

Internal swelling reactions in massive structures: multi-scale experimental approach

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Abstract

Many laboratory studies of internal swelling reactions (Delayed Ettringite Formation (DEF), Alkali-Silica Reaction (ASR) or the concomitance of both) are realized on relatively small size samples. Those laboratory samples do not allow to reproduce the real conditions to which is submitted the real concrete in its environment, in terms of kinetics, volume/surface ratio, thermal conditions especially in massive structures. Therefore, a representative massive concrete structure (2.4 x 1.4 x 1 m³) is realized under controlled and optimized conditions for the development of the concomitant ASR-DEF case to allow better observation and understanding of swelling reactions at structure scale. The scale effect on swelling kinetics is demonstrated by an experimental study comparing the evolution of swelling reaction in the massive samples with the evolution in reconstituted concrete laboratory specimens as well as coring specimens. The measurements are to finely describe and help in understanding the evolutionary mechanisms of structural degradation and can be used subsequently as a reference for numerical simulations. Results show different swelling kinetics in function of sample type, conservation environment, size and boundary conditions.

Keywords: alkali-silica reaction; delayed ettringite formation; massive structure; multi-scale approach; swelling

1. INTRODUCTION

Alkali-silica reaction (ASR) is a chemical reaction between amorphous silica mineral from the aggregates and the alkali hydroxides (Na, K – OH) dissolved in the concrete pore solution. It is one of the main processes affecting the durability of concrete structures around the world [1]. This reaction generates a secondary alkali-silica gel which expands and induces overpressure within the reacting aggregate material and the adjacent cement paste upon moisture uptake from its surrounding environment, thus causing micro-cracking, loss of material's integrity (mechanical/durability) and, in some cases, functionality in the affected structure. Delayed Ettringite Formation (DEF) is an internal swelling reaction that can affect concrete that endured a temperature higher than about 65°C at its early-age [2]. At this high temperature, the ettringite turns unstable and dissolves while the concrete is still plastic and precipitates again after cooling in the hardened material and in moist conditions, thus generating swelling due to crystallisation pressure. The early-age temperature elevation may be due to curing conditions, like in the case of precast elements, or may be due to the exothermic cement hydration process, like in the case of massive structures. Similarly to ASR, the development of this pathology leads to concrete swelling, cracking of the structure, decrease of the mechanical properties of the affected materials and thus potential problems in terms of serviceability of the affected structures [3]. Matter of fact, alkali-silica gels have often been observed in the vicinity of delayed ettringite which often leads to confusion about the causes of damage within the material since both pathologies result in the formation of expansive products.

Swelling pathologies act at different scales: microscopic, mesoscopic and macroscopic. Thus, multi-scale approaches are necessary in the development and enhancement of prediction tools in this area. Hence, the objective of this study is to conduct a multi-scale experimental campaign in order to fill the

gap between conventional laboratory samples and the behaviour of real scale massive structures. In this article, the mesoscopic and the macroscopic scales will be addressed.

The experimental laboratory investigations about the scale effect on internal swelling reactions aims to quantify the expansion magnitudes and kinetics in different specimen types and sizes. Three sample geometries are considered: first, the massive structure mock-up whose dimensions are 240 cm (length), 140 cm (height) and 100 cm (thickness). The second and third specimens geometry studied are concrete cylinders cast in the laboratory and subjected to conservation conditions equivalent to those of the mock-up. The concrete cylinders dimensions are 16 cm (diameter)/32 cm (height), and 11 cm (diameter)/22 cm (height) respectively. In addition to that, three corings were extracted from the massive structure and subjected to conventional test procedures in order to determine the structure's residual swelling. The cores dimensions are equally 11 cm (diameter)/22 cm (height). This article, presents the results of expansion for the different concrete samples. The swelling behaviour of the massive structure and the impact of boundary conditions are discussed. Then the swelling characteristics (magnitude and kinetics) of the different concrete specimens are quantified using a mathematical relation. The impact of conservation conditions is thus demonstrated. Finally, the impact of specimen's size on the swelling magnitude and kinetic parameters is discussed.

2. MATERIALS

The concrete mix used in this study, given in Table 2.1, is selected so that its chemical composition is favourable to the development of ASR and DEF. Concrete is made with calcareous non-reactive sand (0/5), reactive siliceous coarse aggregates (5/12.5 – 12.5/20), and a W/C=0.57. This combination was chosen based on the works of Guedon-Dubied [4], hence the same quarry provides the reactive aggregates. These aggregates are rich in carbonals which, from a mineralogical point of view, is a microcrystalline silica deemed to have a significant alkali reactivity [4]. Furthermore, Monnin showed that the kinetics of alkali consumption increases when the aggregate diameter decreases [5]. This causes a rapid drop in the alkali concentration of the interstitial solution which may limit the final swelling of the concrete. Therefore, by opting for non-reactive sand, the final swelling due to the alkali-granulate reaction is maximized. The binder used is a Portland cement CEM II/A-LL 42.5 R. It contains 1.06% of Na₂eq (by mass), hence providing a total of 4.24 Kg/m³ of alkalis in the mix. As a result, this concrete mix is highly ASR-prone. Moreover, the cement used has a composition satisfying the conditions for developing DEF (aluminates to sulfate ratio equals to 0.7, equivalent sodium content equals to 1,06% of the cement mass). Hence, with the appropriate early-age thermal profile, this concrete mix is DEF-prone as well.

Table 2.1: Concrete mix proportions

	Weight (Kg/m ³)
Cement CEM II A-L	400
Sand 0/5	772
Coarse aggregates 5/12.5	316
Coarse aggregates 12.5/20	784
Plasticizer	1.4

3. MULTI-SCALE EXPERIMENTAL STUDY

The following presents the experimental study conducted on the structural scale as well as the specimen scale.

3.1 Structural scale

The mock-up built is 2.4 meters long, 1.4 meters high and 1 meter thick. The dimensions choice is due to logistical constraints in order to allow the handling and manufacture of this massive sample. However, the thickness of one meter is the desired dimension, representative of a massive structure, and considered to be scale 1 for this study. It's a none-reinforced concrete element. Nevertheless, the mock-up is cast on a metal support designed to allow for the handling of the structure in a manner that

the tensile stress in the structure doesn't exceed 1 MPa while lifting. It consists of a metal plate welded to I-section steel beams and concrete is simply poured on top of it (bottom of the formwork). After the concrete hardens, the support becomes part of the mock-up.

The monitoring methods used are intended to track the behaviour of the structure from its early age and until the swellings' stabilization. Three main parameters are retained: the thermal history and the temperature's distribution in the mock-up's volume (especially at the early age), the internal swelling and the external cracking of the structure. As a result, a suitable instrumentation system has been designed.

A cross section of the mock-up was instrumented with 16 Type J thermocouples. This makes it possible to follow the evolution of the temperature during the hydration of the cement and the thermal gradient in the concrete in both directions. The monitoring of swelling in the structure is ensured by 9 vibrating wire extensometers embedded within the concrete at three levels (Figure 3.1b): 0.25 H, 0.5 H and 0.75 H, where H is the mock-up height (140 cm). At each level, three strain sensors are oriented in the three directions (X, Y, Z) respectively making it possible to continuously follow the deformations over time with accuracy of the order of 2 $\mu\text{m}/\text{m}$. Macroscopic monitoring of the structure is conducted according to the LPC n°47 test method which is a proven industrial methods usually used to characterize in a simple and rapid manner the state of degradation of a concrete structure suffering from swelling pathology and provides a cracking index [6-7]. The cracking index represents an average crack opening (in mm) per square meter of concrete. Figure 3.1a features the axis/area used to monitor the cracking index of the ASR-DEF mock-up and the mock-up's state prior to degradation.

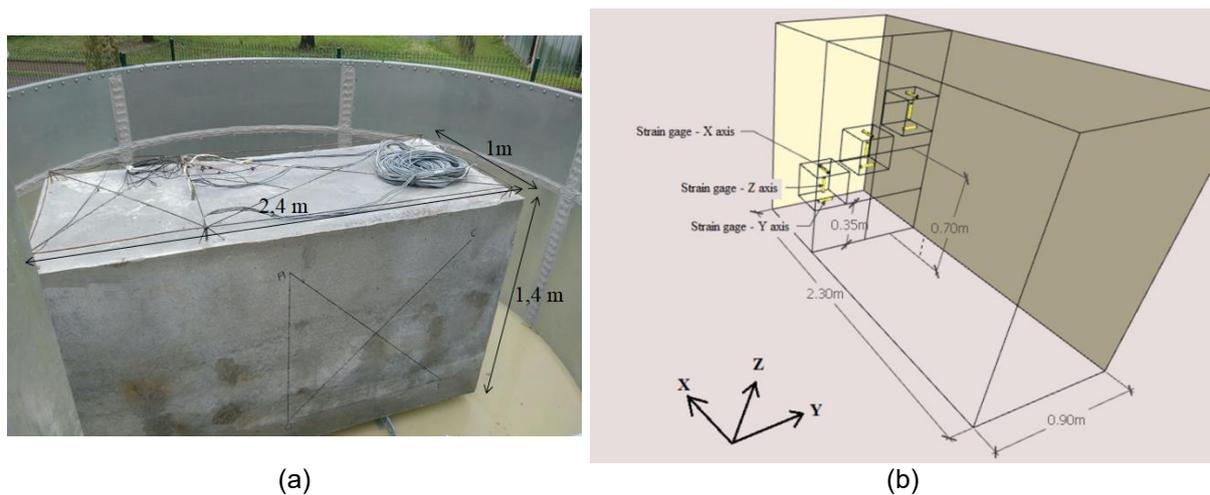


Figure 3.1: (a) Photo of the structure's mock-up (b) Strain sensors placement in the structure (dimensions not including concrete cover)

The early-age heat treatment chosen for the mock-up consists of maintaining the temperature at 80°C during 100 hours and to ensure a homogeneous temperature profile within the structure's volume. Special measures are taken in order to limit the thermal gradients between the mock-up's core and extremities since this massive structure is not reinforced. Hence, a criterion limiting the thermal gradient to 10°C has been defined based on the mechanical performance of concrete at 1 day. Matter of fact, thermal gradients can generate significant mechanical stresses and a risk of cracking at early-age when the tensile strain, arising from or a temperature differential within the concrete section, exceeds the tensile strain capacity of the concrete. The concrete ingredients were heated prior to mixing and multiple blank tests were realized in order to target the required initial temperatures once the concrete is placed in the formworks. An initial concrete temperature of 35 °C is found optimal in order to reach the 80°C objective at the mock-up's core solely due to the exothermic hydration reaction of the cement. The heat treatment of the mock-up was later assured by adding a heating skin containing thermal resistances and glued to the surface of the metal formwork (figure 3.2). Once the concrete has reached 80°C at the core of the mock-up, the heating skin is activated. Its role is only to compensate for heat losses in order to maintain the whole mock-up at 80 °C for 100 hours and to cool it gradually thereafter.



Figure 3.2: The mock-up's early-age temperature regulation system

Figure 3.3 features the early-age heat profile of the structure.

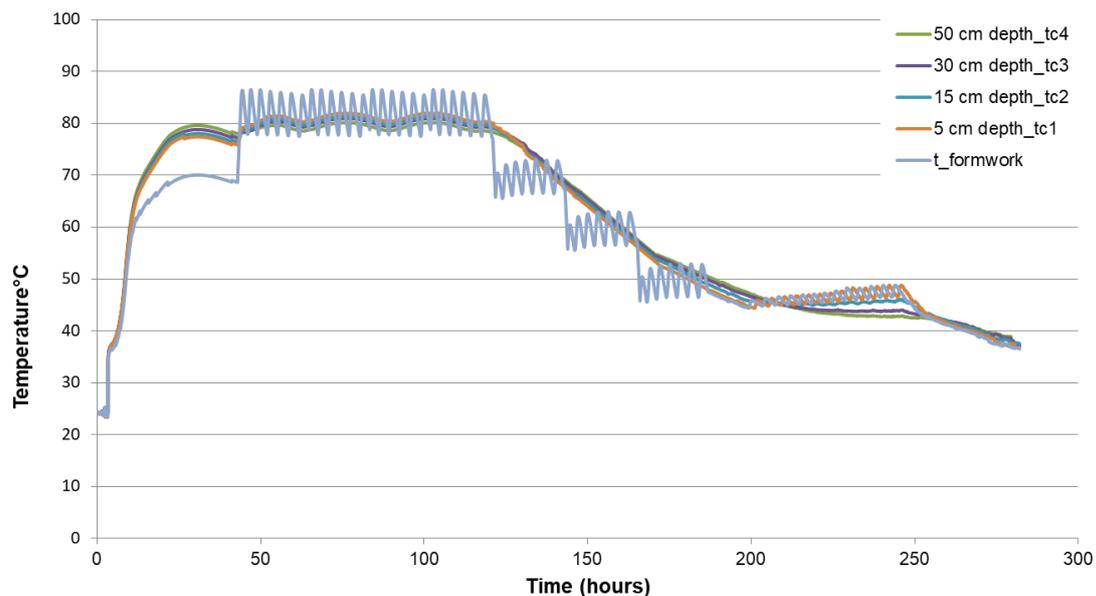


Figure 3.3: The early-age heat profile of the mock-up

Following the early-age heat treatment, the mock-up is conserved fully submerged in a pool constantly maintained at 38°C. The pool is emptied monthly in order to perform a follow up of the structure's surface and to increase alkali-leaching hence accelerating the delayed ettringite formation. Tap water is used to fill the pools.

The experimental program at the structural scale consists of:

- Monitoring the deformations measured by the vibrating wire extensometers placed at various heights inside the mock-ups;
- Maintaining the mock-ups' conservation environment like described above;
- Tracking the mock-ups' cracking index and mapping the surface cracking and various dates;
- Quantifying the alkalis leached during the immersion cycles. Three water samples are taken from the pool at different heights during emptying and one at each filling. These samples undergo Inductively Coupled Plasma (ICP) mass spectrometry analyzes which measures the concentrations of Sodium and Potassium in water to quantify the leaching of alkalis.

3.2 Specimen scale

Six cylindrical concrete specimens (three Ø11H22 and three Ø16H32) were prepared according to the standards NF P 18-400 and NF P 18-422. After casting, the concrete specimens were heat treated. The heat treatment applied is identical to the temperature profile measured in the mock-up's core at the early age (figure 3.3). It was performed in a climatic chamber regulated at 100% relative humidity. Afterwards, the specimens are stored in 20°C water until 28 days age, then they are placed at 38°C in a volume of water respecting the same ratio (Water Volume)/(Exposed Surface) as the structure in the pool and respecting the same renewal cycles as well. Therefore, the specimens have an identical heat profile and an equivalent conservation environment as the corresponding structure.

In addition to that, three Ø11H22 core samples were extracted from the structure at 0.25 H along the Y axis. One of the extracted cores is subjected to the LPC N°44 test method, in which the sample is conserved at 38°C and 100% relative humidity [8]. The remaining two are subjected to the LPC n°67 test method in which the samples are stored at 20°C and immersed in water [9].

A variety of experimental measurements are conducted periodically on concrete specimens including length and mass variation. The expansion is measured using an extensometer. For each sample, the length variation is measured along three longitudinal lines located at 120° from each other in the circumference. The length base used for strain measures is 10 cm for Ø11H22 specimens (cast and coring) and 20 cm for Ø16H32 specimens. In parallel the weight variation is also monitored.

4. RESULTS

The following results illustrate the swelling measured in the different samples. Results show different expansion behaviour in the massive structure in comparison with unrestrained laboratory specimens.

4.1 The effect of boundary conditions on swelling mechanism in massive structures

Figure 4.1 shows the swelling in the mock-up as recorded by the strain sensors. Until the time 235 days, the massive structure is conserved in water at ambient temperature. This phase corresponds to the time required to finalize the construction of the experimental platform and install the temperature regulation system. There's hardly no swelling at this stage. Once the temperature rose to 38°C, swelling in the mock-up started increasing in a similar manner to accelerated tests on laboratory specimens due to the accelerated formation of alkali-silica gel.

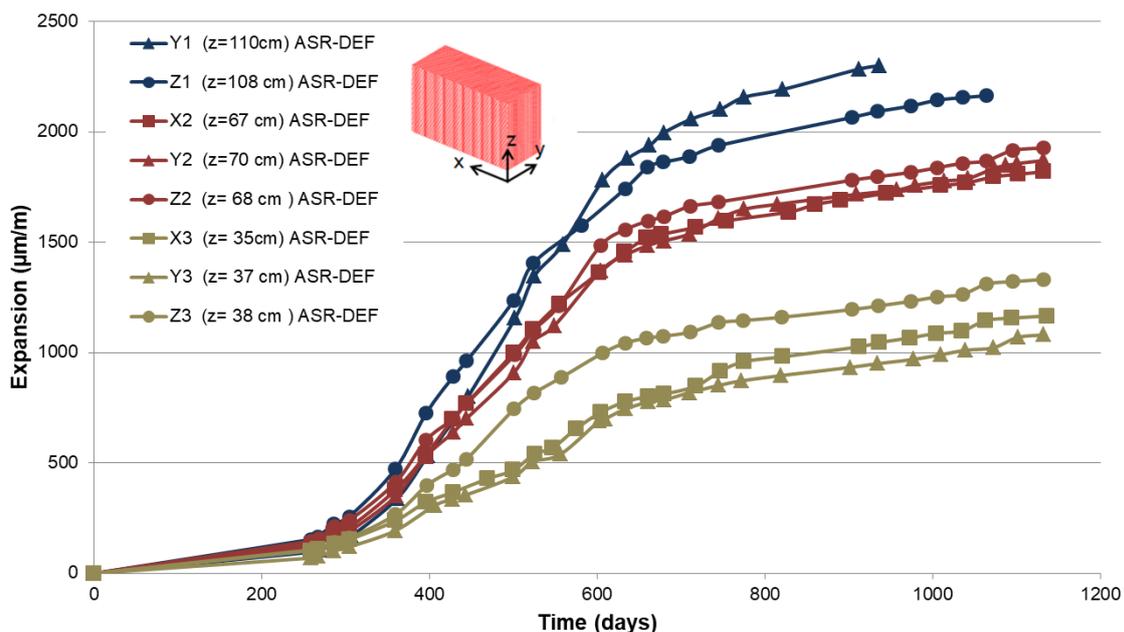


Figure 4.1: Swelling evolution in the massive structure mock-up.

The formation of the alkali-silica gel leads to a drop in the alkalis concentration in the interstitial solution. Such a phenomenon can be assimilated to alkali-leaching hence creating an optimal thermodynamic environment to accelerate the delayed ettringite formation [10].

After 700 days of follow-up, swelling in the structure seems to reach the second inflection point beyond which the expansion tends to stabilize in a linear manner. The maximum amplitude recorded is about 2250 $\mu\text{m/m}$ reaching the maximum capacity of the strain gage. However this value varies according to the position of the sensor and its orientation. As a general rule, the swelling decreases as we approach the base of the structure regardless of the orientation of the sensor. Thus, a $\Delta\epsilon$ of approximately 700 $\mu\text{m/m}$ is measured between the sensors Z1 ($z = 110 \text{ cm}$) and Z3 ($z = 38 \text{ cm}$). A first idea would be to evaluate the impact of the concrete's own weight on the deformations measured by the sensor. If we consider the Z3 sensor, the stress due to the self-weight of concrete is just 0.024 MPa at this point, which discards this hypothesis. Eventually, simulations showed that this behaviour is actually due to the stress generated by the restraint effect due to the metal support on which the structure was cast. The upper part of the mock-up can expand freely whereas the lower part is hindered by the boundary conditions. However, at lower heights, the swellings measured in the horizontal plane parallel to the support (along X and Y) are hindered more than the swelling measured vertically (along Z). Another approach to analyze this behavior would be to consider that the expansion, which is prevented along the directions of the metal support, is compensated by a greater expansion in the unstressed direction (Z). This effect decrease as we approach the upper part of the mock-up. As a matter of fact, the experimental campaign conducted by Larive, has shown that the mean volume swelling remains generally constant when the value of the uni-axial stress applied to the specimens remains below 10 MPa [1].

Macroscopic monitoring of the mock-up permits to correlate the internal swelling of the structure with the evolution of visible cracking. Figure 4.2 features the evolution of the cracking index in the mock-up.

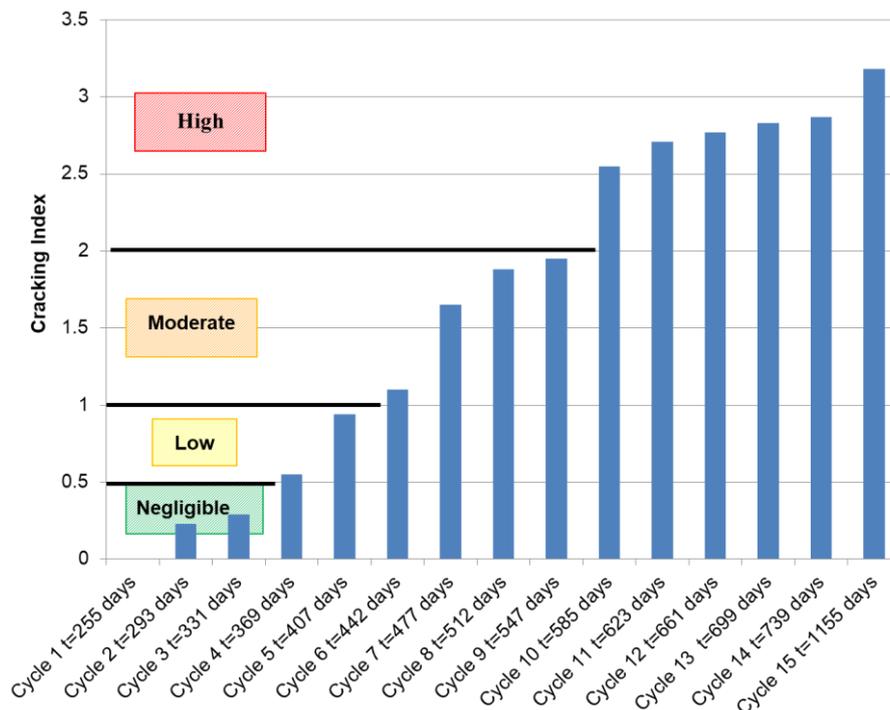


Figure 4.2: Evolution of the cracking index in the ASR-DEF mock-up

According to the classification proposed by this test method, currently the mock-up is subject to a “high” cracking and is considered damaged by the internal swelling reactions. The structure maintained a “negligible” and a “low” cracking during the first cycles which correlates well with the internal swelling measurements. However, a visual inspection of the structure reveals that the cracking is not uniform on its envelope. The cracking index, as a parameter, expresses an average crack opening over one square meter but does not give information on the orientation and distribution of the cracks. Subsequently, in order to enrich the information provided by this test method and to provide a

more global vision of the damage at the structure level, a crack mapping is carried out. The coordinates of the crack points were noted as well as the opening of the corresponding cracks. The cracks are then classified into five categories according to their opening. Figures 4.3 and 4.4 illustrate the map obtained as well as a photograph of the deteriorated mock-up respectively.

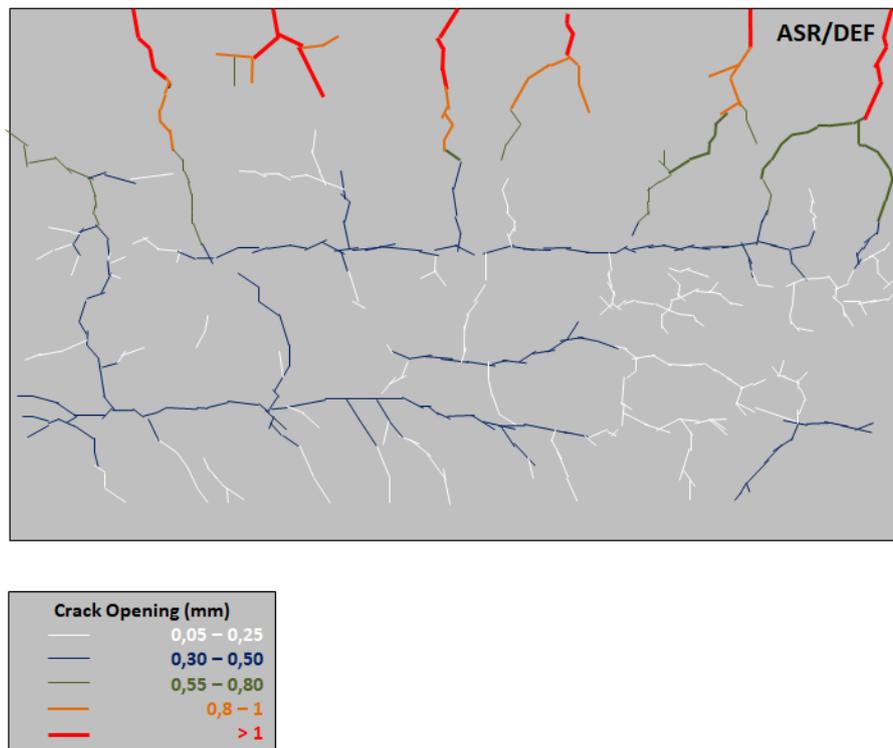


Figure 4.3 : Cracks mapping of the ASR-DEF mock-up at t = 1150 days



Figure 4.4: Photo of the ASR-DEF mock-up at t = 1150 days

We observe the presence of a main longitudinal crack around mid-height and vertical cracks at the top of the mock-up. The opening of the vertical cracks is greater than 1 mm in the upper part and decreases as we approach the lower part. From a mechanical point of view, the vertical cracks on the upper part of the mock-ups correlate directly with the differential deformations measured along the X and Y axis at the 0.75H level in comparison with the 0.25H level. These differential deformations can be explained by the presence of the metal support in the lower part of the structure. In his work on reinforced and non-reinforced beams affected with DEF, Martin [10] shows that the presence of reinforcements reduces longitudinal deformations. In addition to that, the longitudinal cracks are parallel to the metal support of the mock-up. This seems to confirm the hypothesis of anisotropy induced by the presence of steel. In fact, numerous studies show that the cracks orientation of a concrete affected by an internal swelling reaction are a function of the direction of reinforcement or of pre-stressing. The work of Bouzabata [11] show that the swellings induced by DEF are isotropic under free swelling conditions but become anisotropic as soon as a loading is applied. As for ASR, the works of Multon [12] and Charpin [13] join the hypothesis that an external loading (even passive) generates a redistribution of swellings in the least compressed directions.

4.2 The conservation environment effect on the concomitant development of ASR and DEF

Figure 4.5 shows the swelling in the corings subjected to the residual swelling test methods (CR_ASR-DEF_38_100HR and CR_ASR-DEF_20_Imm) in comparison to that of the mock-up.

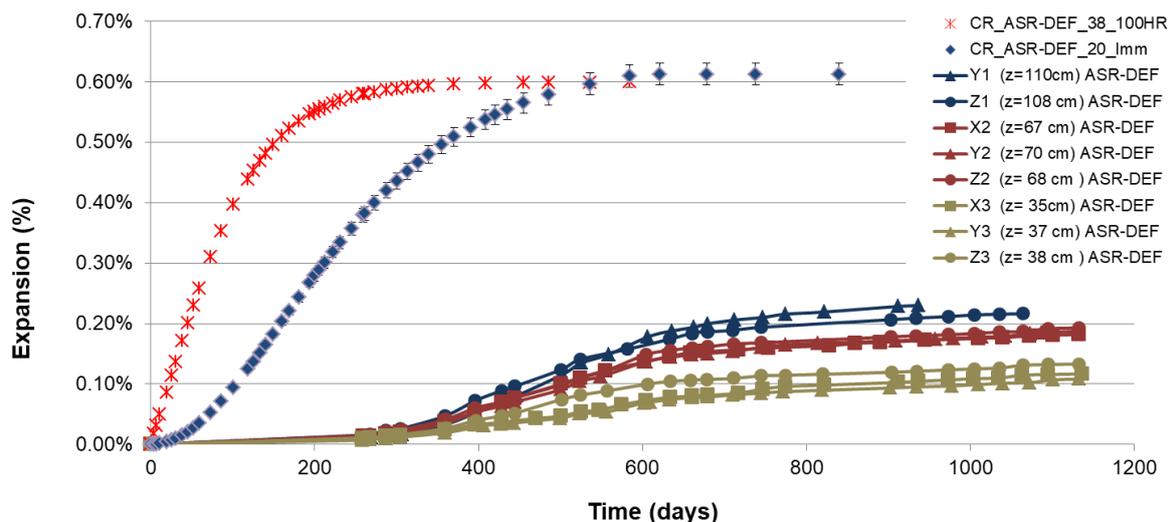


Figure 4.5: Residual swelling in the cores extracted from the ASR-DEF mock-up

The corings yield a residual swelling of 0.59%-0.62% while the maximum strain measured in the upper part of the mock-up is 0.225% which suggests the presence of a scale effect. In addition to that, we notice that the corings exhibit different swelling behaviours depending on the conservation environment.

The specimen stored at 38°C and 100% relative humidity (CR_ASR-DEF_38_100HR) reached a final swelling of 0.59% faster than the specimens immersed at 20°C (CR_ASR-DEF_20_Imm). Various studies show that when a single internal swelling reaction is developing in concrete, the temperature and the conservation environment impact the kinetics and the amplitude of swelling [10, 14-15]. However, when two internal swelling reactions are coupled, the storage temperature can favour a reaction compared to another and thus modify the interactions between pathologies. On one hand, a temperature of 20°C seems to be favourable for the development of DEF as a prelude to the development of ASR, in particular by reducing the solubility product of ettringite. On the other hand, an increase in temperature increases the kinetics of ion transfers and thermo-activates the formation of alkali-silica gel by increasing the solubility of silica [2] which in his turn accelerates DEF by acting by as an additional engine for alkali-leaching. However, a temperature of 38°C decreases the solubility product of ettringite (Baghdadi, 2008), consequently the final volume of delayed ettringite precipitated

is less important, which may explain why the final swelling is less important at 38°C (0.59%) than at 20°C (0.62%).

From another point of view, if we consider solely the effect of the surrounding humidity, the fact that the CR_ASR-DEF_20_1mm specimens show a higher swelling may be due to the fact that they are totally immersed hence providing more water compared to a 100% relative humidity environment. In their works on DEF, Famy [16] and Al-Shamaa [17] have shown that swellings increase in amplitude and in kinetics with higher moisture exposure. This is due to the combined effect of the alkalis leaching which promotes the desorption of sulfates and therefore the precipitation of ettringite, and to the fact that water is a necessary reagent for the formation of ettringite [18]. In conclusion, these results confirm those obtained by Martin [10] in the context of his work on the effect of the conservation environment on the concomitant development of ASR and DEF.

4.3 Scale-effect on swelling kinetics and magnitude

Figure 4.6 shows the swelling in the concrete specimens cast in the laboratory in comparison to that of the structure. A correction was applied to the expansion results of the mock-up taking into account the temperature increase at 235 days and the difference in maturity conditions, thus applying a shift to the left.

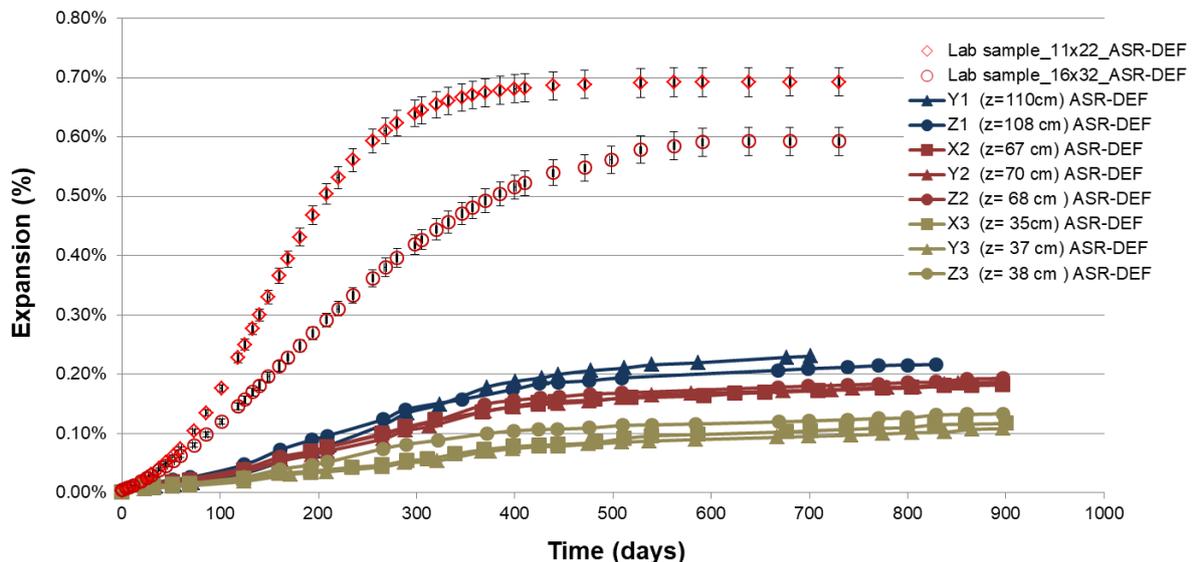


Figure 4.6: Comparison of the swelling evolution between the laboratory scale specimens and the massive structure behavior

By comparing the expansion of the laboratory samples with that of the mock-up, we notice different swelling behaviours: the swelling kinetics and the final expansion decrease as the sample size increase. Knowing that all samples geometries have the same concrete composition, early-age heat profile and an equivalent conservation environment, the swelling characteristics (magnitude and kinetics) are quantified using a mathematical relation (Kchakech's law) given in Equation 1 [19]:

$$\varepsilon(t) = \varepsilon_{\infty} \frac{1 - e^{-\frac{t}{\tau_c}}}{1 + e^{-\frac{t - \tau_L}{\tau_c}}} \cdot \left(1 - \beta \cdot e^{-\frac{t}{\alpha}}\right) + \varepsilon_{hyd} \text{ with } \alpha > 0 \text{ and } 0 \leq \beta \leq 1 \quad (1)$$

Where ε_{∞} is the final magnitude of expansion, τ_L and τ_c are respectively the latency and the characteristic times, and α and β are two parameters to take into account a linear evolution of the last phase of swelling [19].

Table 4.1 recapitulates Kchakech's swelling parameters obtained for the different specimens' series by minimizing the differences between the curve and the experimental points using the least squares method. Sensors in the upper part of the mock-up are considered for this comparison knowing that this part of the structure expands "freely".

Table 4.1: Parameters identification of Kchakech's law

	LabSample ø11H22	LabSample ø16H32	Mock-up – sensor Y1 (z= 110 cm)	Mock-up – sensor Z1 (z= 108 cm)
ε^∞	0.69%	0.61%	0.2225%	0.2050%
τ_L	135	150	290	250
τ_C	56	125	90	90

We notice that the latency and characteristic times increase and that the final swelling decrease as the sample size increase.

From a mechanical point of view, by assuming that the swelling in the concrete generates a tensile solicitation, then the decrease in the final swelling can be explained by the Weibull's effect [20]. Rossi's research regarding the scale effects on the mechanical behaviour of concrete suggests that the tensile strength of the concrete decreases as the volume of the specimen increases [20].

Given that the ISR kinetics can be assimilated to a diffusive phenomenon, figure 4.7 shows a normalized evolution of the swelling in the different specimens function of $(\sqrt{t})/r$, where t stands for time (days) and r is the radius of the specimen (m). An equivalent radius was computed for the massive structure taking into account the shape factor (parallelepiped vs cylinder) and yielding the same cross-section area ($r_{\text{mock-up}} = 0.668$ m).

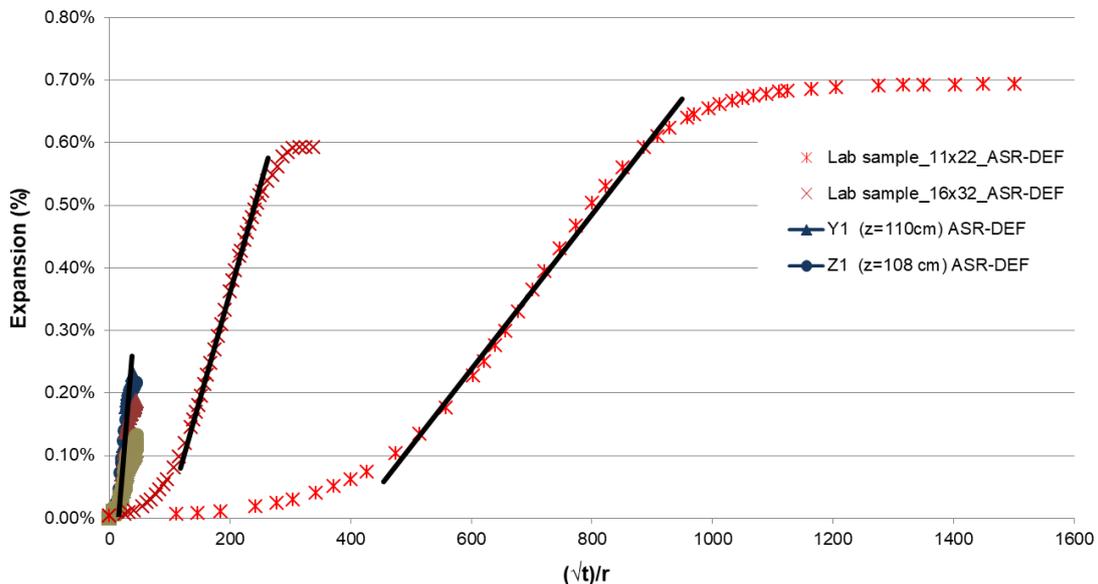


Figure 4.7: Normalized evolution of the swelling in the different specimens sizes

Figure 4.7 reveals different swelling slopes in the normalized graph. This suggests different reaction kinetics and swelling mechanisms. If we consider that we have the same chemical advancement in the three specimen sizes (same pore distribution, same aggregate-cement paste interface...); then a possible explanation is the following: as the sample size increase, crack occurrence increase hence creating stress release points and additional space for swelling products thus slowing down the overall expansion mechanism. Moreover, the fact that the samples vary in geometry and size and are immersed in water, yield various volume to exposed surface ratios ($V_{\text{concrete}}/S_{\text{exposed}}$) as detailed in table 4.2.

Table 4.2: Volume to exposed surface ratios

	∅11H22	∅16H32	Mock-up
Concrete Volume (L) - V_c	2,091	6,43	3360
Exposed Surface (m ²) - S_e	0,095	0,20	11,92
Average radius r_{avg} (V_c/S_e) (m)	0,022	0,032	0,282
$r_{avg}/r_{\phi 11H22}$	1	1,45	12,82

This factor impacts the ionic transfers between the porous medium of the concrete and its conservation environment, and more precisely the diffusion of the alkalis in the conservation water. Figure 4.8 features the relative amounts of equivalent alkalis leached at each cycle for both categories: laboratory samples and the mock-up. The values are expressed in percentage with respect to the initial quantity contained in the test specimen.

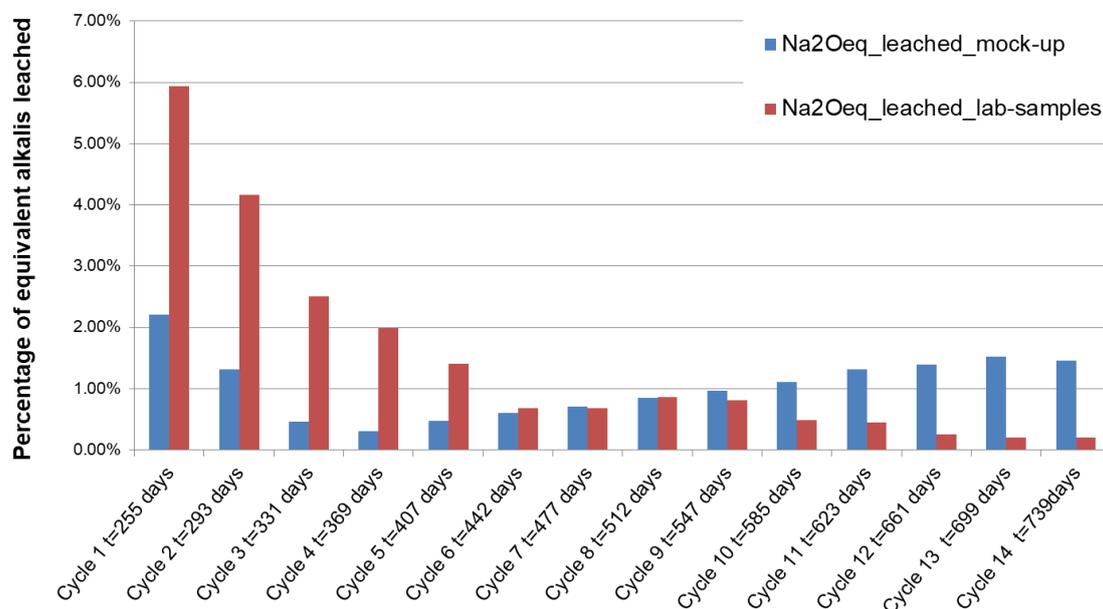


Figure 4.8: Quantities of alkalis leached at each cycle

We see that the quantity of leached alkalis gradually decreases over time in the case of the laboratory samples. Conversely, in the case of the mock-up, the quantity of leached alkalis decreases during the first cycles then re-increases after cycle 4. The reduction in the first cycles is due to the leaching of the alkalis found in the concrete skin. However, the increase in the quantities of potassium and sodium leached after cycle 4 is due to the development of cracking in the structure, as shown in figure 4.2, which increases the interaction surface between water and concrete.

In summary, despite the provisions taken in order to create an equivalent conservation environment, the laboratory samples reproduce neither the mechanical behaviour nor the ionic transfers as the massive structure and specifically the alkalis leaching. This phenomenon has a huge impact on the kinetics of ISR, either by delaying ASR or by accelerating DEF or by promoting a pathology compared to another in the case of the concomitant development of ASR and DEF. Results indicate the presence of a scale effect on the development of ISR in terms of kinetics and amplitude. However this point ought to be clarified by further research considering additional intermediate geometries and microscopic observations.

5. CONCLUSIONS

We show in this paper that expansion due to internal swelling reactions in massive structures varies greatly from analogous laboratory samples. Boundary conditions could hinder expansion along certain directions thus compensated by a higher expansion along unstressed directions which leads to an anisotropic behaviour in the material. Moreover, the conservation environment and in particular the storage temperature and the moisture exposure have a huge effect on the concomitant development of ASR and DEF. It can favour a reaction compared to another and thus modify the interactions between pathologies. The most important observation is that we identified the existence of a scale effect on internal swelling reactions. By comparing the strain evolution in different samples sizes subject to analogous concrete composition, thermal history and conservation environment, we found that the final swelling amplitude and the swelling kinetics are reduced by increasing the specimen's size. This behaviour may be explained by the mechanical behaviour of the different specimens' categories and/or by the kinetics and the quantities of the alkalis leached in each case.

It ought to be mentioned that similar results were obtained in a parallel study investigating the Delayed Ettringite Formation (DEF) and ASR separately.

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