which leads to a decrease in their mechanical strength in the longer term.

3.2. *Expansion*

The length variations of the mortar specimens are shown in Figs. 2–4 for the reference mortar, mortar A (containing calcium sulfate) and mortar S (containing sodium sulfate). The percentage expansions were calculated using the baseline measurement made at 28 days.

The reference mortar and mortar A did not show any expansion after 800 days, whether the mortars were heat-cured or not.

On the other hand, all the specimens of heat-cured mortars S expanded markedly after about 50 days whereas the specimens cured at room temperature did not. Some cracks visible to the naked eye appeared in heat-cured mortars for an expansion near 0.15%.

Table 2
Flexural and compressive strength at 28 days (MPa)

	Flexural strength	Compressive strength
C-NT-NP-H	7.2	58
C-T-NP-H	5.3	48
A-NT-NP-H	7.3	56
A-T-NP-H	5.7	51
S-NT-NP-H	6.8	50
S-T-NP-H	5.9	38

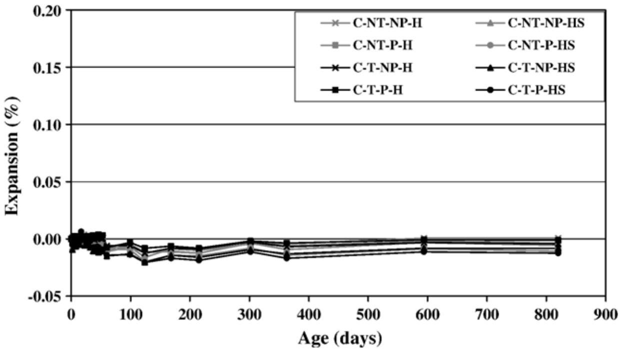

Fig. 2. Length variations of reference mortar (containing only cement).

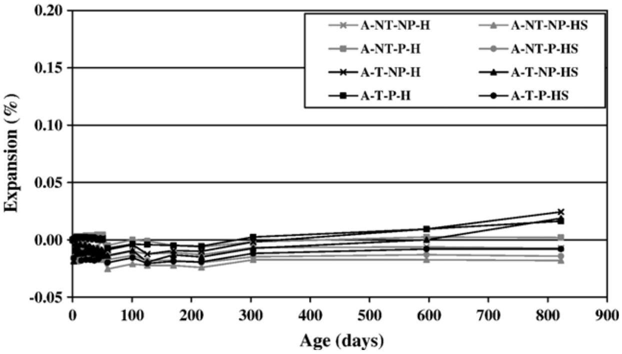

Fig. 3. Length variations of mortar containing cement +3% anhydrite.

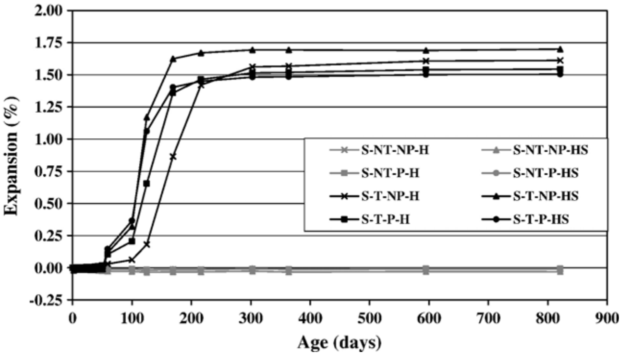

Fig. 4. Length variations of mortar containing cement +3.1% sodium sulfate.

For the heat-cured mortars, the supplementary treatments like preliminary microcracking or wetting/drying cycles modified the rate of expansion slightly (the wetting/drying cycles accelerated the reaction more than the preliminary microcracking) but did not influence the ultimate value of the expansion.

3.3. *Weight change*

The weight changes of the mortar specimens are presented in Figs. 5–7 for the reference mortar, mortar A and mortar S. The

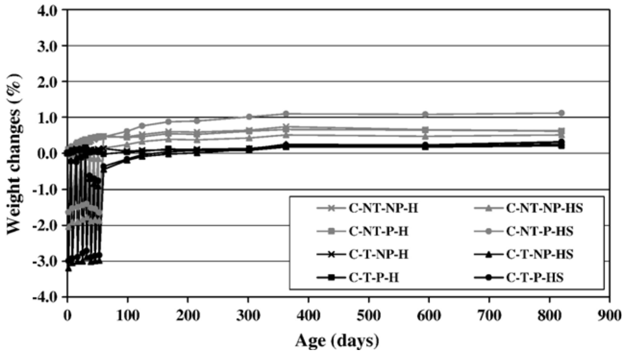

Fig. 5. Weight changes of reference mortars (containing only cement).

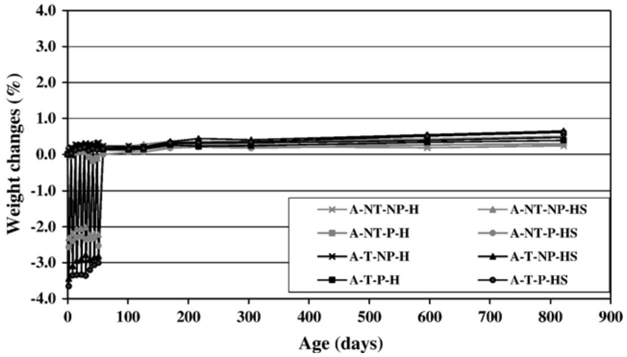

Fig. 6. Weight changes of mortar containing cement +3% anhydrite.

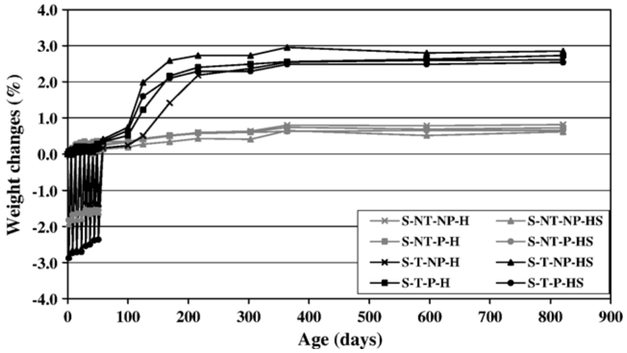

Fig. 7. Weight changes of mortar containing cement +3.1% sodium sulfate.

weight change was calculated using the baseline measurement at 28 days.

As was the case with the results of expansion, the weight of the reference mortar and mortar A did not change significantly (outside the period of wetting/drying cycles). The mortars S cured at room temperature had the same behavior.

On the other hand, the heat-cured specimens showed a significant weight gain that occurred at the same time as the expansion. The preliminary microcracking and the wetting/drying cycles slightly modified the rate of weight gain (wetting/drying cycles accelerated the weight gain more than preliminary microcracking did) but did not influence the ultimate value.

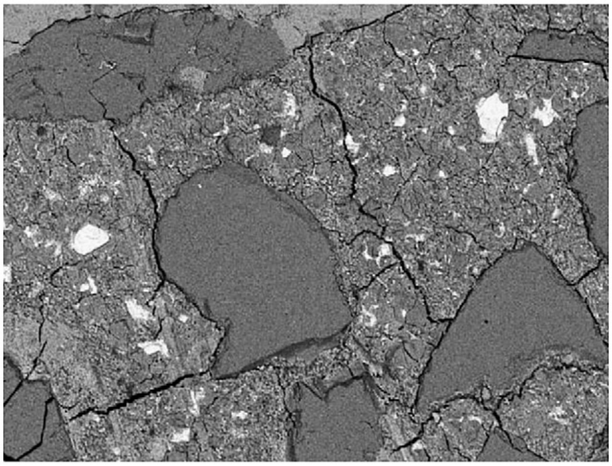

Fig. 8. BEI of reference mortar cured at room temperature (800 days, x200).

3.4. Scanning electron microscopy

In reference mortar cured at room temperature, ettringite was associated with portlandite in pores whereas, after thermal treatment, only portlandite was observed in pores or in paste/aggregate interfaces.

No delayed ettringite development can be observed on the 800-day-old reference mortars (Figs. 8 and 9). The empty cracks that can be clearly seen on the polished sections are the consequence of drying during sample preparation for the SEM. The same type of microstructure could be observed on mortar A.

Fig. 10 is representative of the mortars S cured at room temperature. No delayed ettringite formation can be observed either in the paste or at the paste/aggregate interfaces: the sulfur is evenly distributed in the paste (Fig. 11).

Figs. 12–14 are representative of the heat-cured mortars S. Delayed ettringite bands can be observed at most of the paste/aggregate interfaces and in the paste too. The width of ettringite rims is locally significant (Fig. 13). The elemental mapping of sulfur presented Fig. 15 shows the local concentration of ettringite. Moreover, EDS analyses of mortar show the lack of

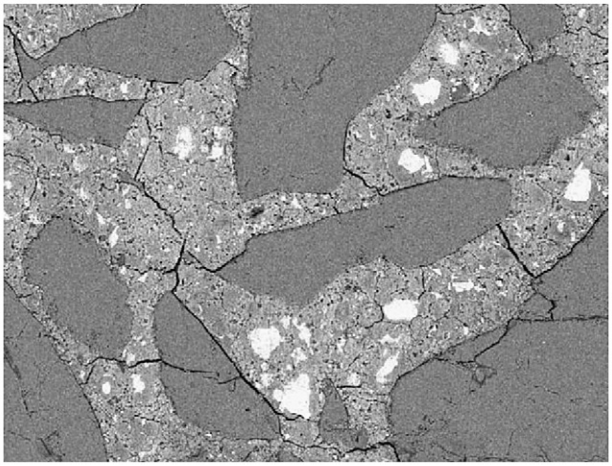

Fig. 9. BEI of heat-cured reference mortar (800 days, x200).

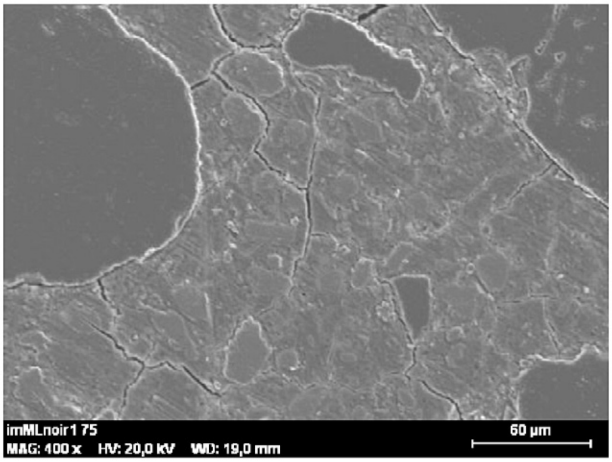

Fig. 10. SEI of mortar S cured at room temperature (800 days, x400).

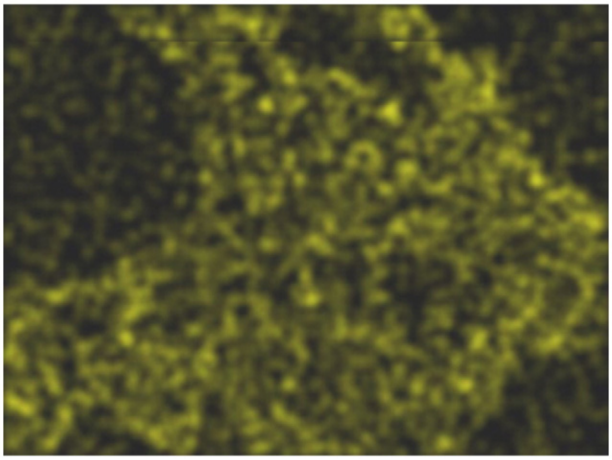

Fig. 11. Elemental mapping of Sulfur by EDS of the area of mortar S captured on picture presented Fig. 10.

alkali (and in particular sodium) in the composition of the cement paste (Fig. 16).

4. Discussion

The following points merit discussion:

- the effect of the chemical composition of the binder on the occurrence of delayed ettringite, by comparing our results with those found in the literature,
- the particular effect of alkali, by comparing the results of the mortars A and S,
- the effect of preliminary microcracking on the rate and the ultimate value of expansion.

Table 3 gives the chemical compositions of the three binders used during this study (reference mortar, mortar A and mortar S).

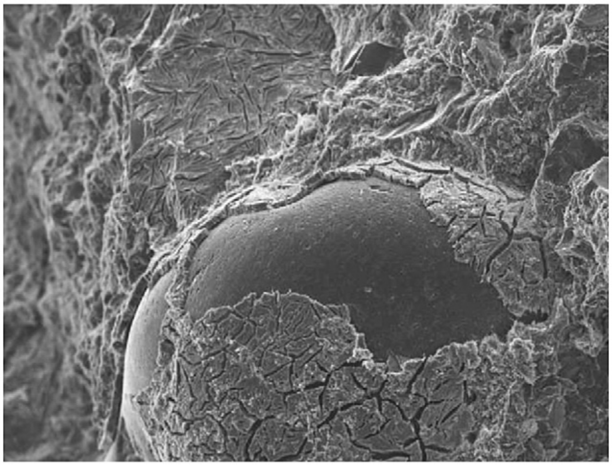

Fig. 12. SEI of heat-cured mortars S: delayed ettringite at the paste/aggregate interface (800 days, x200).

Numerous researchers have studied the influence of composition on cement's susceptibility to DEF. In the literature, SO_3 and the SO_3/Al_2O_3 molar ratio are often considered to be the preponderant factors [10, 23–26]. In two recent papers [27,28], Zhang et al. have studied the relation between DEF-affected mortars and the SO_3/Al_2O_3 molar ratio. Their conclusions are based upon their own results and those of Fu [20] and Famy [30]. The results of Zhang et al. show that expansion occurs for mortars made from cements with molar ratios falling between 0.85 and 1.4, which is in accordance with the results of other authors. The results of the present study show that the SO_3 content and SO_3/Al_2O_3 molar ratio are not sufficient to predict the occurrence of DEF. In fact, these factors were the same for mixtures A and S whereas their behavior was quite different (no expansion in the first case, high expansion in the second). In their paper [28], Zhang et al. propose a DEF-Index defined by the following equation:

$$\text{DEF index} = (SO_3/Al_2O_3)_m \times [(SO_3 + C_3A)_w/10] \times \sqrt{Na_2O_{eq}}$$

where $(SO_3/Al_2O_3)_m$ is the molar ratio of SO_3 to Al_2O_3 in the cement; $(SO_3+C_3A)_w$ is the combined weight percentage of SO_3

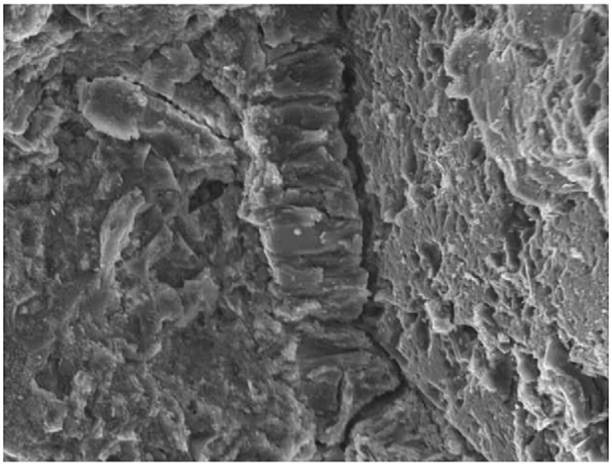

Fig. 13. SEI of heat-cured mortars S: delayed ettringite at the paste/aggregate interface (800 days, x1000).

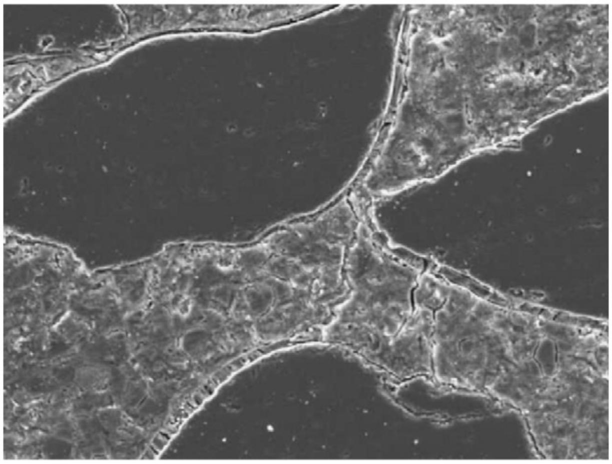

Fig. 14. SEI on polished section of heat-cured mortars S: delayed ettringite at the paste/aggregate interface (800 days, x400).

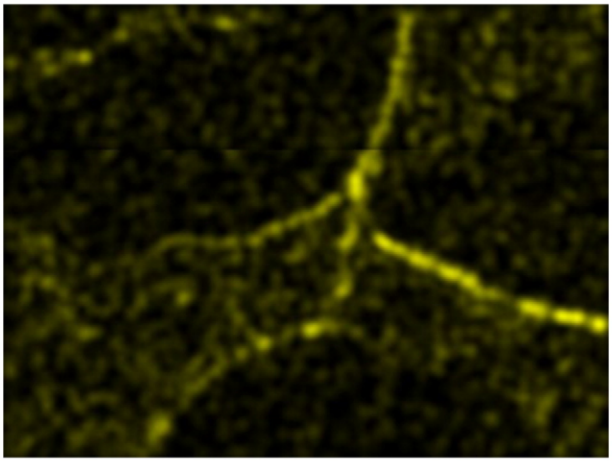

Fig. 15. Elemental mapping of sulfur of the area of mortar S captured on picture presented Fig. 14, showing the local concentration of ettringite.

and Bogue-calculated C_3A; and $\sqrt{Na_2O_{eq}}$ is the square root of the weight percentage of Na_2O_{eq}.

Zhang et al. conclude that there is no expansion for an index below 1.1 and that there is always expansion above this value. Once again, the results of the present study do not confirm those of Zhang et al. because the mortar A did not expand although its DEF-Index was equal to 1.5.

The results reported in this paper show that the alkali content is a significant parameter in the occurrence of delayed ettringite, which is in accordance with the conclusions of numerous authors [4,16,26,28]. In a review on delayed ettringite formation, Taylor et al. [4] discuss the effects of the addition of alkalis in the initial mix (in the form of KOH and K_2SO_4). Taylor et al. base their discussions on the work of Kelham [26], the conclusion of which is that expansion is favored by high alkali contents for cement high in SO_3 and that alkali has no significant effect in the case of low SO_3 cement. According to Taylor et al., at least two mechanisms could explain these observations. First, increased alkali content tends to increase the rate of alite hydration at early ages that could increase the expansion due to the DEF according to Taylor et al. Secondly, the presence of alkali increases the pH of the pore solution, which favors the presence of monosulfate rather than ettringite, with relatively high contents of sulfate in the C–S–H and the pore solution. In a same way, Divet and Randriambololona [14] have shown that increased basicity of the pore solution favors the adsorption of sulfates on C–S–H. The lowering of the basicity of the pore solution can be one of the reasons for the apperance of DEF. It could happen during ASR (as studied for example by Brown and Bothe [29]) or during the storage of specimens in water, the consequence of which is the leaching of alkalis. Diamond [15], who draws his inferences from the works of Famy [30] and Zhang [31], agrees with these conclusion and postulates: “leaching of alkali hydroxide from the pore solutions within the mortar specimens is required as a trigger for the sequence of responses that result in DEF derived expansions and other symptoms of distress”.

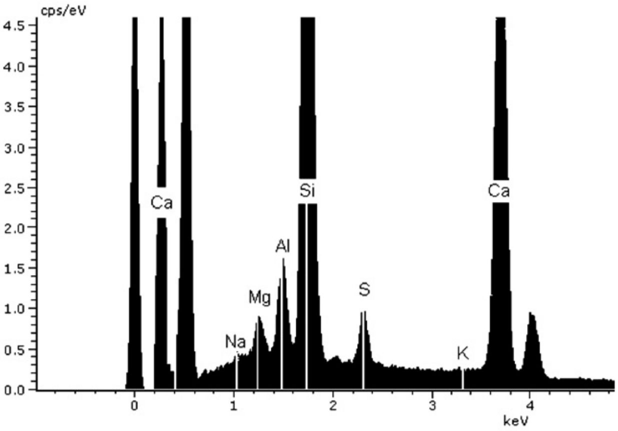

Fig. 16. EDS analyses of the area of mortar S captured on picture presented Fig. 14.

The mechanisms linked to a high alkali content that occur during DEF cannot be explained by the results of the present work but they confirm that high levels of alkali increase the risk of expansion after heat curing, as has already been widely demonstrated. Further work is required to better understand these mechanisms.

As stated in the introduction, pre-existing microcracking could, according to some authors, be a significant, and indeed

Table 3
Analyses of the “binders” used

	Cement	Cement+3% $CaSO_4$	Cement+3.1% Na_2SO_4
SiO_2 (%)	19.2	18.7	18.6
CaO (%)	64.3	63.6	62.3
MgO (%)	2.9	2.8	2.8
Al_2O_3 (%)	5.1	5.0	5.0
Fe_2O_3 (%)	2.5	2.4	2.4
K_2O (%)	0.7	0.7	0.7
Na_2O (%)	0.3	0.3	1.6
SO_3 (%)	3.4	5.0	5.0
SO_3/Al_2O_3 (molar ratio)	0.8	1.3	1.3
DEF index	0.9	1.5	2.6

necessary, factor for the occurrence of delayed ettringite. The results of this study do not confirm this and show that preliminary microcracking (thermal shrinkage or wetting/drying cycles) only accelerates the phenomenon. These results are in accordance with those obtained by Petrov and Tagnit-Hamou [32]. These authors, who worked on mortars and concrete, showed that microcracking caused by thermal shock did not promote expansion. On the contrary, this microcracking decreased the ultimate value of expansion. Petrov and Tagnit-Hamou conclude that the microcracks increase the availability of water and so accelerate the occurrence of delayed ettringite. Following the argument of Diamond [15], the microcracking of cement-based materials susceptible to DEF would accelerate the leaching of alkalis because the water could penetrate into the pores more easily. So, the alkalis would leave the specimens faster, allowing faster formation of the delayed ettringite.

5. Conclusions

The main conclusions of this study on Delayed Ettringite Formation in heat-cured mortars stored in water are:

1. Thermal treatment is one of the factors indispensable for the occurrence of delayed ettringite. This has been well established and was one of the principal conclusions of the RILEM workshop [1]. So, from a practical point of view, the recommendations aiming to limit the temperature reached in the concrete seem to be justified.
2. Supplementary treatments, such as preliminary microcracking or wetting/drying cycles, are not necessary for the occurrence of DEF but they contribute to a slight acceleration of the reaction. Moreover, they do not modify the ultimate value of the expansion.
3. The type of sulfated addition is a very important factor in the occurrence of delayed ettringite. No expansion was observed after 800 days for the reference mortar or for the mortar containing calcium sulfate, in contrast to the case for the mortar containing sodium sulfate. The difference between additions of calcium sulfate and sodium sulfate is due to the sodium ions contributing to the pore solution chemistry. The results of the present work confirm that high levels of alkali increase the risk of expansion after heat curing. Sodium sulfate can be present in large amounts in the waste wash water coming from concrete plants. So, it is necessary, as recommended in the standard NF EN 1008, to limit the sulfate and alkali content of this type of water reused in concrete plants.

The DEF mechanisms have still not been totally elucidated and, in particular, the precise role of alkalis. Other experiments will be necessary to study the effects of alkalis on DEF like, for example, the link between their leaching and the occurrence of delayed ettringite in heat-cured mortars. This will involve new experiments based not only on the measurement of expansion but also on the study of the evolution of the storage solution and the pore solution by chemical analyses. Moreover, numerous parameters that control the leaching behavior would have to be considered, such as the shape, size and porosity (for example due to preliminary microcracking) of the specimens, and the nature of the storage solution. These laboratory studies may not predict the behavior of field concretes but their results could help to better understand the DEF mechanisms and the relative importance of the different parameters governing the occurrence of delayed ettringite in heat-cured cement-based materials.

References

[1] Internal sulfate attack and delayed ettringite formation, in: K. Scrivener, J. Skalny (Eds.), Proceedings of the International RILEM TC 186-ISA Workshop, RILEM Publications, Villars, Switzerland, 2002.
[2] J. Skalny, Internal sulfate attack — points of agreement and disagreement, in: K. Scrivener, J. Skalny (Eds.), Proceedings of the International RILEM TC 186-ISA Workshop, RILEM Publications, Villars, Switzerland, 2002, pp. 265–275.
[3] S. Diamond, Delayed ettringite formation — processes and problems, Cement and Concrete Composites 18 (1996) 205–215.
[4] H.F.W. Taylor, C. Famy, K.L. Scrivener, Delayed ettringite formation, Cement and Concrete Research 31 (2001) 683–693.
[5] P.K. Mehta, Sulfate attack on concrete — separating myths from reality, Proceedings of Fifth CANMET/ACI International Conference of Durability of Concrete, Supplementary Papers, Barcelona, Spain, 2000, pp. 1–12.
[6] M. Collepardi, Ettringite formation and sulfate attack on concrete, Proceedings of Fifth CANMET/ACI International Conference of Durability of Concrete, Supplementary Papers, Barcelona, Spain, 2000, pp. 25–41.
[7] M. Collepardi, A state-of-the-art review on delayed ettringite attack on concrete, Cement and Concrete Composites 25 (2003) 401–407.
[8] Y. Fu, J.J. Beaudoin, Mechanisms of delayed ettringite formation in Portland cement systems, ACI Materials Journal 93 (4) (1996) 327–333.
[9] Y. Fu, J.J. Beaudoin, Microcracking as a precursor to delayed ettringite formation in cement systems, Cement and Concrete Research 26 (10) (1996) 1493–1498.
[10] E. Grabowski, B. Czarnecki, J.E. Gillott, C.R. Duggan, J.F. Scott, Rapid test of concrete expansivity due to internal sulfate attack, ACI Materials Journal 89 (5) (1992) 469–480.
[11] A. Pavoine, L. Divet, Delayed ettringite formation in concrete: tests based on drying and wetting cycles, Proceedings of sixth CANMET/ACI international conference of durability of concrete, Greece, 2003, pp. 989–1003.
[12] D. Damidot, F.P. Glasser, Thermodynamic investigation of the CaO–Al2O3–CaSO4–H2O system at 25 °C and the influence of Na2O, Cement and Concrete Research 23 (1) (1993) 221–238.
[13] F.P. Glasser, The role of sulfate mineralogy and cure temperature in delayed ettringite formation, Cement and Concrete Composites 19 (1996) 187–193.
[14] L. Divet, R. Randriambololona, Delayed ettringite formation: the effect of temperature and basicity on the interaction of sulphate and C–S–H phase, Cement and Concrete Research 28 (3) (1998) 357–363.
[15] S. Diamond, The revelance of laboratory studies on delayed ettringite formation to DEF in field concretes, Cement and Concrete Research 30 (2000) 1987–1991.
[16] C. Famy, K.L. Scrivener, A. Atkinson, A.R. Brough, Influence of the storage conditions on the dimensional changes of heat-cured mortars, Cement and Concrete Research 31 (2001) 795–803.
[17] Y. Shimada, J. Francis Young, Thermal stability of ettringite in alkaline solutions at 80 °C, Cement and Concrete Research 34 (2004) 2261–2268.
[18] D. Mc Donald, Delayed ettringite formation and heat curing — implications of the work of Kelham, Cement and Concrete Research 28 (12) (1998) 1827–1830.
[19] P.E. Grattan-Bellew, A discussion of the paper “delayed ettringite formation in heat-cured Portland cement mortars” by R. Yang, C.D. Lawrence, C.J. Lynsdale, J.H. Sharp, Cement and Concrete Research 30 (2000) 665–666.
[20] Y. Fu, J. Ding, J.J. Beaudoin, Expansion of Portland cement mortar due to internal sulfate attack, Cement and Concrete Reasearch 27 (1997) 1299–1306.

[21] P.E. Grattan-Bellew, J.J. Beaudoin, V.-G. Vallée, Effect of aggregate particle size and composition on expansion of mortar bars due to delayed ettringite formation, Cement and Concrete Research 28 (8) (1998) 1147–1156.

[22] R. Yang, C.D. Lawrence, J.H. Sharp, Effect of type of aggregate on delayed ettringite formation, Advances in Cement Research 11 (3) (1999) 119–132.

[23] J. Stark, K. Bollman, K. Seyfarth, Investigation into delayed ettringite formation in concrete, Proceedings of the nineth international congress on the chemistry of cement, New Dehli, 1992, pp. 348–354.

[24] I. Odler, Y. Chen, Effect of cement composition on the expansion of heat-cured cement pastes, Cement and Concrete Research 25 (4) (1995) 853–862.

[25] C.D. Lawrence, Mortar expansions due to delayed ettringite formation. Effects of curing period and temperature, Cement and Concrete Research 25 (4) (1995) 903–914.

[26] S. Kelham, The effect of cement composition and fineness on expansion associated with delayed ettringite formation, Cement and Concrete Composites 18 (1996) 171–179.

[27] Z. Zhang, J. Olek, S. Diamond, Studies on delayed ettringite formation in early-age, heat-cured mortars I. Expansion measurements, changes in dynamic modulus of elasticity, and weigth gains, Cement and Concrete Research 32 (2002) 1729–1736.

[28] Z. Zhang, J. Olek, S. Diamond, Studies on delayed ettringite formation in early-age, heat-cured mortars II. Characteristics of cement that may be susceptible to DEF, Cement and Concrete Research 32 (2002) 1737–1742.

[29] P.W. Brown, J.V. Bothe, The stability of ettringite, Advances in Cement Research 5 (18) (1993) 47–63.

[30] C. Famy, Expansion of heat-cured mortars, PhD thesis, Imperial College of Science, Technology and Medicine, University of London, 1999.

[31] Z. Zhang, Delayed ettringite formation in heat-cured cementitious systems, PhD thesis, Purdue University, 1999.

[32] N. Petrov, A. Tagnit-Hamou, Is microcracking really a precursor to delayed ettringite formation and consequent expansion? ACI materials journal (2004) 442–447.