



Impact of unrestrained Delayed Ettringite Formation-induced expansion on concrete mechanical properties

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ABSTRACT

The consequences of Delayed Ettringite Formation (DEF) on the mechanical properties of concrete still remain imperfectly known. It is generally recognised that this pathology can decrease the strength of the material and its Young modulus.

The comparative study between the expansion of concrete and the evolution of its dynamic modulus carried out in this study on a great number of samples, demonstrates that two types of swelling behaviours can be observed: a linear and a sigmoidal.

A relationship between modulus and expansion rate is highlighted but it only remains in the case of linear swelling, which does not generate any significant damage.

In the case of sigmoidal swelling, the damage process starts for expansions greater than 0.1% that can consequently reduce by 60% the dynamic modulus and by 65% the compressive strength. The relationship between modulus and expansion rate established for linear swelling cases is temporary verified for sigmoid swelling ones at the inflection point. At this point, the supersaturation is assumed to be mostly consumed and cannot further damage the matrix. Thereafter, the phase of stabilization of the expansion begins, and can be concomitant with a rehealing of the matrix induced by the continuation of cement hydration.

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1. Introduction

Delayed ettringite formation (DEF) is a pathology of cementitious materials that has been widely studied these past years. If ettringite forms only partially during the beginning of cement hydration (its solubility increases with temperature and alkali content [1–4]), the late crystallization of the remaining ettringite in a hardened material stored in wet conditions, without any external sulphate supply, can be associated with a global swelling of the material. DEF can be identified if this swelling generates damage at the level of the material or the structure. This phenomenon can cause concrete specimens to expand by about 2% in unrestrained conditions of expansion, leading to severe damage in field structures as cracks affecting the whole concrete.

The swelling of concrete specimens affected by DEF is usually determined by the longitudinal expansion, gain of weight, which consists of gain of water, and reduction of mechanical characteristics such as ultimate tensile and compressive strength and Young's modulus.

DEF-induced expansions become significant after an induction period varying from several days to few months. The swelling curve,

whose shape is sigmoidal, reaches a plateau within 1 to 2 years [4–6]. Final expansion of concrete specimens can reach 2% [7].

Zhang et al. [9] established a correlation between the reduction of the dynamic modulus measured by sonic resonance and the expansion. This reduction may exceed 10% and even 40% for damaged specimens whose expansion reaches 1.7%. Pavoiné [7] observed a fall of 75% of the ultimate compressive strength for concrete specimens that have expanded by 1.6%.

In the case of ASR-induced expansions, the main mechanical characteristic that is affected is the Young's modulus (static elastic modulus) because the ultimate compressive stress is only slightly affected [10]. DEF-induced expansions seem at the same time more homogeneous and of greater amplitude than ASR-induced ones (up to 2% for DEF cases [7] against 0.5% for usual ASR cases [10] of concrete specimens).

This paper uses a complete set of designed experiments to provide an interpretation of the simultaneous evolution of swelling and mechanical characteristics of concrete specimens affected by DEF. Details concerning the structure and statistical analysis of the design of the experiments and the impact of each parameter on DEF-induced expansions can be found in another paper [11]. These experiments allowed to organize into a hierarchy the parameters and interactions between parameters that affect DEF-related expansions. Among all effects generated by the set of the studied parameters, the most relevant ones were (in decreasing

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Table 1
Chemical and mineralogical composition of the cement

CEM I 52.5 cement	[%]
<i>Chemical composition</i>	
SiO ₂	19.0
Al ₂ O ₃	4.1
Fe ₂ O ₃	3.75
CaO	64.6
MgO	1.09
Na ₂ O	0.13
K ₂ O	0.56
Equivalent Na ₂ O	0.50
Total SO ₃	3.36
Lost on ignition	1.48
Insoluble	1.14
Free lime	0.67
CO ₂	0.96
<i>Mineralogical composition</i>	
C ₃ S bogue	69.0
C ₂ S bogue	2.35
C ₃ A bogue	4.45
C ₄ AF bogue	11.4

order) coupling between temperature and duration, single effects of duration, temperature and aggregate type and coupling between duration and aggregate nature.

2. Design of experiments

In the first set of experiments, called “heating”, parameters related to heating and alkali content of the concrete were chosen. The sample-name convention is [temperature °C]-[Alkali content %]-[heating duration]. For example, 85-1.00-2D means a heating at 85 °C lasting 2 days of a concrete that contains 1.00% alkali. Recommendations often deal with threshold temperatures ranging from 65 °C to 85 °C. As a consequence, both temperatures have been kept in the “heating” experiments. Two hours and 6 h reflect common duration of heat treatment in the case of a precast concrete. Two and 10 days have been chosen to simulate the duration of heating in massive concrete members.

To change the value of the alkali content, KOH was added to mixing water. The minimal value of the alkali content is set by the CEM I 52.5 cement chosen for the study that contains itself 0.50% equivalent alkali (Footnote: Table 1). Pavoine previously revealed that a concrete made with this cement can generate DEF-related expansions in some particular conditions of curing and subsequent storage [7]. To reach higher values that are expected to promote DEF, two additions were studied: 0.25% and 0.50% equivalent alkali leading to a total alkali content of 0.75% and 1% respectively. Siliceous aggregates and a W/C ratio of 0.48 were used.

Table 2
Mix designs of the concrete, unit: Kg/m³

Siliceous aggregates			Calcareous aggregates		
	W/ C=0.48	W/ C=0.35		W/ C=0.48	W/ C=0.35
<i>Particle size [mm]</i>					
0/0.315	183	192	Dry sand (S)	711	769
0.315/1	134	141			
1/4	217	228			
2/4	232	244	Dry gravels (G)	1067	1154
4/8	180	189			
8/12.5	842	885			
Water reducer: 0.6%	/	2.52	Water reducer: 0.6%	/	2.63
KOH to 0.75%	1.52	1.60	KOH to 0.75%	1.54	1.66
KOH to 1.00%	3.04	3.19	/	/	/
Water (W)	192	146	Water (W)	199	159
Cement (C)	400	420	Cement (C)	405	438

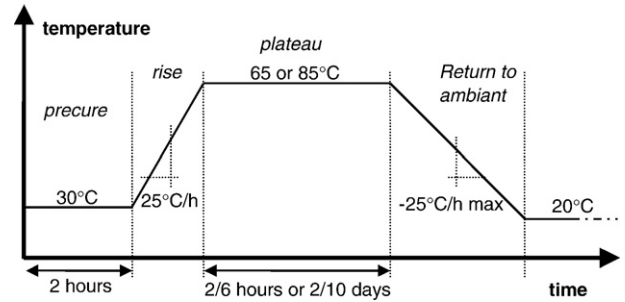


Fig. 1. Heat treatment phases.

In the second set of experiments, called “concrete-mix design”, parameters suspected to mostly affect the kinetics of DEF have been tested. The sample-name convention is [W/C]-[aggregate type]-[heating duration]. For example, 0.35-Si-10D means a heating lasting 10 days of a concrete that contains siliceous aggregate with a W/C=0.35. An additional value of W/C (equal to 0.35) was used to account for high performance concretes. The concrete-mix design has been optimized with respect to slump for the mix design having a W/C ratio of 0.48. The concrete-mix design with W/C=0.35 is obtained from the previous concrete by reducing the amount of mixing water. To improve the resulting slump, an amount of high-range water reducer is equal to 0.6% of the cement weight was added. Two kinds of French aggregates were used: siliceous aggregates from Palvadeau's quarry and calcareous aggregates from Boulonnais' quarry. Both Palvadeau's and Boulonnais' aggregates are classified as none reactive with respect to alkali-silica reaction by the XP P18-594 test [19]. Moreover severe conditions were chosen for temperature and alkali content (85 °C and 0.75% of equivalent alkali).

Table 2 summarizes the mix designs of these different concretes. Samples were made in a mixer of 100 L. The mixing and placing protocols matched French normative standards: NF P 18-400 [20], NF P 18-421 [21] or NF P 18-422 [22], NF P 18-404 [23], NF P18-404 [24]. Each concrete sample was cast and cured in 3 cylindrical moulds whose dimensions are 11 cm (diameter) and 22 cm (height) to generate 3 identical concrete specimens. The moulds were covered so as to limit water exchange. The applied heat treatment followed a cycle divided into four different steps (Fig. 1). Some concrete samples were not heat treated but were conventionally cured at 20 °C to be used as reference.

After the heat treatment, samples were stored at 20 °C in 100% relative humidity until 28 days. Next, wetting and drying cycles were applied in accordance with LPC no. 59 pre-test method [12]. The objective of these cycles is to increase the kinetics of DEF reaction without changing its triggering conditions [7]. Samples are subjected to 2 cycles, each one lasting 14 days. A cycle consists of 7 days of drying

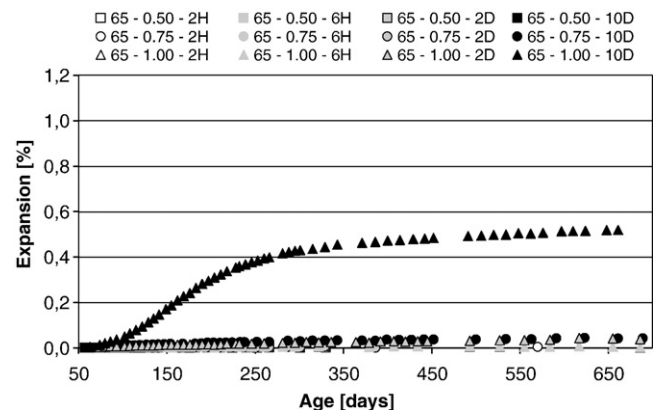


Fig. 2. Swelling of concrete specimens treated at 65 °C, “heating” design of experiments.

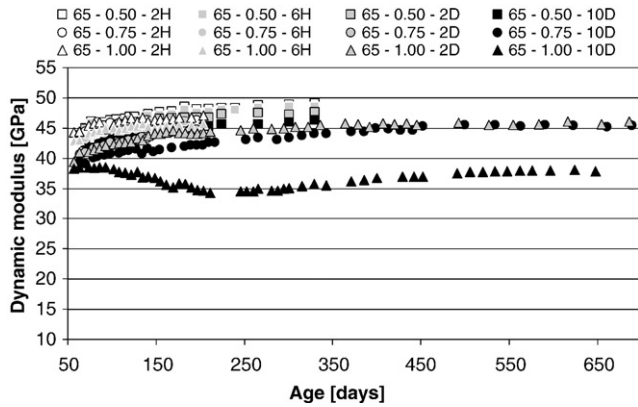


Fig. 3. Evolution of the dynamic modulus of concrete specimens treated at 65 °C, "heating" design of experiments.

at 38 °C and 30% relative humidity followed by 7 days of immersion in tap water at 20 °C. The volume of water with respect to the volume of the sample is kept under 1.5 to limit leaching effects. Once the cycles are finished, samples are stored into 3 individual hermetic boxes (to avoid carbonation) whose dimensions barely exceed those of the sample, to minimize the amount of water required for their immersion. During this last period, expansion measurements were performed with an extensometer whose resulting measurement dispersion on the basis of three concrete specimens is less than 0.002%.

Additionally, resonance frequency measurements of each concrete specimen were performed to determine the dynamic modulus of the material. This technique is a non destructive method that gives access to mechanical properties. The principle of sonic resonance is to measure the speed of sound propagation through a sample of a given material. This speed depends on the dynamic elastic modulus of this material which is linked to Young's modulus. Hence, measuring an evolution of this speed reveals a modification of the mechanical properties of the material. The resonance frequency is obtained by the measurement of the first longitudinal oscillation frequency of a concrete specimen which has been subjected to an impact (Dirac impulsion) at the centre of one of its bases. This self-oscillation frequency is linked to the dynamic modulus of the material by the following equation:

$$f_{\text{longi}} = \frac{1}{2L} \sqrt{\frac{E_{\text{dyn}}}{\rho}}$$

f_{longi} longitudinal self-oscillation frequency [s^{-1}]
 L height of the specimen [m]
 E_{dyn} Dynamic longitudinal elastic modulus [Pa]
 ρ density of specimen [$\text{kg}\cdot\text{m}^{-3}$]

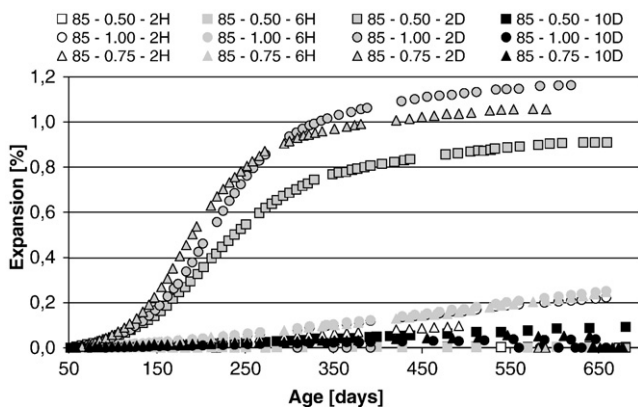


Fig. 4. Swelling of concrete specimens treated at 85 °C, "heating" design of experiments.

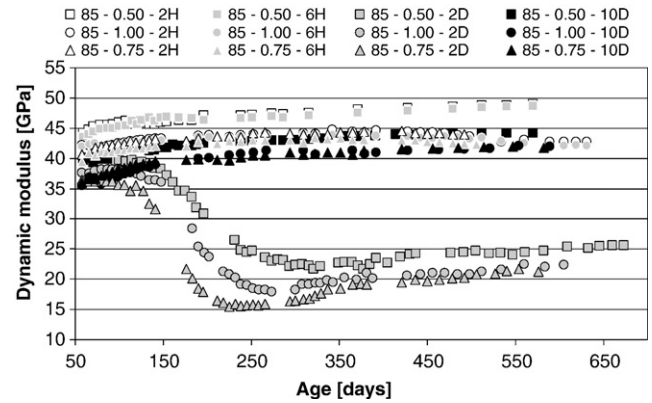


Fig. 5. Evolution of the dynamic modulus of concrete specimens treated at 85 °C, "heating" design of experiments.

Initially, the density was determined using hydrostatic weighing. The weight is measured along with time. The volume change is assessed using the expansion measurement, assuming that this expansion is isotropic. The effect of the density value on the resulting dynamic modulus is not significant unless expansion exceeds 1% (i.e. volumetric expansion exceeds 3%).

3. Results

Swelling and dynamic modulus evolution with time are presented in Figs. 2–5 for the "heating" design of experiments at 65 and 85 °C respectively. Similar curves for the "concrete-mix design" design of experiments are given in Figs. 6–9.

Initial dynamic moduli vary from 35 to 50 GPa, mostly depending on the heating duration (Figs. 3 and 5) and the W/C ratio (Figs. 7 and 9): the shorter the heating duration and the lower the W/C ratio, the higher the initial dynamic modulus.

When no significant expansion is measured (Fig. 8), the evolution of the dynamic modulus consists of a more or less progressive improvement depending on the concrete (Fig. 9). This is certainly due to the improvement of the mechanical properties associated with the normal hydration process of residual anhydrous cement grains, whose amplitude depends on the amount of residual anhydrous cement grains.

When expansion becomes very significant, which can be defined as an expansion exceeding 0.2% at 700 days [11], the evolution of the dynamic modulus is strictly different (Figs. 3, 5, 7). For such concrete specimens, there is a specific time from which the value of the dynamic modulus starts falling. From a mechanical point of view, a decrease in elastic modulus means damage.

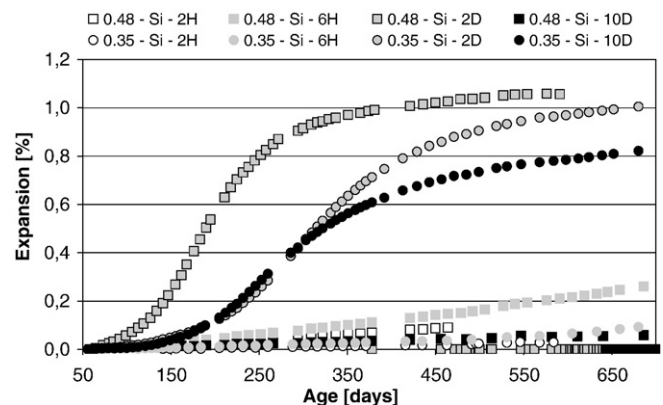


Fig. 6. Swelling of concrete specimens with siliceous aggregates, "concrete-mix design" design of experiments.

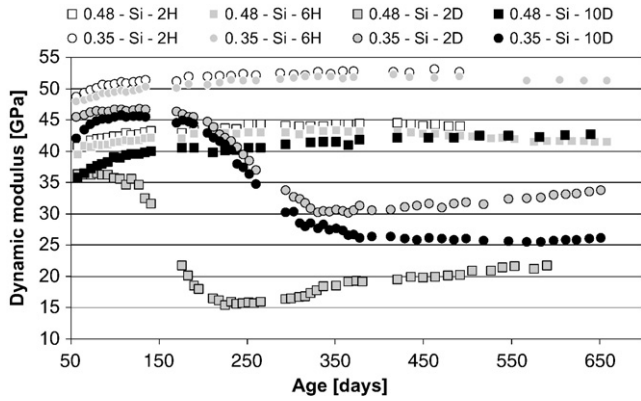


Fig. 7. Evolution of the dynamic modulus of concrete specimens with siliceous aggregates, “concrete-mix design” design of experiments.

Loss in dynamic modulus always starts at an expansion by about 0.1%. More precisely, it depends on the slope of the swelling curve at the 0.1% value:

- If the curvature corresponds to a clear acceleration, the loss in dynamic modulus can reach about 60% (specimens 0.48-Si-2D, 0.35-Si-2D and 0.35-Si-10D in Figs. 6 and 7);
- If the curve stays almost linear, the loss in dynamic modulus may not be significant (specimens 0.48-Ca-2D in Figs. 8 and 9).

Moreover, an expansion of 0.1% corresponds to the first appearance of visible cracks at the surface of concrete specimens. SEM observations support that macroscopic observation: an expansion of 0.1% corresponds to the first visible separation between paste and aggregate as commonly reported [12].

When expansion stabilises, a progressive improvement in dynamic modulus is usually observed. This could be explained by the effect of a progressive filling of previously-opened cracks by ettringite massive deposits, as observed by SEM [13].

Dynamic modulus measurements performed in this study are in agreement with Zhang et al. [9] about the fact that the amplitude and the rate of the expansion can induce significant changes of dynamic modulus.

Destructive mechanical tests have been done to verify sonic resonance's trends. These compressive tests have only been applied to a selection of interesting cases: significantly damaged specimens (once stabilised) and some unaffected ones (Fig. 10).

In the case of 0.48-Si concretes (“heating” experiments), a relationship can be obtained between the ultimate compressive strength (residual strength) and the final expansion achieved. Typically, the strength of unaffected 0.48-Si concrete specimens reaches about 40 MPa. The most damaged specimens of 0.48-Si concretes fall down to 15 MPa, which represents a very significant

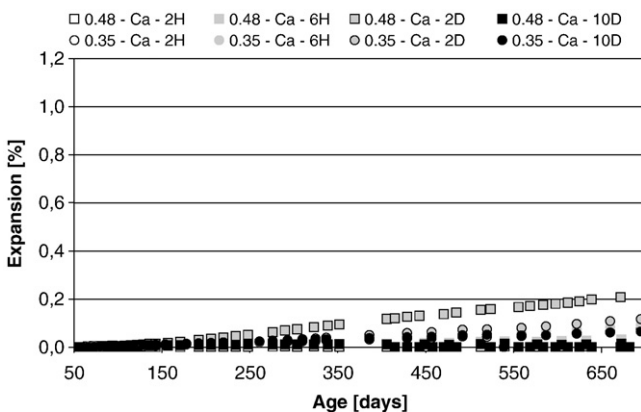


Fig. 8. Swelling of concrete specimens with siliceous aggregates, “concrete-mix design” design of experiments.

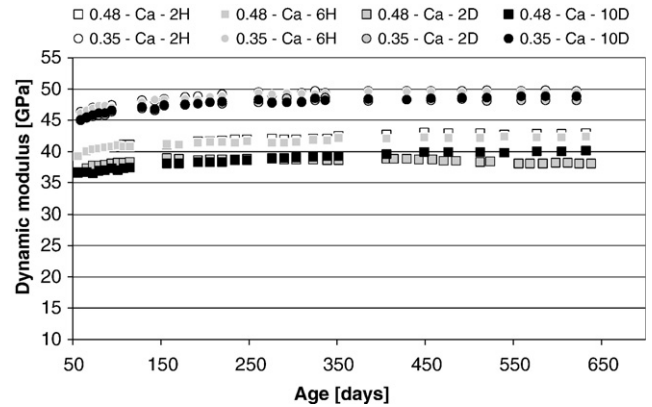


Fig. 9. Evolution of the dynamic modulus of concrete specimens with calcareous aggregates, “concrete-mix design” design of experiments.

decrease. Pavoiné [7] observed a fall of strength by 75% for concrete specimens that have expanded by 1.6%. In Fig. 10, the linear approximation would forecast a fall of strength on the order of 90% for a 1.6% expansion. This value, certainly exaggerated by the linear approximation, serves to verify that high expansion (over 1%) leads to very low residual mechanical strength. The linear approximation proposed in Fig. 10 seems to imply that a DEF-related expansion lower than 0.20% will not generate significant loss in compressive strength. For higher expansions the mechanical properties are affected. Fig. 11 shows the typical behaviour of a slightly damaged (~17% of strength) concrete specimen whose expansion reaches 0.54%; the Young's modulus (static elastic modulus) achieves 28 GPa. Fig. 12 represents the typical behaviour of a very damaged (~65% of strength) concrete specimen whose expansion achieves 1.06%; it is no longer possible to determine the Young's modulus. The strain corresponding to the maximum stress increases from $3.2 \cdot 10^{-6}$ for the slightly damaged specimen to $5.9 \cdot 10^{-6}$ for the more damaged one.

The last cycle of loading in Fig. 11 presents a concrete sample which has been mechanically damaged. This resulting concrete obviously presents a much lower Young's modulus; the maximum strain also decreases. Most damage models are based on this hypothesis of compressive test damage process [8] and produce concrete whose incremental plastic strains decrease with cycles.

The behaviour of the much DEF-affected specimen cannot be obtained from the behaviour of the less affected one using a damage model which is based on compressive test damage process. This means that the damage process of DEF does not produce cracks that can be compared with those produced by compressive test. DEF-related cracks, instead of decreasing the plastic strain of concrete,

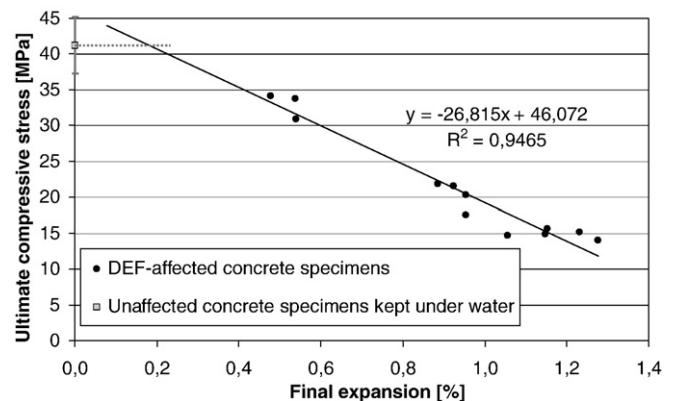


Fig. 10. Compressive stress versus final expansion for the concrete specimens of the “heating” design of experiments.

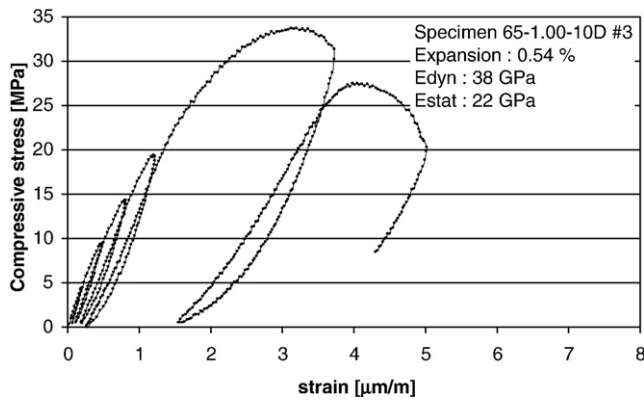


Fig. 11. Stress/strain relationship of the 65-1.00-10D #3 specimen, once stabilised.

increase it. As a consequence, the ductility of concrete increases with DEF-related expansions.

4. Discussions

Because Young's modulus is affected by porosity and cracks, the dynamic modulus can be plotted against the expansion to test for a correlation. Nevertheless these results are not in agreement with the conclusion of Zhang et al. [9] because no clear and direct relationship links dynamic modulus and expansion. A plot against fractional reduction in modulus is not better. Damage is neither proportional to expansion amplitude nor expansion rate. In fact, a parameter related to magnitude and rate is relatively clearly linked to dynamic modulus: this is the mean rate of expansion determined at the inflexion point of the swelling curve as demonstrated by Fig. 13.

Note that for concrete specimens that present a linearly swelling, every point of the swelling curve can be considered as inflexion point, because expansion rate and dynamic modulus are almost constant.

Fig. 13 was obtained by plotting the expansion divided by the time at the inflexion point as a function of the dynamic modulus measured at the same point for every sigmoidal swelling case: a very good relationship is thus observed. Another relationship can also be found for samples exhibiting linear expansion as the rate of expansion and the dynamic modulus are almost constant over time. It is very interesting to observe that linear behaviours extend the sigmoidal ones. Thus both relationships are totally compatible as long as the aggregate type is not changed.

The relationship proposed in Fig. 13 implies that during a linear expansion, there is no direct damage to the material, and that the expansion rate is only a function of the dynamic modulus of the material. As a consequence, linear swelling means that the material

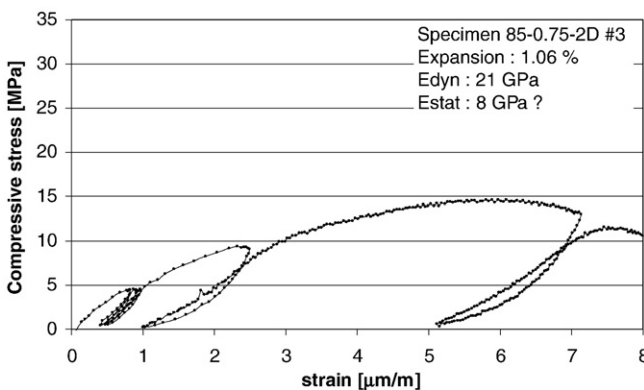


Fig. 12. Stress/strain relationship of the 85-0.75-2D #3 specimen, once stabilised.

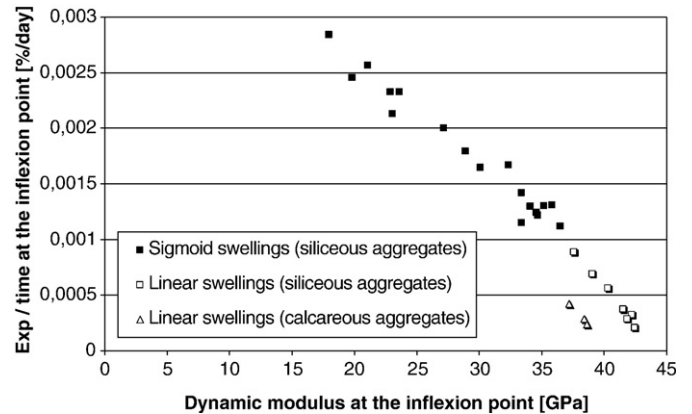


Fig. 13. Expansion/time ratio versus dynamic modulus at the inflexion point for each individual sigmoid-swelling and linear-swelling concrete specimens.

can stand the expansion without any damage (i.e. the supersaturation is insufficient to damage the matrix); the expansion rate is limited by the stiffness of the matrix. The strain remains within the linear elastic limit until the expansion reaches 0.1%.

In the case of sigmoidal swelling, the mean rate of expansion rapidly exceeds the value that would be expected by plotting the initial modulus in Fig. 13. As soon as the expansion exceeds 0.1%, the material cannot stand the expansion and gets damaged. The mean rate of expansion increases whereas the dynamic modulus decreases until both of them match the relationship presented in Fig. 13. The damage process is assumed to stop when most of the supersaturation is consumed and cannot longer open new cracks. At this point (the inflexion point), the material temporary behaves like linear-expansion-affected concrete specimens. Then, the expansion slows down while the dynamic modulus stops falling; this is probably due to an increment of the opening of existing cracks with no new cracks generated.

Since the elastic limit is exceeded for 0.1% expansion, most of the behaviour of the sigmoidal-expansion-affected specimens cannot be described using an elastic model. The existence of a single relationship that links the expansion behaviour of damaged specimens and undamaged ones (Fig. 13) implies that there is a clear link between the stiffness of the matrix and the mean rate of expansion, and not the expansion amplitude.

Unfortunately, the difference between the relationship obtained with siliceous-aggregate concretes and with calcareous-aggregate concretes means that the relationship is concrete-mix-design dependent.

Calcareous-aggregate concretes behave with respect to DEF like siliceous-aggregate concretes whose dynamic modulus would be 4 GPa higher.

SEM observations do not indicate any difference in the cement paste between these two cases nor any evidence of ASR gel for siliceous-aggregate concretes. However, the paste/calcareous-aggregate interface transition zone shows a better continuity between the paste and the aggregate, in particular because of the higher roughness of the calcareous aggregates in comparison with siliceous aggregates, which are much smoother [13]. The difference in porosity between the interfacial transition zone (ITZ) of a calcareous aggregate and the cement paste is less pronounced than in the case of siliceous aggregates. As ITZ contributes to the transport of matter in the concrete [14] and also to the mechanical strength, a better ITZ as found for calcareous aggregates could explain the difference in behaviour: the expansion rate could be limited by the transport properties and/or the enhanced mechanical bond.

Petrov et al. [15], Collepardi et al. [16], Fu et al. [17] established that microcracking of the matrix can increase the expansion rate. Microcracking necessarily involves a decrease in dynamic modulus.

In accordance with Fig. 13, at the inflexion point for sigmoidal behaviour or at any point for linear behaviour, a decrease in dynamic modulus would correspond to a higher expansion rate. This could be a mechanical explanation of the impact of initial microcracking on DEF-related expansion.

5. Conclusion

Three types of behaviours can be defined for concrete subjected to heat treatment and thus that can potentially suffer from DEF:

- Negligible swelling, i.e. expansion never exceeds 0.04%. No macroscopic indicator makes it possible to diagnose any activity related to a process of internal swelling. The dynamic modulus of these concretes slightly improves with time and reaches a plateau within 1 to 2 years.
- Weak swelling, which results in linear expansion whose final amplitude lies between 0.04% and 0.2%. The expansion rate is limited by the stiffness of the material according to a linear relationship between the rate of expansion and the dynamic modulus. This behaviour does not induce any notable modification of the mechanical properties of the concrete, at least until an expansion by 0.1%.
- Large swelling, which results in sigmoid expansion whose final amplitude generally exceeds 0.4%. This behaviour causes a fall of the dynamic modulus of the material, which begins towards an expansion by 0.1%, immediately followed by an acceleration of the expansion. According to Fig. 13, at the inflexion point, the material is found temporarily in a state where the mean expansion rate and the dynamic modulus are controlled by the same law as weak swellings. The fall of compressive strength of the concrete can reach 63% for an expansion of 1.2%. When the dynamic modulus stops falling, the expansion progressively reaches a plateau; swelling is at the stage of stabilization. Thereafter, the dynamic modulus improves gradually, probably due to the progressive filling of previously-opened cracks by the ettringite during the phase of stabilization of the swelling. For some cases (ex: specimen 65-1.00-10D of the “heating” experiment), this reheating even makes it possible to recover the value of the initial dynamic modulus.

The value of a 0.2% final expansion is a good indicator to distinguish weak and linear swellings from potentially sigmoidal ones. At this stage of knowledge, this value of final expansion cannot constitute a tolerance level with respect to DEF. On one hand, weak swellings can cause disorders at the scale of the structure if resulting displacement exceeds structural working tolerance (ex: blocking of the valves of a dam [18]), even if the swelling does not generate damage at the scale of the material. On the other hand, no data are currently available to confirm that weak swellings observed on unrestrained concrete specimens can generate significant expansion in a real structure (i.e. under partially restrained conditions).

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