Available online at www.sciencedirect.com

ScienceDirect

Cement and Concrete Research 36 (2006) 2144 – 2151

Cement and Concrete Research

A concrete performance test for delayed ettringite formation: Part II validation

Alexandre Pavoine *, Loïc Divet, Stéphane Fenouillet

Laboratoire Central des Ponts et Chassées, 58 Bd Lefebvre, 75732 Paris Cedex 15-France

Received 25 November 2004; accepted 18 September 2006

Abstract

Delayed ettringite formation (DEF) is a rare problem of concrete, whose reaction mechanisms have been investigated by a large number of studies. In order to develop a performance test, the authors have conducted a feasibility study and an optimization study followed by this validation study. A performance test was previously developed to evaluate the risk of expansion as a result of DEF for a given "concrete/heating" combination to be evaluated. This paper presents the results of the validation study and explains the necessary conditions that must be applied for this test to be used as part of a rigorous preventive procedure. Data has been collected to reproduce the heat development in concrete under actual conditions on site or in a prefabrication factory. Concrete/heating combinations were studied for which 10 or 20 years' experience of the use of the concrete in wet environments existed. After nearly 300 days of testing, all the laboratory results reproduced the behaviour observed in-situ. On the basis of these macroscopic measurements and microscopic observations, DEF susceptible concretes can be distinguished from concretes that have never caused problems, despite being heated to 80 °C in early age. This test program therefore, confirms the reliability of the proposed performance test.
© 2006 Elsevier Ltd. All rights reserved.

Keywords: Delayed ettringite formation; Expansion; Sulfate attack

1. Introduction

Delayed ettringite formation (DEF) is a chemical reaction which can cause damage to concrete. The expansion mechanism of this reaction is complex, and involves many parameters. As a result of a large number of laboratory studies and experience gained in the field means, progress has been made towards understanding the possible causes of this [1], and several recommendations have been made with a view to the long-term prevention of DEF [2–7] . These recommendations lay down constraints with regard to concrete mix design or heating in relation to the environment and the size of the member.

The reaction mechanism of DEF involves a large number of parameters whose importance is more or less understood. The most influential parameters are temperature [8–10], cracking [11–13], the alkali content of the cement [10,14,15], the sulfate content of the cement [11,16,17] and the presence of water [2]. The risk may be reduced if at least one of these factors is absent or constrained. However, we are unable to establish practical limits for these parameters due to the lack of field data. For this reason, recommendations are usually based on the precautionary principle.

To meet the expectations of the owners of structures, contractors and concrete manufacturers, it is necessary to develop tests to evaluate the susceptibility of concrete to DEF without waiting for all the reaction mechanisms to be fully understood. Several tests have been proposed in the literature [18–20], which are essentially based on subjecting cement pastes or mortars to wetting and drying cycles. Because high temperatures are applied on one or more occasions after the initial curing and because of the relative small size of the specimens, the results of these tests do not always reflect reality. More recent research [21] appears to confirm that a satisfactory performance test can be conducted when concrete temperature doesn't exceed 60 °C after the initial curing and hardening. It's important to note that the temperature mentioned here, is the 'test temperature' not the temperature imposed on the concrete during hardening.

The feasibility of a test based on wetting and drying cycles was presented in a previous paper [22] and part 1 of this paper presents an optimization study for a test method for concrete specimens [23]. This method has the advantage that the specimens used are sufficiently large to limit alkali leaching. It also showed that a

* Corresponding author.
E-mail address: alexandre.pavione@lcpr.fr (A. Pavoine).

0008-8846/$ - see front matter © 2006 Elsevier Ltd. All rights reserved.
doi:10.1016/j.cemconres.2006.09.010

maximum test temperature of 38 °C is appropriate, as drying at 60 °C is not appropriate because it results in the expansion of a concrete that remains healthy in-situ.

The proposed test involves four stages

1. Manufacture of concrete specimens with a real mix design;
2. A heat treatment that simulates heat treatment in a factory or the temperature rise that takes place in mass cast in-situ concrete;
3. Two cycles of wetting and drying, each cycle taking 14 days and consist of the following two phases: drying for 7 days in an enclosure at 38 °C with a relative humidity of 30% followed by immersion for 7 days in tap water at 20 °C.
4. Monitoring longitudinal expansion of the specimens immersed in water at 20°C [23].

The performance test must be validated in order for it to be used in the framework of a rigorous preventive approach. At the present time, the duration of test is not fixed. This duration should be stated in the next few months with the results of a repeatability study conducted with several laboratories and a large experimental study.

Validation necessitates the study of concrete that has been heated (to more than 70 °C) at early age and whose long term in-situ performance is thoroughly known. To do this, we have selected four "concrete mix design and heating cycle" combinations which are representative of concrete members manufactured in a factory or massive parts of structures. On site, these members have been exposed to a wet environment for more than 10 years and we therefore know from experience their durability with respect to DEF. For this study, two types of concrete were manufactured and investigated:

- concretes which are representative of the materials used in mass structural members which have suffered DEF-induced damage [24],
- concretes which are representative of members that are prefabricated in a factory which have never exhibited any problems due to DEF.

Tests were also conducted on core samples taken from both sound and damaged parts of a structure to see whether the performance test can also provide a means of assessing the potential for future expansion of in-situ concrete for diagnosis/prognosis purposes.

2. Experimental description

For this study, six cylindrical concrete specimens (110× 220 mm) were manufactured for each "concrete mix design and heating cycle" pair. These were divided into two batches:

- 3 specimens were subjected to the performance test, i.e., two wetting and drying cycles followed by permanent immersion;
- 3 specimens were directly immersed permanently in water. These are referred to as the control specimens (without cycles).

The control specimens (without cycles) were monitored in order to evaluate how the wetting and drying cycles affected expansion of the concrete.

2.1. Manufacture of concrete representative of massive structural members

2.1.1. Concrete mix design

Two types of concrete, designated "Bridge 1" and "Bridge 2" were investigated. Details of the mix design were obtained from the construction records of the structures concerned. Concrete specimens were manufactured using ingredients that were identical or similar to those used in the structure. The aggregates were taken

Table 1
Concrete mix design

"Bridge 1"			"Bridge 2"		"Prefab 1"		"Prefab 2"	
Material	Kg/m^3		Material	Kg/m^3	Material	Kg/m^3	Material	Kg/m^3
Siliceous aggregate 10/20	810	Quartz (30%) Feldspaths 20% Oxides 30% Phylliteous as chlorite 20%	Siliceous aggregate 14/20	640	Sillicalcareous aggregate 4/12,5	855	Sillicalcareous aggregate 4/12	1196
Siliceous aggregate 4/10	395		Siliceous aggregate 10/14	130				
			Siliceous aggregate 6/10	290				
Siliceous sand 0/4	555		Siliceous sand	800	Sillicalcareous sand 0/4	925	Sillicalcareous sand 0/5	605
Calcareous sand 0/3	140							
CEM I cement	350		CEM I cement	350	CEM I cement PM-ES*	400	CEM I cement	377
Water	165		Water	190	Water	180	Water	156
					Additive (plasticizer)	3,5	Additive (plasticizer)	4

Proportioning in kg/m^3.
*French label for low SO_3 and C_3A cement.

from the same quarries. These aggregates were classified as non alkali-reactive by several reactivity tests [25]. The siliceous sand and aggregates are made of quartz (30%), feldspars (20%), phyllitic minerals as chlorite (20%) and various oxides (30%). The cements were procured from the same factories and care was taken to ensure that their mechanical, physical, chemical and mineralogical characteristics resembled those used at the time the structures were built. Tables 1, 2 and 3 set out the concretes mix design and the cements composition.

It is important to mention that not all parts of the structures were affected by DEF. The only parts so affected were a cross head in Bridge 1 and a pier in Bridge 2. These are both massive members which had been cast in summer. They are also both exposed to a high level of humidity or the presence of water in view of their location in a tidal zone.

This localization of damage, in a structure where only one mix design was used throughout supports the view that AAR was not responsible for the degradation of these concretes.

2.1.2. Estimation of the heat development at the core of massive members

To carry out the performance test for real cases, it is necessary not only to cast concrete specimens using materials that are identical to those in the structure but also to subject them to the same heating cycle that existed within the mass member. To estimate this parameter, we modelled the temperatures attained in the concretes during the construction of the mass members, by means of a finite element computation using the LCPC CESAR program, in particular the TEXO module of this program. In order to specify the characteristics of heating over time, the following parameters were needed:

- the thermal properties of the concrete;
- the initial temperature of the materials, the site and the members in contact with the concrete. The calculation generally uses the external temperature;
- the conditions of heat exchange (formwork, hardened concrete, soil, air, etc.);

Table 2
Chemical composition of the Portland cements (% by weight)

Constituent	"Bridge 1"	"Bridge 2"	"Prefab 1"	"Prefab 2"
SiO_2	20.42	20.09	20.60	20.24
Al_2O_3	4.36	4.36	3.40	5.18
TiO_2	0.26	0.26	0.42	0.29
Fe_2O_3	2.55	3.59	4.03	2.28
CaO	63.56	62.86	65.00	64.45
MgO	1.15	1.33	2.04	1.00
Na_2O	0.17	0.15	0.10	0.11
K_2O	1.03	1.54	0.44	1.19
SO_3	3.15	3.52	2.65	3.70
P_2O_5			0.19	0.21
Cl	<0.01	<0.01	0.02	0.02
S_2	Néant	Néant	Néant	Néant
Insoluble residue	1.15	0.39	0.15	0.14
Loss on heating to 975 °C	0.91	1.43	0.49	1.14
MnO	0.02	0.07	0.04	0.05
Na_2Oeq*	0,85	1.16	0.39	0.89

*Na_2O+0,658*K_2O.

Table 3
Bogue computations of chemical and mineralogical composition of the Portland cements (%)

	"Bridge 1"	"Bridge 2"	"Prefab 1"	"Prefab 2"
C_3S	51.2	51.0	72.0	60.0
C_2S	19.9	18.9	4.7	12.7
C_3A	7.2	6.1	2.2	9.9
C_4AF	7.7	10.9	12.2	6.9

- the dimensions of the concrete member;
- how the concrete member was constructed (number of layers, phasing, etc.).

The thermal properties of the concrete in the course of hydration were determined using an adiabatic test. This was conducted on a concrete mixture whose formulation (aggregate, fines, cement, water, additives) was representative of that used on the site.

The finite element calculation, based on classical laws of heat flow and hydration processes, was then performed using this data. The calculation quantified the binder hydration process and temperature change throughout a concrete member. The uncertainty of the temperatures calculated by this software is of the order of 10% [26].

The calculations showed that similar temperatures rises occurred in the core of the cross head in Bridge 1 and the pier in Bridge 2 (Fig. 1). The duration of heating and the temperatures attained during the heating cycle were representative of those recorded in mass members. The core of the concrete reached a temperature close to 80 °C after 15 h of hydration and remained at temperatures of above 70 °C for more than three days.

For the laboratory tests, a heat treatment was applied that was similar to the results of the numerical computation (Fig. 1). However, the cooling phase was accelerated to avoid thermal shock on removal from the controlled environment enclosure (20 °C) after 14 days of treatment.

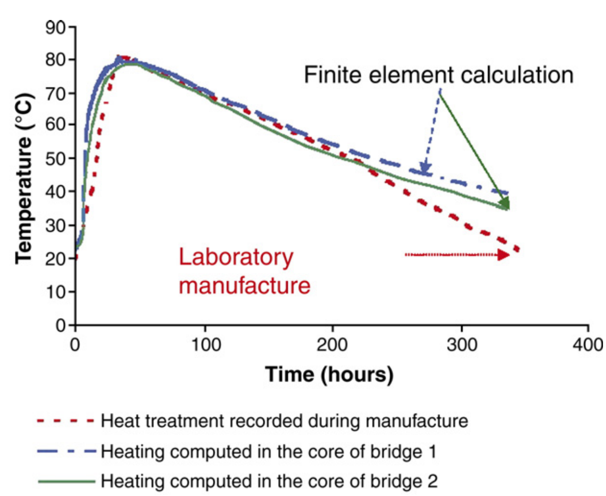

Fig. 1. Heat treatment applied to the concrete specimens that are representative of massive structural members. Comparison with the temperatures calculated in the core of the studied concrete members from Bridge 1 and 2.

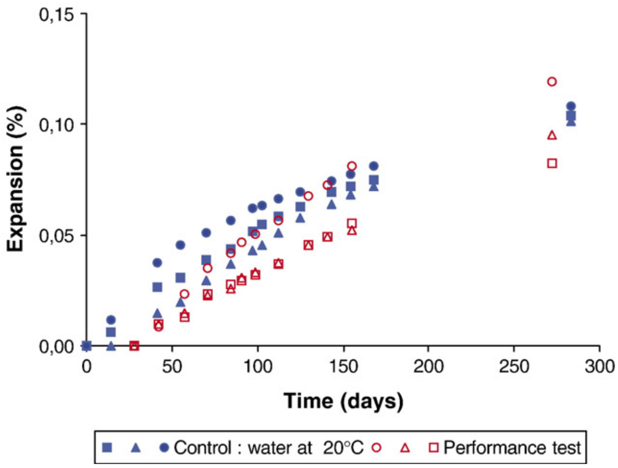

Fig. 2. Bridge 1. Expansion of three specimens subjected to the performance test and three control specimens (without cycles) stored in water at 20°C. t_0=end of heat treatment.

2.2. Manufacture of concrete representative of prefabricated members

Two concrete mixtures were used that are representative of those used in the manufacture of pipes or gutters. In the practice, these concretes are subjected to an optimized heat curing with a maximum temperature of 80 °C and then exposed to a moist environment during use. They have demonstrated good durability over a period of more than 10 years of service. These concretes are referred to as "Prefab 1" and "Prefab 2". The concrete mix design and the composition of the cements used to manufacture concrete specimens are given in Tables 1, 2 and 3. The heat treatment used for these concretes consisted of the four following stages:

- Pretreatment phase: 30 min at 20 °C;
- Temperature increase: 2 h at a rate of 30 °C/h;
- Maintenance at the plateau temperature: 1 h at 80 °C;
- Cooling phase: 30 min to reach a final temperature of 45 °C.

2.3. Core samples of concrete taken from the structure

Several core samples of concrete were removed from Bridge 1. One cross head in this bridge has suffered from a localized delayed ettringite formation [15]. The core samples were taken from zones of the cross head which exhibited varying degrees of cracking and in a non-cracked zone of a pier which did not show signs of delayed ettringite formation.

The core samples were 100 mm in diameter and 220 mm long. For the tests, samples were taken nearest to the core of the members, i.e., zones which had undergone the greatest heating.

2.4. Measurements and examinations

Expansion measurements were made with a Pfender roller extensometer. The average expansion measured on 3 locations placed at 120 degrees from each other around the diameter of each concrete core specimen has been plotted.

Examination with a scanning electron microscope and an energy dispersion spectrometer were conducted on both polished sections and fresh fractures. Numerous examinations were conducted at various intervals during the test. It is important to note that a specific polishing protocol using alcohol was used to limit the risk of leaching.

3. Results

3.1. Concrete representative of mass members in structures affected by DEF

3.1.1. The monitoring of expansion

In the case of Bridge 1, monitoring of the control core specimens (not subjected to cycles) revealed considerable expansion after 250 days of immersion (Fig. 2). This expansion reached a level of 0.1% and may show the existence of an internal swelling reaction. The concrete specimens that were subjected to the performance test had similar macroscopic behaviour and showed an expansion of 0.10±0.01% after 250 days of testing.

In the case of the control specimens (without cycles) that were representative of Bridge 2, the expansion plateau was reached after 150 days of immersion (Fig. 3). The characteristic parameters of the expansion curve [23,27], show that this concrete is extremely susceptible to DEF (the latency time [23] was reduced to 87 days and the magnitude of expansion was 1.6%). Monitoring of the three specimens showed that the average difference between the expansion of the three specimens was low, only 0.03% after 170 days. The concrete specimens that were subjected to the performance test were also highly susceptible to DEF. The magnitude of expansion (1.5%) was close to the level measured on the control specimens (without cycles), for which the average difference between the expansion of the three specimens was greater, 0.08% after immersion for 150 days. However, this difference is still small considering the kinetics and large magnitude of expansion exhibited by specimens.

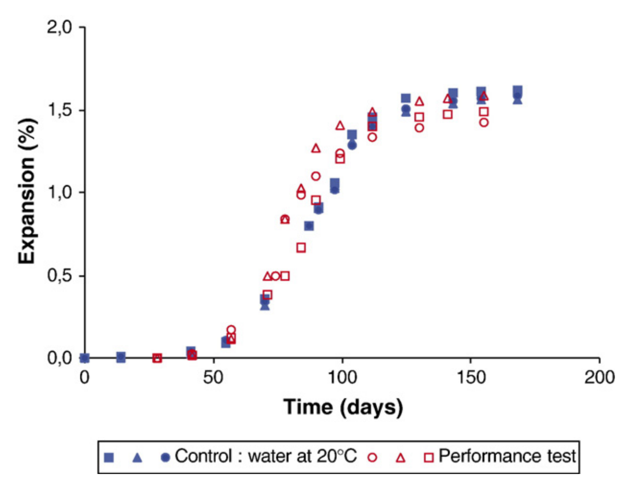

Fig. 3. Bridge 2. Expansion of three specimens subjected to the performance test and three control specimens (without cycles) stored in water at 20 C°. t_0=end of heat treatment.

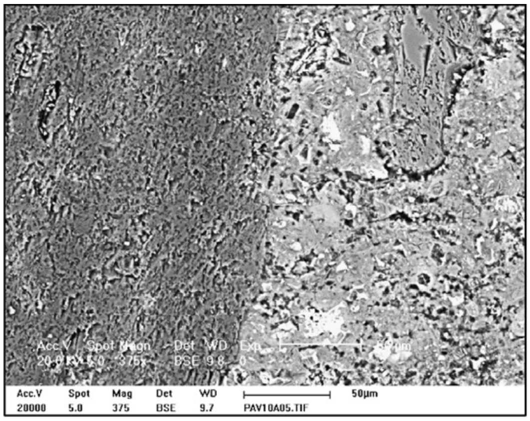

Fig. 4. Scanning electron micrographs of concrete with average longitudinal expansion of 0.01%.

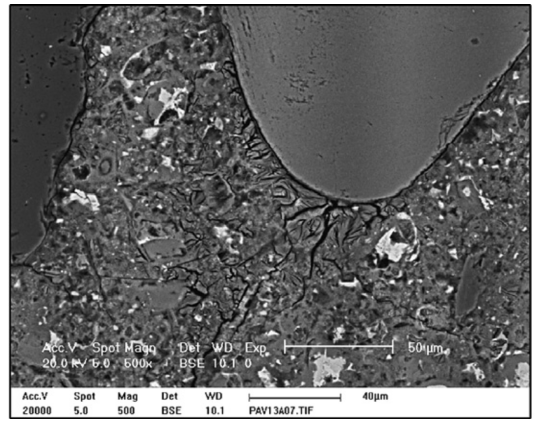

Fig. 6. Scanning electron micrographs of concrete with average longitudinal expansion of 0.44%.

3.1.2. Microscopic study

To determine the cause of the expansion, additional specimens representative of Bridge 1 were used. These specimens were subjected to cycles after heat treatment and examined with scanning electron microscope at different stages of expansion (Figs. 4–9).

After the heat treatment (24 h), the microscopic observations do not show the presence of any deleterious substances (Figs. 4 and 5). The concrete exhibited good cohesion within the cement matrix and at the aggregate/cement paste interfaces. A small amount of ettringite was observed in the form of isolated randomly distributed clusters within the cement paste. Generally only few cracks were observed, which were not associated with a localized concentration of ettringite.

Examination of specimens after 42 days of immersion, with an average expansion of 0.44% revealed important changes (Figs. 6, 7) compared with the specimens before the expansion phases. The presence of ettringite both in the cement paste and at the aggregate/paste interfaces was clearly evident. Some pores and cracks were also filled with. The ettringite wasn't only present in dispersed zones but as veins running through the paste. The cohesion between aggregate particles and the cement paste was good even though a given particle may have been enveloped by ettringite at proximity.

In the case of concrete whose average expansion was 0.81% (49 days immersion), ettringite was seen around most of the aggregate particles and within the cement paste (Figs. 8 and 9). The large masses of ettringite were preferentially located as bands of up to 50 μm thicken, at the aggregate/paste interfaces. The considerable reduction in binder cohesion was characterized by the appearance of a large number of cracks in all the specimens.

3.2. Concretes elements heat cured at 80 °C but without any in-situ durability problems

The expansion curves of the concrete specimens Prefab 1 and Prefab 2 are given in Figs. 10 and 11. Monitoring of the specimens that have not been subjected to cycles showed that these concretes did not expand during the phase of permanent immersion. Their measured expansion was small (less than 0.02%) and is attributed to water absorption during immersion. The results from the performance tests were similar; no significant expansion being measured after 300 days of

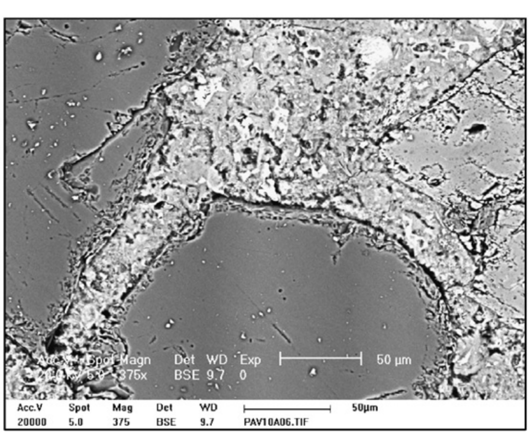

Fig. 5. Scanning electron micrographs of concrete with average longitudinal expansion of 0.01%.

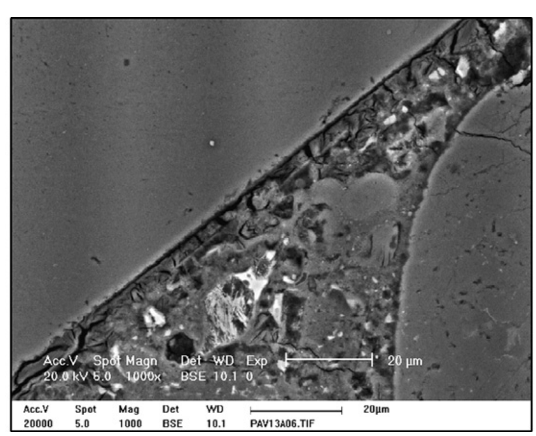

Fig. 7. Scanning electron micrographs of concrete with average longitudinal expansion of 0.44%.

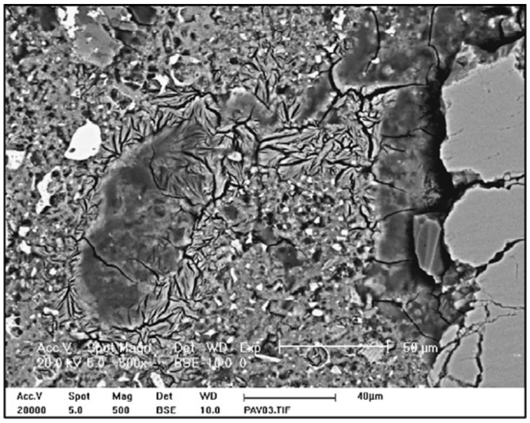

Fig. 8. Scanning electron micrographs of concrete with average longitudinal expansion of 0.81%.

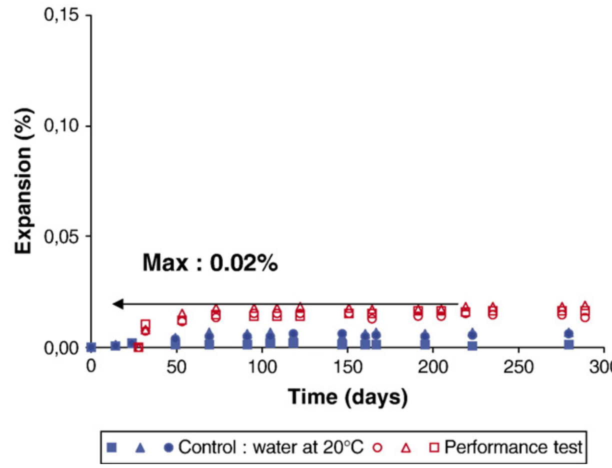

Fig. 10. *Prefab 1.* Expansion of three specimens subjected to the performance test and three control specimens (without cycles) stored in water at 20°C. t_0=end of heat treatment.

monitoring. Scanning electron microscope examination of the samples did not show the presence of significant amounts of ettringite.

3.3. Core samples of concrete taken from Bridge 1

Representative expansion curves for concrete core samples are given in Fig. 12. The behaviour of the core samples from Bridge 1 depends on their locality. A large expansion was observed for the specimens taken from slightly-cracked zones in the crosshead, whereas the samples taken from highly-cracked zones of the crosshead or crack-free zones of the pier did not expand during the test.

The reason that the very highly-cracked zone of this bridge did not expand is probably because it has attained the expansion plateau. However, the defects observed in the zones with little cracking are likely to become more serious if exposure to water is not restricted. Lastly, the sound parts of the structure (the piers) do not seem to have any potential for DEF. The explanation for this is that, with the same mix design, the concrete was subjected to less heating as it was not manufactured in the summer and the member had a shape which promoted heat loss.

4. Discussion

Tests conducted in laboratory on concrete specimens which had developed a delayed ettringite formation in service was found to be representative of behaviour in the field. It has been noted that applying wetting and drying cycles is not always justified as they do not systematically accelerate the phenomenon. These cycles don't cause any unrealistic expansion. In fact, the final magnitude of expansion was identical for the control specimens (without cycles) and those which underwent the test. However, in some cases, these cycles accelerate the expansion process [23].

Concrete cores taken from part of a structure which has exhibited expansion in-situ also expanded during the laboratory

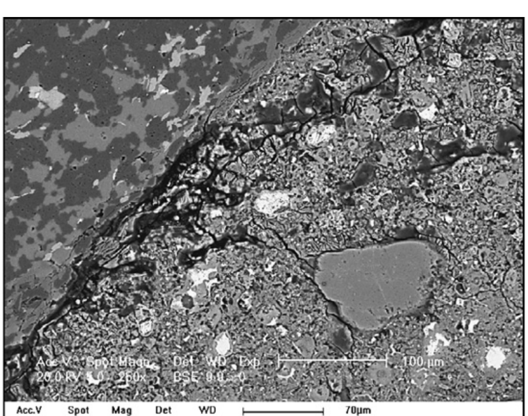

Fig. 9. Scanning electron micrographs of concrete with average longitudinal expansion of 0.81%.

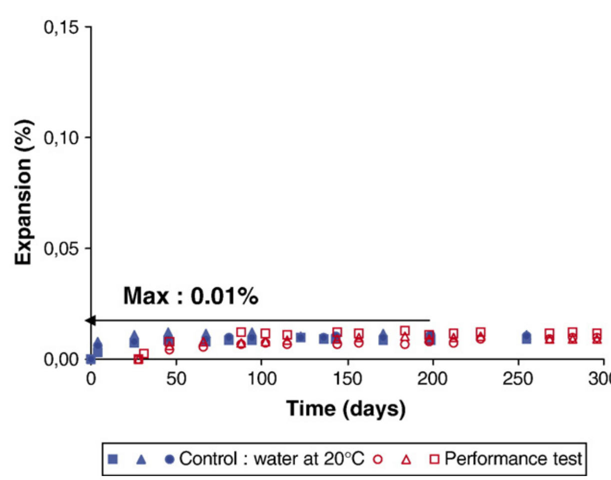

Fig. 11. *Prefab 2.* Expansion of three specimens subjected to the performance test and three control specimens (without cycles) stored in water at 20°C. t_0=end of heat treatment.

test. Essentially, the concrete was characterised by high heating temperature (>70 °C) at early age, which was maintained for more than 3 days, an alkali content in the cement of more than 0.8% and siliceous aggregate. Furthermore, the concrete was subjected to high relative humidity. The DEF process is probably due to the combination of these factors.

We observed that the two concretes that were representative of massive structural members have very different behaviours, although they were subjected to the same heat treatment in early age. It is possible to draw up a list of the parameters with respect to which these concretes differ (Table 4). Essentially, these parameters are: alkali content, the nature of the fine aggregate fraction, and the cement/water ratio. The difference in the reactivity (both kinetics and amplitude) of these two concretes must be linked to these parameters. However, we can only note the differences between these two concretes without being able to reach any conclusion about how these parameters affect expansion.

The results obtained for concrete mixes which have not exhibited in-situ durability problems are very important and confirm the selectivity of this test. The equivalent alkali content of the cement used in the concrete we refer to as "Prefab 2" was 0.89%, and the C_3A and SO_3 contents were respectively 9.9% and 3.7%. This concrete therefore exhibits a combination of parameters which in the literature are judged to be critical for DEF [17]. This concrete's good performance must be due to the limited duration of its exposure to high temperatures. These observations and findings suggest that the length of time the concrete is subjected to heating plays a decisive role.

This test did not cause expansion in all the specimens from Bridge 1. The representativity of the test is judged to be realistic as the concrete cores taken from sound zones did not expand during the test. Furthermore, the core samples taken from slightly-cracked parts of the cross head exhibited considerable potential for expansion in the long term. However, the core sample taken from the highly-cracked part of the cross head displayed no potential for further expansion, probably due to the fact that the maximum degree of expansion had already been obtained. This difference in the deterioration of the concrete in the cross head can probably be explained by different levels of exposure to water [24], and perhaps other factors.

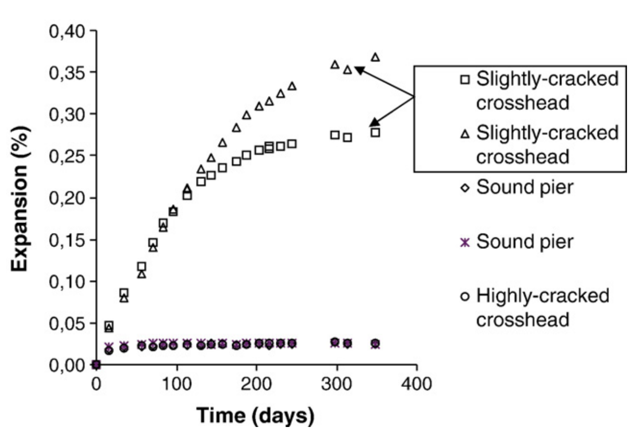

Fig. 12. Longitudinal expansion of 5 core samples taken from a bridge with localized DEF-induced damage.

Table 4
Decisive factors for the two concrete mix designs in Bridge 1 and 2

	"Bridge 1"	"Bridge 2"	"Prefab 1"	"Prefab 2"
Equivalent alkali content	0.85	1.16	0.39	0.89
$\frac{SO_3^2}{Al_2O^3\text{(active)}}$ (threshold 2.0) [17]	3.7	5.4	8.5	3.7
SO_3 in cement	3.15	3.52	2.65	3.70
C_3A in cement	7.2	6.1	2.2	9.9
Type of aggregate	Aggregate: siliceous Sand: sillicalcareous	Siliceous	Sillicalcareous	Sillicalcareous
Water/cement	0.47	0.54	0.45	0.45

5. Conclusions and outlook

As a result of this research a test procedure has been proposed to evaluate the risk of expansion due to DEF for concrete produced under a given heat curing regime. The current status of the test is that of a draft Laboratoires des Ponts et Chaussées test procedure [28]. The results obtained for existing structures confirm the good agreement between the measurements made on the laboratory specimens and observations in the field. Unlike some other tests that are proposed to evaluate the risk of DEF-induced expansion, this test procedure does not lead to expansion in concretes that have not been subjected to a high degree of heating in early age [22]. Validation of the performance test is crucial in order to propose its application. On the basis of these first investigations, the test can be proposed with certain reservations awaiting full validation [28]: more data must be collected on existing cases in order to further improve the test's validity. The Laboratoire Central des Ponts et Chaussées has, therefore, begun an additional validation program to ensure that the test is representative with regard to a larger number of existing cases. It should also be noted that the French working group "Civil and Urban Engineering Network" (GranDuBé) has initiated a program with the participation of some ten laboratories in order to quantify the reproducibility of the test. The test procedure may be modified if necessary as a result of these studies.

Adequate data is still not available to allow an upper value to be established, above which it could be considered that a concrete is certain to be susceptible to DEF. So far, the results provided by this test are only indicative and expert knowledge is required to distinguish "concrete/heating" pairs which are susceptible to DEF from those which are not. Nevertheless, the results show that concrete mixtures which do not expand in the long term expand by between 0.02% and 0.06%. This expansion occurs during the first days of immersion and can be ascribed to water absorption. In order to establish a critical threshold it is not necessary for the maximum expansion to be reached. At the present time, both the duration of the test and a critical threshold for expansion are not fixed. This topic is actually discussed on the basis of complementary studies.

Acknowledgements

The authors would like to thank the Association Technique de l'Industrie des Liants Hydrauliques (Technical Association for Hydraulic Binders) and the Centre d'Etude et de Recherche de l'Industrie du Béton (Manufactured Concrete Industry Study, Design and Research Center) for their financial and technical support.

References

[1] J.P. Skalny, Internal sulfate attack — point of agreement and disagreement, International RILEM TC 186-ISA Workshop on Internal Sulfate Attack and Delayed Ettringite Formation, 4–6 September, Villars, Switzerland, 2002, pp. 265–275.

[2] L. Divet, Comment se prémunir des réactions sulfatiques dans les bétons: point sur les normes actuelles et quelques recommandations. Note technique, Bulletin des LPC-240-Sept.-Oct., Réf. 4447, 2002, pp. 87–94.

[3] German Committee for Reinforced Concrete, Recommendation on the Heat Treatment of Concrete, Deutscher Ausschuss fur Stahlbeton, DIN Deutsches Institut fur Normung e.b. Bindesalle 216/218, D-1000 Berlin, 1989, p. 13, 15, Sept.

[4] C.D. Lawrence, J.A. Dalziel, D.W. Hobbs, Sulfate attack arising from delayed ettringite formation, Interim Techical Note, 12, British Cement Association, Wexham Springs, Slough, may 1990, U.K.

[5] NF EN 1323–1, Application ferroviaire voies traverses et supports en béton, Chap. 6.2 - Procédés de fabrication, 1998.

[6] NF EN 13369, Règles communes pour les produits préfabriqués en béton, 2001 Oct.

[7] SCA A23.4–00, Precast Concrete — Materials and Construction/Code for Architectural and Structural Precast Concrete Products, CSA International, 2000.

[8] H.F.W. Taylor, C. Famy, K.-L. Scrivener, Delayed ettringite formation, Cem. Concr. Res. 31 (2001) 683–693.

[9] R. Barbarulo, Comportement des matériaux cimentaires: actions des sulfates et de la température. Thèse de doctorat en Génie Civil. Ecole Normale Supérieure de Cachan. Faculté des études supérieures de l'Université de Laval. Sept. 2002. 271 pp.

[10] F.P. Glasser, The stability of ettringite, International RILEM TC 186-ISA Workshop on Internal Sulfate Attack and Delayed Ettringite Formation, 4–6 September, Villars, Switzerland, 2002, pp. 43–64.

[11] M. Collepardi, The influence of sulfate content in clinker or cement and curing temperature on DEF-related expansion of concrete, International RILEM TC 186-ISA Workshop on Internal Sulfate Attack and Delayed Ettringite Formation, 4–6 September, Villars, Switzerland, 2002, pp. 212–228.

[12] W. Scherer, Factors affecting cristallisation pressure, International RILEM TC 186-ISA Workshop on Internal Sulfate Attack and Delayed Ettringite Formation, 4–6 September, Villars, Switzerland, 2002, pp. 348–354.

[13] K. Scrivener, A. Guidoum, V. Mathier, Role of cracks in delayed ettringite formation, International RILEM TC 186-ISA Workshop on Internal Sulfate Attack and Delayed Ettringite Formation, 4–6 September, Villars, Switzerland, 2002, pp. 259–262.

[14] D. Damidot, F.P. Glasser, Thermodynamic investigation of the CaO-Al_2O_3-$CaSO_4$-H_2O system at 50 °C and 85 °C, Cem. Concr. Res. 22 (1992) 1179–1191.

[15] L. Divet, Les réactions sulfatiques internes au béton: contribution à l'étude des mécanismes de la formation différée de l'ettringite, Etudes et Recherches des Laboratoires des Ponts et Chaussées, Ouvrages d'Art, n° 40, Paris, 2001, décembre, 227 pp.

[16] M.C. Lewis, K.L. Scrivener, S. Kelham, Heat curing and delayed ettringite formation, Mater. Res. Soc. Symp. Proc. 370 (1995) 67–76.

[17] R.L. Day, The effect of secondary ettringite formation on the durability of concrete: a literature analysis, Research and Development Bulletin RD108T, Portland Cement Association, Skokie, Illinois, U.S.A., 1992, 115 pp.

[18] C.R. Duggan, J.F. Scott, New test for deleterious expansion in concrete, The 8th International Conference on Alkali-Aggregate Reaction, Kyoto, Japan, July 17–20, 1989.

[19] Y. Fu, Delayed Ettringite Formation in Portland Cement Products, PhD thesis in Civil Engineering. 1996, Ottawa, 199 pp.

[20] J. Stark, K. Bollmann, Investigation into delayed ettringite formation in concrete, Proc. of the 9th International Congress on the chemistry of cement, New Delhi, India, vol 5, 1992, pp. 348–354.

[21] N. Petrov, Influence of Microckacking on expansion cause by DEF. PhD Thesis, University of Sherbrooke, 2003, 190 pp.

[22] A. Pavoine, L. Divet, S. Fenouillet, V. Randazzo, J.-P. Davy, Impact de conditions de cycles de séchages et d'humidification sur la formation différée d'ettringite dans les bétons, Mat. Struct./Matér. Constr. 36 (2003) 587–593 (November).

[23] A. Pavoine, L. Divet, S. Fenouillet, A concrete performance test for delayed ettringite formation: part I. Optimisation, Cem. Conc. Res. 36 (2006) 2138–2143.

[24] L. Divet, A. Pavoine, J.-L. Clement, G. Le Mestre, La réaction de gonflement interne des bétons due à la formation différée d'ettringite: Méthode de diagnostic et de pronostic, Ann. Bâtim. Trav. Publics 4 (2003) 14–24.

[25] AFNOR, Test Methods on Reactivity to Alkalies. XP P 18–594, 2004 33 pp.

[26] J.-L. Clement, Calcul de l'histoire thermique des pièces en béton, Une nouvelle pathologie dans les bétons, Ponts Formation Edition, Paris, 2003, jeudi 12 juin, 13 pp.

[27] C. Larive, Apports combinés de l'expérimentation et de la modélisation à la compréhension de l'alcali-réaction et de ses effets mécaniques, Etudes et Recherches des LPC. OA 28, 1998.

[28] A. Pavoine, L. Divet, Réactivité d'une formule de béton vis-à-vis de la réaction sulfatique interne. Essais de performance, Techniques et méthodes des LPC. Projet de méthode d'essai des lpc n°59, 2003 Mai, 17 pp.