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A B S T R A C T

Delayed Ettringite Formation (DEF), is one of the different Internal Sulfate Attack (ISA), is a slow chemical reaction that can be responsible for the degradation of cementitious materials, through the swelling of the material followed by crack diffusion in the structure. The objective of this study is the experimental characterization at the local scale (10 x 10 x 30 mm^3 samples) of the concrete reached by DEF. The composite samples, consist of a 15 mm thick cement paste placed in contact with a siliceous aggregate. The shape of these samples makes it possible to study the mechanical behavior of the interface between the cement paste and the aggregate. This zone is identified as a privileged zone of the development of DEF. Given the slowness of this pathology, the experimental setting in the laboratory is accelerated by choosing conditions favoring the appearance of DEF. The tests are exploited at the local scale by measuring expansions by image correlation of the degraded samples and by scanning electron microscope observations. The results showed a higher expansion at the cement paste-aggregate interface compared to the cement paste. A tensile test performed at local scale allowed to characterize the impact of DEF on mechanical properties of the cement paste-aggregate interface. A drop in strength was observed with heterogeneous formation and localization of ettringite inside the interface.

Keywords:
Cement paste
Cement-aggregate interface
Delayed ettringite formation
Swelling
Local scale

1. Introduction

At young age, the concrete temperature rise may be due either to the exothermic reaction of cement hydration, especially in the massive structures (dams, bridges, nuclear power plants ...) or to the prefabrication where a warm-up is applied to the material to accelerate concrete curing and allow quicker stripping. Under other environmental conditions, this temperature elevation can induce delayed ettringite formation pathology after concrete hardening causing swelling of the structure and cracks formation [1,2]. The phenomena and parameters at the DEF origin are not yet all well identified.

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Various studies are performed to identify parameters influencing the DEF. Several factors have been identified, but their precise roles and degrees of influence are often not well understood. Among the mentioned parameters:

- Curing temperature: this is the most studied parameter. The majority of the scientific community [3–7,1,2] accepts its role. It has been shown that the expansion is strongly related to the applied cure temperature and that the DEF may appear at temperatures between 60 and 70 °C under certain environmental and chemical conditions (alkali content, sulfates, relative humidity).

- Effect of conservation conditions: water plays a key role in the appearance of this pathology. It is one of the reagents necessary for the ettringite formation and serves as a reaction medium. Most of the studied cases are on structures exposed to environments with high humidity or even completely submerged structures. [8,9,4]

have shown that for Relative Humidity (RH) of less than RH = 90% the specimens do not swell, while for RH ≈ 100% or samples immersed in water present swelling may occur.

- Cement characteristics: alkalis play a vital role in the DEF. The high concentration of alkali in cement increases the solubility of ettringite [10–15]. In addition, studies have shown that as the equivalent alkali content increases, the kinetics and the amplitude of this pathology increase [16,17].

Microscopic observations made on materials affected by DEF have many similarities: ettringite is present in large quantities. It is often localized in porous zones: pores and cement paste-aggregates interfaces [17,6]. The occupation of a large quantity of ettringite in these spaces after the cement has hardened causes cracking of the concrete. Most of the experimental studies on DEF are carried out at macroscopic scale on 11 cm x 22 cm or 16 cm x 32 cm cylindrical specimens [2,10,6,13,14]. The development of this pathology at the microscopic scale, and more particularly at the scale of the interface between the cement paste and the aggregates, remains little discussed in the literature. Therefore, the main objective of this study is the characterization of the delayed ettringite formation at the cement paste-aggregate interface scale by monitoring the swelling, by performing mechanical tensile tests at this scale and by realising microscopic observations during the pathology progress [18,19].

2. Experimental program

The tests proposed in the literature are essentially based on macroscopic samples. There are few tests at the scale of the cement paste-aggregate interface, and even less on the DEF pathology. This study is dedicated to the multi-scale characterization of the concrete degradation by DEF on the one hand, by performing at local scale (ITZ), a microscopic visualization with the Scanning Electron Microscope (SEM), then a local characterization by measurement of the swelling at the cement paste-aggregate interface. On the other hand, one analyzes and compares the results obtaines at a Representative Elementary Volume (REV) scale, on macroscopic concrete samples with those of the literature in order to validate our experimental protocol.

In this section, the experimental approach employed is defined by presenting the chosen protocol to define realistic tests as well as the data relating to the experimental materials and techniques selected.

2.1. Size samples

To identify the location of degradation at the cement paste-aggregate interface, local samples are made using aggregates bonded with a cement paste (Fig. 1). Called composites [18,19], the samples have a parallelepipedic shape (10 × 10 × 30 mm^3). Cement paste samples with the same size as composites serve as reference. For the macroscopic samples, 11 × 22 cm^2 cylindrical specimens were chosen, called REV samples. Tests at the REV scale were carried out in order to choose the materials and the effective experimental protocol to trigger DEF and not to compare directly the results.

2.2. Materials

The chosen materials help to accelerate the pathology appearance. We emphasized that the nature of the aggregates and the chemical composition of the cement played a decisive role. Numerous studies carried out on concrete with different nature of aggregates have shown the impact of aggregate mineralogy on DEF development [20,21]. Siliceous aggregates are associated with greater swelling than of limestone aggregates. Considering the important role played by the cement paste-aggregate interface in the DEF reaction mechanisms [22–24], siliceous aggregates were used during this study (from the Oscar Savreux quarry). This type of aggregate has demonstrated its responsiveness to DEF [17,2].

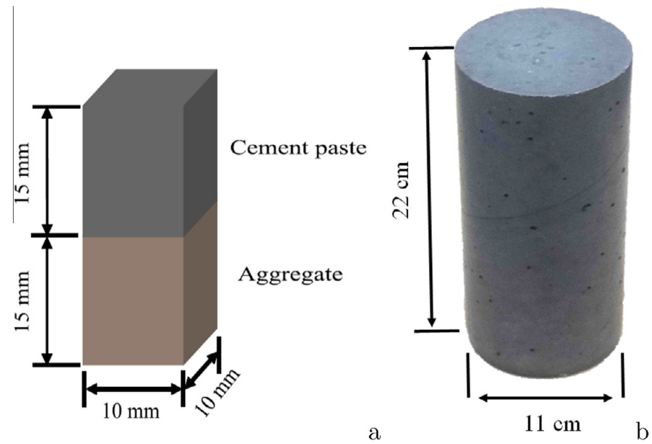


Fig. 1. Geometric configurations and sizes of the samples: a) composite, b) REV sample.

For the composites, the aggregates were taken from a siliceous rock. The rock was cut to obtain the final shape of the aggregate: parallelepipedic shape of dimensions 10 × 10 × 15 mm^3. For REV samples, two sand granular cuts 0/4 mm (sand) and 4/ 12.5 mm (gravel) from the Oscar Savreux quarry also are chosen.

The cement used is a CEM I 52.5 R CE CP2 NF. This cement is rich in alkalis and its contents of sulfates and aluminates are in proportions supposedly favorable to DEF (it is rich in sulfur trioxide 3.59%). The equivalent alkali content (Na_2O_{eq}) of this cement is equal to 0.35%. Tables 1 and 2 present the chemical composition of this cement and the formulation of concrete.

Among the essential chemical parameters for the onset of DEF, there is the equivalent alkali content. Studies have shown that when the equivalent alkali content increases (⩾ 1%), the kinetics of DEF increases [16,17]. It was thus decided to increase the alkali content (initially 0.35%) to 1.2% [8,25,14] by adding Na_2SO_4 to the cement with a proportion of 6.77 kg/m^3 for both types of samples Table 2.

2.3. Heat treatment

It is accepted that the temperature reached by the concrete at young age is a decisive parameter for the DEF development [6].

Table 1
Chemical composition of CEM I 52.5 R.

components	K_2O	SO_3	Fe_2O_3	Al_2O_3	MgO	Na_2O	Na_2O_{eq}
content (%)	0.35	3.59	3	4.15	1	0.16	0.35

Table 2
Concrete formulation for REV sample.

Components	Dosage kg/m^3
Cement	424
Sand	669
Gravel	1102
Na_2SO_4	6.77
Water/Cement ratio	0.47

In order to accelerate the occurence of this pathology, the samples underwent an hydrothermal treatment, directly after their manufacture, using a climatic chamber (Weiss fo REV samples and Memmert CTC256 for composite samples) which allows imposing the desired thermal and hydrous cycles.

The chosen thermal cycle is both close to those used in various laboratory studies [26,4] and representative of a heat treatment carried out in the concrete industry. The thermal cycle shown in Fig. 2 is adopted for REV samples with 3 days at 80 °C at 95% RH.

For the composites, our first choice was to apply the same heating as to the REV samples. After applying this thermal cycle to the composites, the samples did not resist and were damaged prematurely: given the samples sizes, the thermal cycling is too aggressive and produces cracking of the composite at the cement paste-aggregate interface, as shown in Fig. 3.

Given the small size of the samples and the sensitivity of the interface cement paste-aggregate (Fig. 1), the heat treatment must be adapted by reducing the duration of the cure temperature which is generally accepted for macroscopic samples [27]. After several tests to determine the appropriate cycle, it was retained an hydrothermal cycle with a rise in temperature of 5 °C/hour followed by a step of 2 h at 80 °C while maintaining a relative humidity close to RH = 95% (Fig. 4). After the heat treatment, the REV samples underwent two drying/humidification cycles to accelerate only the kinetics of expansions by pre-damage [28,17]. The drying/humidification cycle has not been applied to the composites because it causes the cracking of the interface.

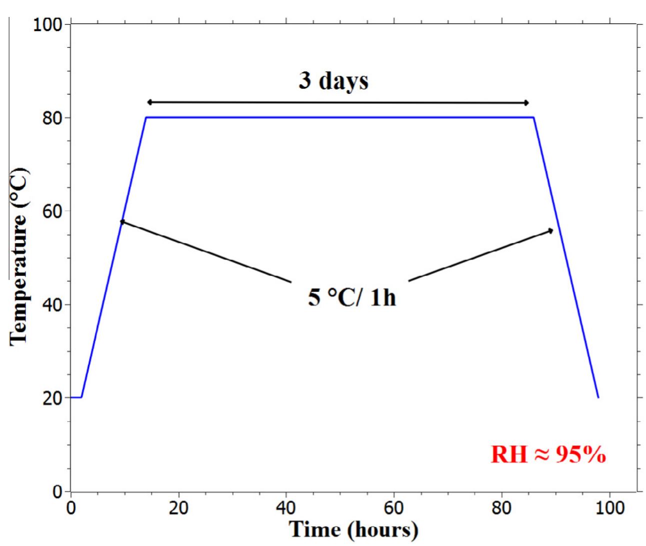


Fig. 2. Hydrothermal treatment applied to the REV sample.


Fig. 3. Composite cracking after the thermal cycle of 80 °C during 3 days.

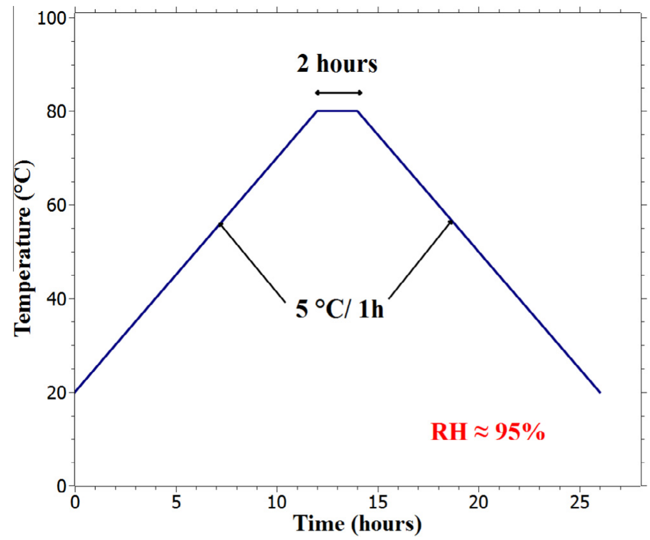


Fig. 4. Hydrothermal treatment applied to composites.

Table 3
Summary of the DEF experimental protocol.

	REV	Composite
Temperature	80 °C	80 °C
Humidity	95%	95%
Duration	3 days	2 h
2*Drying/humidification	7 days/7 days	no
Conservation	water (38 °C)	water (38 °C)

After applying this thermal cycle, the samples are kept in a non-renewed distilled water tank at a temperature of 38 °C. The Table 3 presents a summary of the experimental protocol for the study of DEF at two scales.

2.4. Tracking expansions

Tracking expansions are different with the two shape and size samples. For the REV samples, two measurements of expansion of the specimens are made:

- the external measurement using an extensometer of resolution less than one micrometer (Fig. 5a), where six studs are glued on each test piece after the heat treatment;

- the internal measurement using special gauges embedded in the concrete sample (Fig. 5b).

For the composites, several methods were tested (gauges, extensometer and image correlation) to track sample expansions. The image correlation seemed more suitable for the size and conditioning of these samples. This method includes depositing a speckle pattern (Fig. 6a) on one side of the sample and then photographing this face every seven days during the development of the pathology. The speckle is applied in our case with a projection of white paint on the surface of the black painted sample. To locate the interface position, red marks were placed on the sample. The expansion measurement is performed by calculating the distances between well identified points (Fig. 6b).

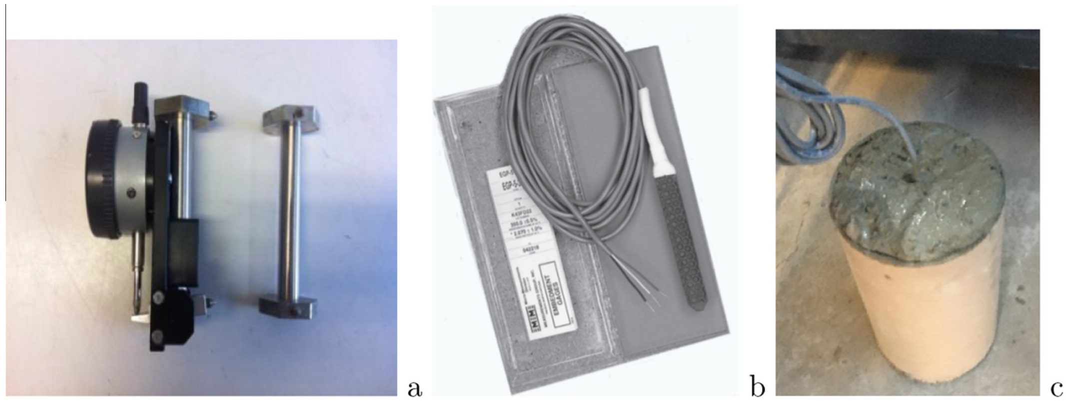


Fig. 5. a) Extensometer, b) Strain gauge c) Strain gauge embedded in the sample.

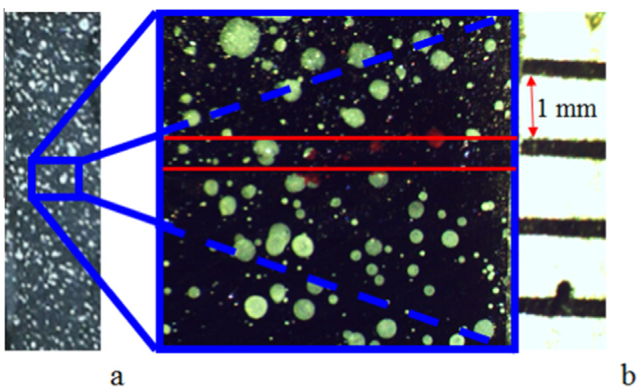


Fig. 6. a) Composite with his speckle, b) zoom of the speckles at the interface: 3 mm scale with the graduation beside.

At a given instant, Δl displacement measurements is systematically performed on the degraded composites $\Delta l_c = l_c - l_{c0}$ where l_{c0} and l_c are respectively the initial lengths (index 0) and the lengths at a given moment of the composite degradation (index c) between two selected points (Fig. 7). Other measurements were made on the cement paste $\Delta l_p = l_p - l_{p0}$ where l_{p0} and l_p are respectively the initial lengths (index 0) and lengths at a given moment of the cement paste degradation (index p).

The displacement measured on the composite l_c does not represent the displacement at the interphase. Nevertheless, by means of the displacement measurements l_p^{ref} carried out on the reference samples in cement paste (supposed homogeneous), one can deduce the displacement at the interface l_i of the composite by the following Eq. (1):

$$\Delta l_c = \Delta l_g + \Delta l_i + \Delta l_p \tag{1}$$

with Δl_g and Δl_i displacements of the aggregate and the interface during the degradation. The measurements of the aggregate expansions show that they are negligible ($\Delta l_g = 0\ \mu m$). Therefore, the expansion at the interphace ϵ_i can be written:

$$\epsilon_i = \frac{\Delta l_i}{l_{i0}} = \frac{\Delta l_c - \Delta l_p}{l_{i0}} = \frac{\epsilon_c \times l_{c0} - \epsilon_p^{ref} \times l_{p0}^{comp}}{l_{i0}} \tag{2}$$

where ϵ_c and ϵ_p represent respectively the expansion of composite and cement paste. The length $l_{i0} = 20\mu m$ is the initial interface thickness measured at SEM and l_{p0}^{comp} is the initial thickness of the cement paste in the composite.

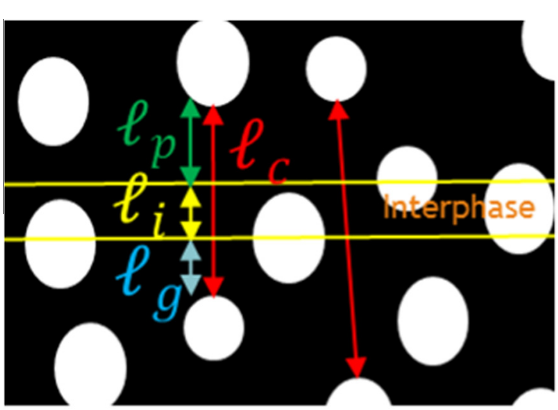


Fig. 7. Expansion measurement method for composite.

2.5. Mechanical tests

The mechanical tests were performed for REV and composites samples. Both sound and degraded samples were subjected to mechanical tests.

For the REV samples, compression tests were performed at different times of hydration and degradation. In order to perform the tests, the surface of the samples was rectified, then placed and centered under the press (Fig. 8a). The test is controlled by loading with a load rate of 0.5 MPa/s.

For composites samples, direct tensile tests were performed at constant loading speed of 0.01 mm/s. The direct tensile test is carried out with specific accessories which were produced and adapted to local testing. The sample is fixed to the machine by means of the rods. The Fig. 8b shows the used experimental device. For each time of hydration and degradation, at least three samples are subjected to tensile test. Before each test, the dimensions and weight of each sample were measured. The mechanical devices were detailed in [18,19].

3. Results

In this section, the expansion due to the DEF is presented and discussed. A preliminary study was carried out on sound samples to characterize the mechanical properties during the hydration process at two scales: cement-aggregate interface and macroscopic.

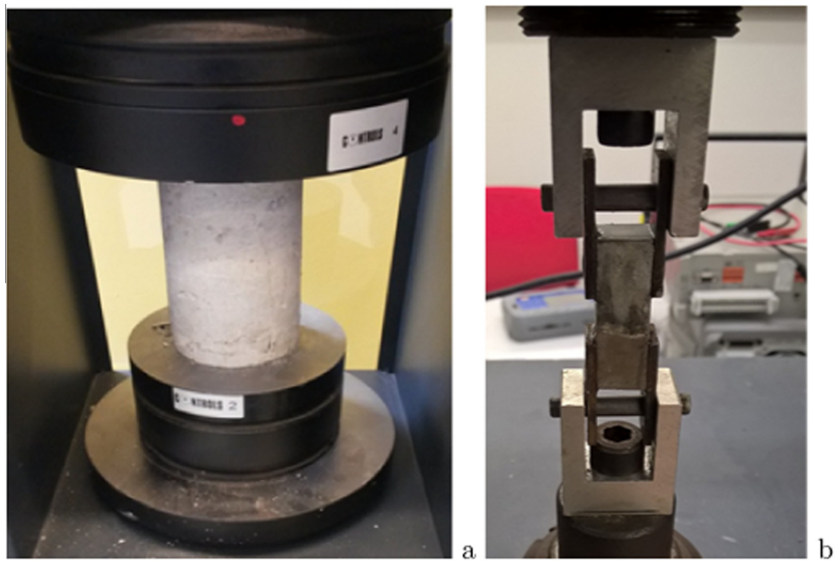


Fig. 8. a) Experimental devices for compression test (REV) and b) direct tensile test (composite).

3.1. Sound samples

The sound samples are conditioned in water saturated with lime directly after preparation. The results on the mechanical properties characterization of sound samples during hydration are presented on Figs. 9 and 10. At each hydration time (2, 7, 15 and 28 days), three samples are submitted to compression and direct tensile tests. At local scale, this study makes it possible to evaluate the mechanical properties of cement paste-aggregate interface (reference samples).

Figs. 9 and 10 respectively show the evolution of the compressive strength of REV samples and tensile strength of the composite and cement paste according to hydration time.

We observe that compressive and tensile strength increase with hydration and stabilize around 15 days. At local scale, the values of cement paste tensile strength are higher than those of the composites. The difference observed at a local scale between the cement paste and the composite is due to the presence of ITZ, which is characterized by a higher porosity. As well as siliceous aggregates are not reactive with the cement matrix. Therefore a low strength is observed.

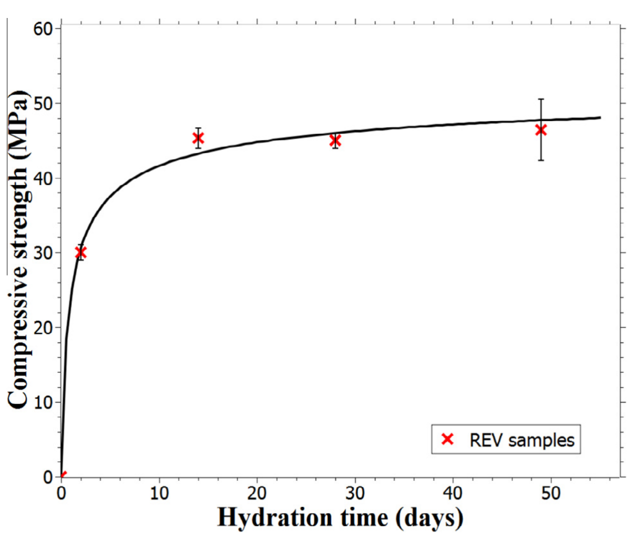


Fig. 9. Evolution of the compressive strength as a function of the hydration time of REV samples.

The mechanical properties of the composite, cement paste and REV sample are presented in the Table 4.

3.2. Delayed ettringite formation

3.2.1. Macroscopic scale

The expansion measurements are made with an "Extensometer" and "Internal gauge", respectively. Fig. 11 shows sample

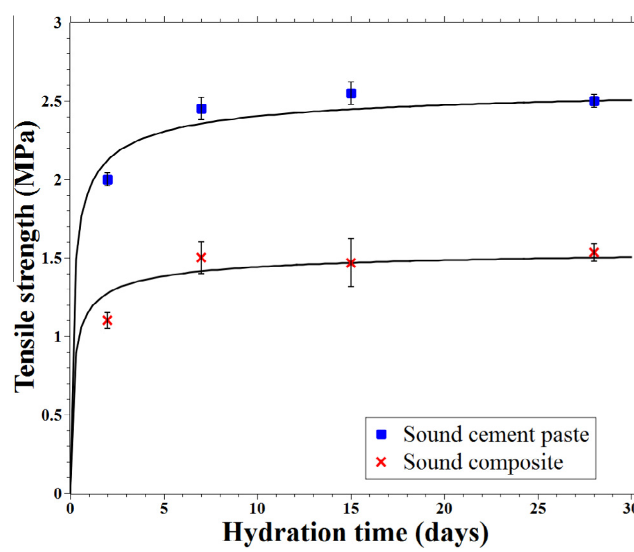


Fig. 10. Evolution of the tensile strength as a function of the hydration time of cement paste and composite.

Table 4
Mechanical properties of the composite, cement paste and REV sample

Sound	cement paste	composite	REV
Tensile strength at 28 days (MPa)	2.5	1.5	no
Compressive strength at 28 days (MPa)	no	no	45

expansions as a function of time in water. It is observed that the concretes which have been subjected to thermal treatment, drying/humidification and immersed in the water develop a swelling. The speed of swelling starts to increase clearly from 200 days, with similar results for both measurement types (internal and external). A sinusoidal shape is obtained similar to that observed in the literature and a stabilization is obtained after 350 days.

3.2.2. Interface scale

An image correlation protocol is being developed to control the suitability of expansion measurements at the cement paste-aggregate interface. The monitoring of these quantities is fixed at a single measurement per week. Each value determined for the composite corresponds to the average of three sample expansion measurements. Other expansion measurements are made on the cement paste (ref) to compare the results with the same frequence.

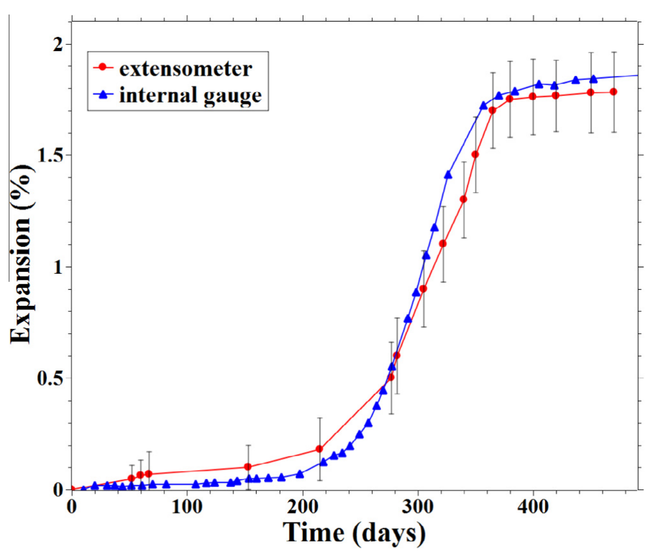


Fig. 11. Average expansions of macroscopic samples at 38 °C without water renewal.

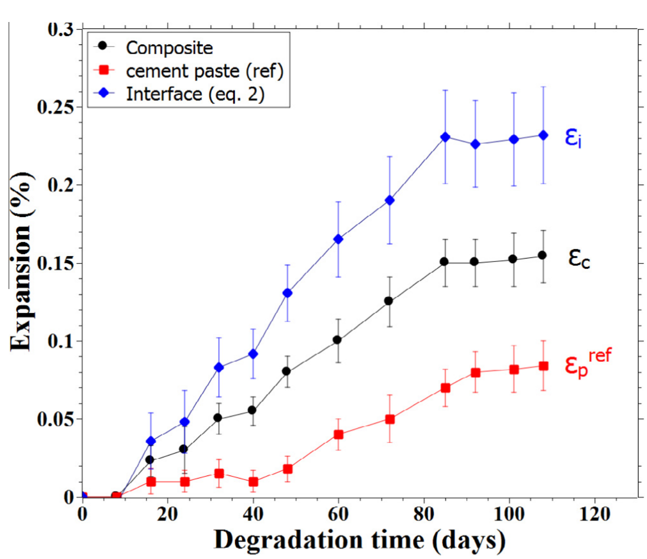


Fig. 12. Average expansions of composites and cement paste stored at 38 °C without water renewal after 110 days.

Fig. 12 shows the expansion at the cement paste-aggregate interface (ϵ_i), in the composite (ϵ_c) and the expansion of the cement paste (ϵ_p^{ref}) depending on the conservation time in water.

Much more swelling is observed at the cement paste-aggregate interface than in the cement paste. The kinetics of expansion of the interface increases significantly from 40 days with a higher kinetics for composites than cement paste. The experimental procedure adopted made it possible to trigger the phenomenon of DEF at the cement paste-aggregate interface scale. However, the crystallization of ettringite at the cement paste-aggregate interface does not lead to the development of sufficiently high crystallization pressures compared to REV samples. To confirm that the observed expansions are due to the delayed ettringite formation, a scanning electron microscopy is performed.

3.3. Observations by scanning electron microscopy

Scanning electron microscopy (SEM) provides important informations on the phase distribution at the microstructure level and the hydrate texture of the cement paste. The aim here is not to report in detail the synthesis of the observations made at the SEM, but rather to identify the main characteristics affected by the DEF degradation of the concrete, and to confirm that the observed expansions are due to the DEF. SEM observations are performed on all the degraded samples on polished surfaces for both types of samples: REV and composites (Fig. 13).

For macroscopic samples, the observations are made on areas favoring the formation of ettringite as cement paste-aggregate interfaces, pores and cracks. Fig. 14 shows three BSE (Backscattered electron) images.

The symptoms of DEF are easily identifiable at macroscopic scale: ettringite is precipitated in mass around aggregates and in cracks. Here, we observed that there was no ettringite around some of aggregates, or only on a part of them. To confirm this remark, SEM observations were made on the aggregates surfaces by using the technique of fractography. Fig. 15 shows the pictures obtained after 200 days of degradation. For a REV sample, the study of a fracture zone of a concrete during degradation shows the localized ettringite on the aggregate surface.

The main difficulty for the composite is the SEM preparation of the samples without damaging them, since the composites present a fragility zone at the interface which is accentuated by the thermal cycle at the young age triggering the pathology. An example of visualization at the interface scale is shown in Figs. 16 and 17: ettringite (rich in Al and S) precipitated in mass around aggregate.

The observed symptoms are identical to those reported by other authors in proven cases of DEF at macroscopic scale. This is explained, according to [29,30,6], by the migration of the sulfate ions contained in the paste towards the porous zones such as the

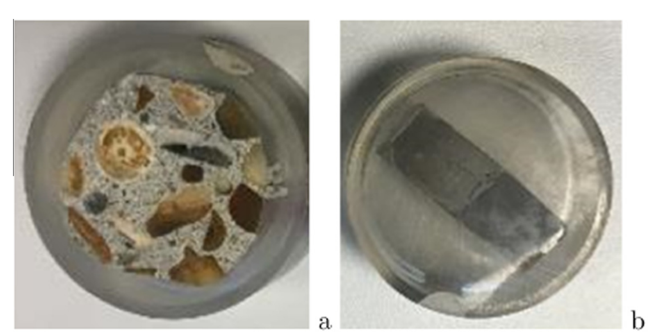
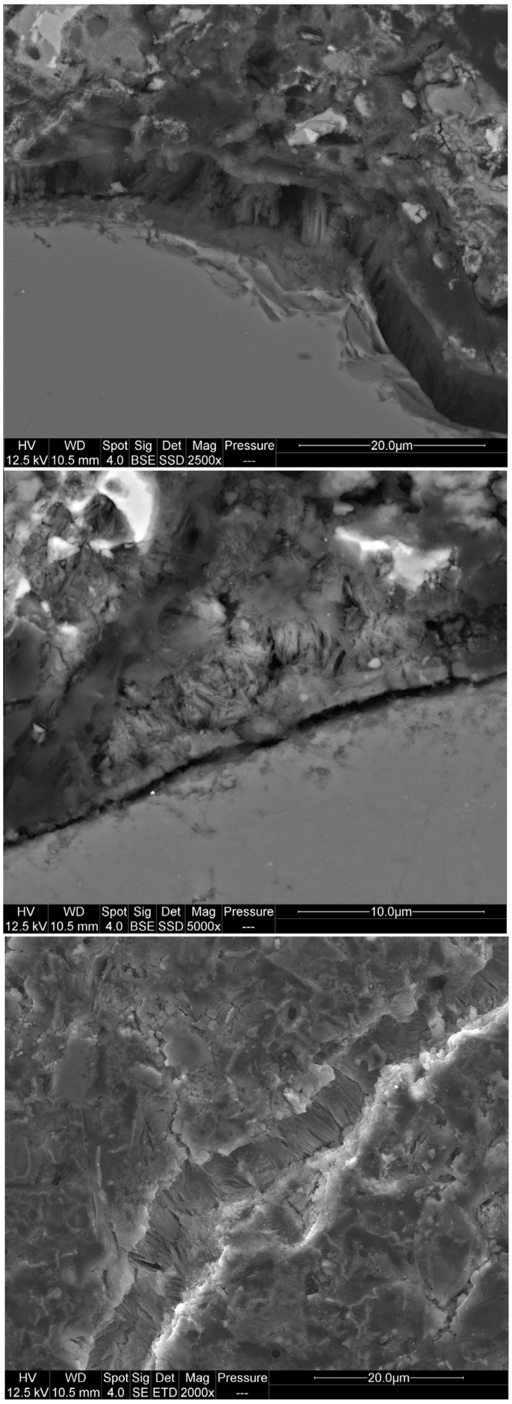


Fig. 13. a) SEM observations: example of a REV sample and b) a composite sample embedded in epoxy resin.



a

b

c

Fig. 14. a) Massive ettringite in all cement paste-aggregate interface after 160 days of degradation b) ettringite on a localized area of the interface after 160 days of degradation c) massive ettringite in a crack after 380 days of degradation of REV samples.

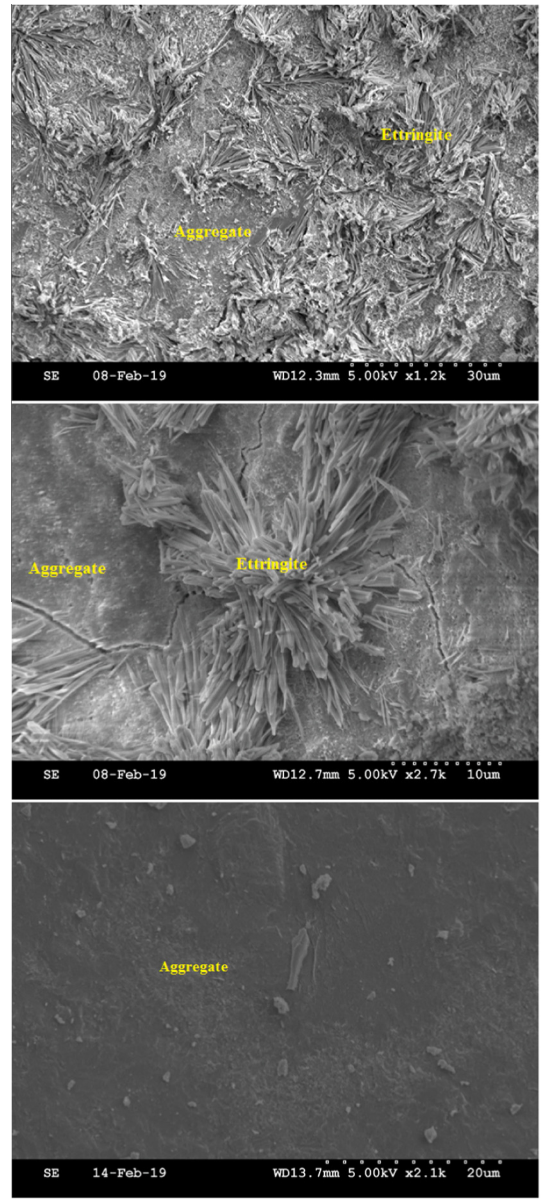


Fig. 15. Fractography observation of the aggregates surface at different area of the same REV samples after 200 days of degradation.

cement paste-aggregate interface, due to favorable transfer conditions, and formation of ettringite. The SEM observations have also made it possible to explain that the ettringite formation at the cement paste-aggregate interface does not lead to the development of significant crystallization pressures (if we compare the expansion at two scales) due to ettringite precipitating in the form of parallel needles (Fig. 16) [31]. Also, these observations show that there is no alkali reaction because no characteristic symptom of a possible alkali-silica reaction was found [32]. These results allow concluding that the applied thermal cycle induces some DEF at this scale.

The tests carried out on the composites make it possible to confirm observations made on the macroscopic samples in the literature [33], namely that the zone of the cement paste-aggregate interface is the privileged zone for the development of the DEF. During the SEM observations, it was noted that ettringite does not form homogeneously over the entire interface of the same sample: areas with a higher concentration of ettringite than other areas at the interface are observed. Fig. 18 shows an area with a low content of delayed ettringite from the same sample shown in the Fig. 16. Fig. 16 displays a high content of delayed ettringite at the interface.

Ettringite formation at the cement paste-aggregate interfaces is a well-known symptom of delayed ettringite formation. It is even one of the main diagnostic criteria for this pathology on concrete. While, ettringite does not form on the entire aggregate surface. This remark was observed on the macroscopic scale also. Therefore,

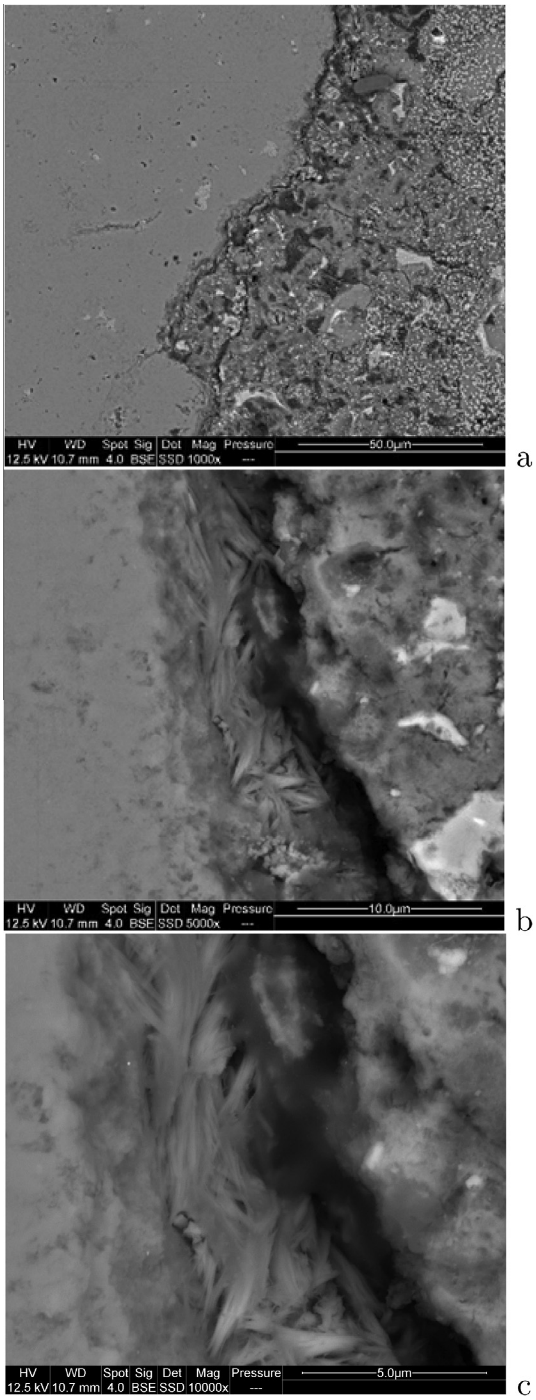


Fig. 16. a) SEM observation of ettringite located at the cement paste-aggregate interface after respectively 32 days and b) 80 days of degradation and c) a zoom on the zone of the concentration of ettringite.

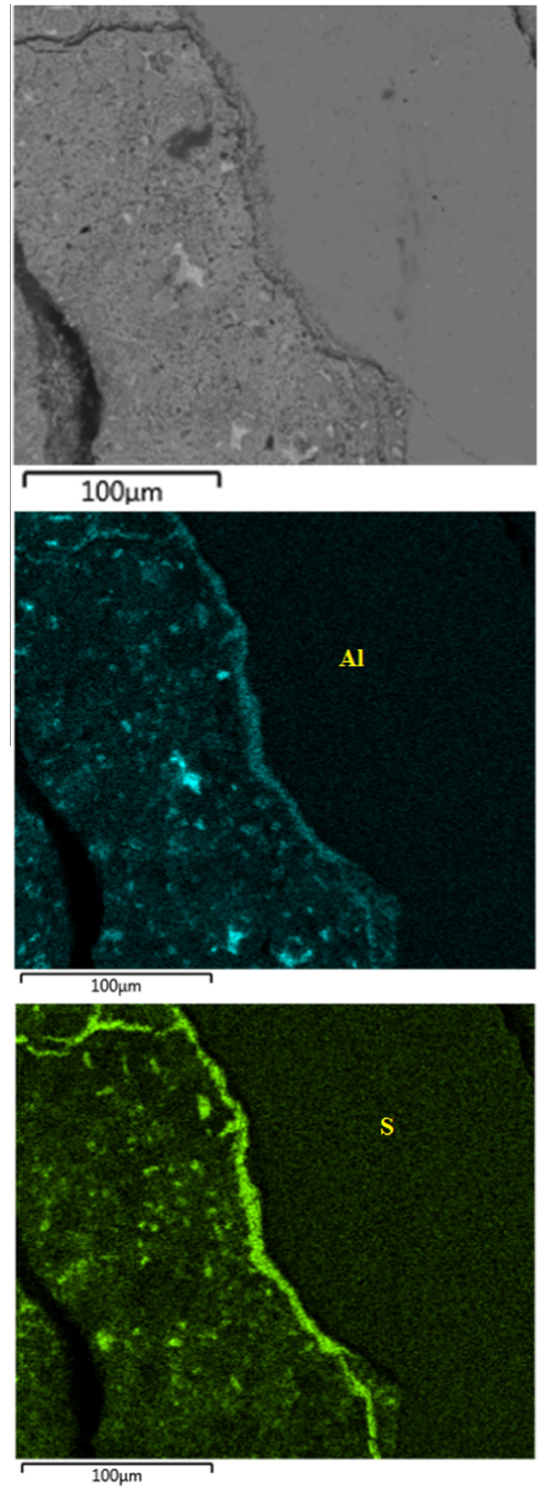


Fig. 17. SEM mapping of cement paste-aggregate interface after 100 days of degradation.

this non–homogeneous formation of ettringite at the interface will influence the mechanical properties of this zone.

3.4. DEF effect on mechanical properties

In this section, we present the results obtained in mechanical tensile tests on samples chemically degraded by DEF.

Compression tests on REV samples were carried out to study the DEF effect at the macroscopic scale. Fig. 19 shows the evolution of the compressive strength as a function of degradation time. A small decrease in compressive strength is observed at this scale. It can be concluded that the DEF does not influence much the compressive properties.

At each degradation time interval, the composites were subjected to direct tensile test. Fig. 20 shows the evolution of the tensile strength as a function of degradation ratio $\epsilon_d/\epsilon_{max}$, where ϵ_d represents the interface expansion at day "d" and ϵ_{max} represents the maximum expansion of interface.

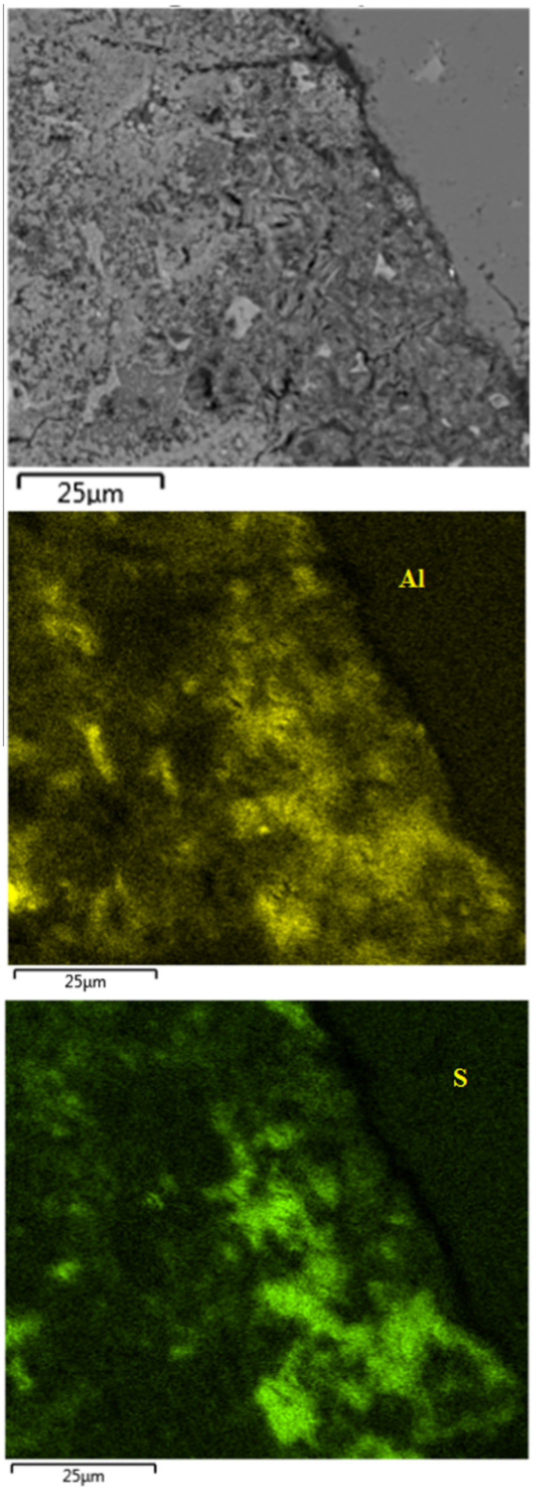


Fig. 18. Lack of ettringite in cement paste-aggregate interface after 100 days of degradation.

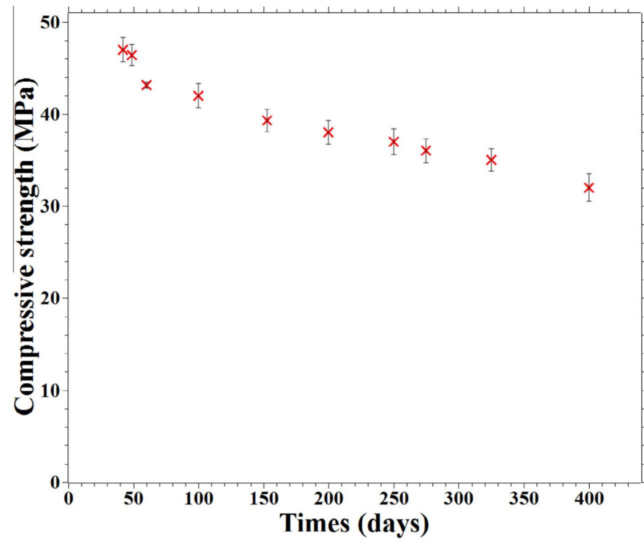


Fig. 19. REV samples: evolution of the compressive strength as a function of degradation time.

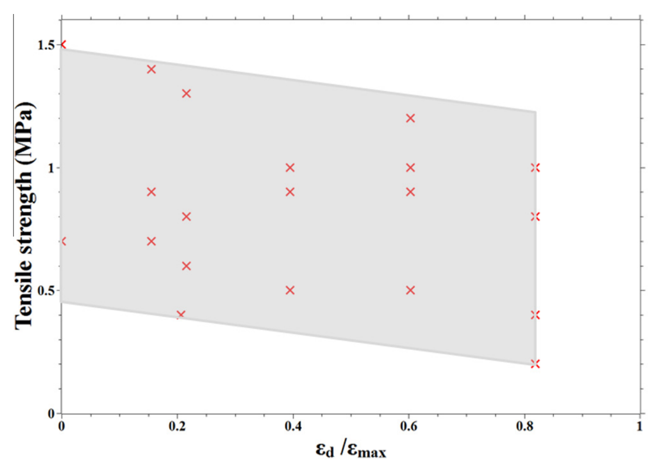


Fig. 20. Evolution of the tensile strength as a function of the degradation ratio of composites.

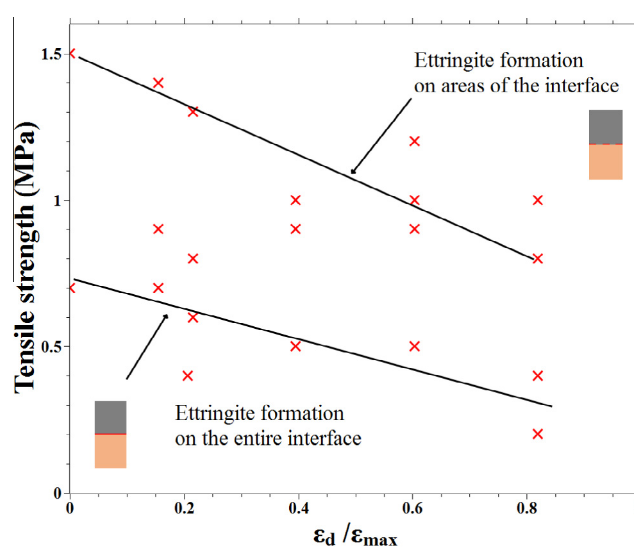


Fig. 21. Evolution of the tensile strength as a function of the degradation ration and assumption of the existence of two zones.

The results of the tensile tests show a dispersion in strength at each given degradation time. Comparison of the sound samples results (Fig. 10) with the degraded samples results (Fig. 20) show a decrease in strength as the degradation progresses. This dispersion can be explained by the non-homogeneous ettringite formation at the interface, observed by SEM for two samples (REV and composite). Despite the scattered results, a drop in strength is observed. At expansion equal to zero, the dispersion of results is also observed, unlike the sound sample, it may be due to the heat treatment at a young age or the absence of expansion despite the formation of ettringite at the interface to occupy the existing vacuum.

According to the observation of the Fig. 20, it can be assumed that there are two zones:

- The first zone would correspond to samples with localized ettringite at the interface and high tensile strength that decreases progressively with the development of pathology.

- A second zone with low resistance, relative to the first, which decreases with the swelling of the sample. This area would correspond to composites with a large amount of ettringite at the interface (Fig. 21).

4. Discussion

An experimental protocol allowing ruling 'more quickly' on a risk of DEF at the cement paste-aggregate interface scale is proposed in this study. The shape of composites samples allowed us to distinguish between the expansion of the cement paste and the expansion of the cement paste-aggregate interface. The main objective of this work is to understand the DEF phenomenon at the local scale (ITZ). The boundary conditions of this chosen composites shape are different from what is observed in concrete at the REV scale. But, this shape allows the swelling of the interface and the cement paste to be separated. which cannot be determine with REV samples.

The main challenge of this work is the performing of the experimental device for the samples preparation and the acceleration of DEF pathology on this scale. The size and shape of the samples make it difficult to prepare and perform heat treatment for DEF acceleration. Much of this work has been devoted to adapt the experimental protocol at the VER scale to composites at the local scale. Preliminary tests were carried out before choosing the acceleration protocol adopted for this study. The results showed that the swelling rate of the interface is greater than that of the cement paste. These results confirmed the observations at the macroscopic scale. Leklou [17] has shown that after 200 days (with an expansion of 0.10%), "expansive" ettringite is easily observed at the cement paste-aggregate interface. On the other hand, no "expansive" ettringite is found in the cement paste. However, after 450 days (expansion of 1.84%), cracks filled with ettringite located inside the cement paste are identified in addition to the ettringite observed around the aggregates.

The SEM observations confirmed the results of the composites swelling. A concentration of ettringite at the cement paste-aggregate interface was observed. The ettringite formation at the interface scale was different for each sample. We could observe samples with ettringite throughout the interface and samples with ettringite only in areas at interface.

The tensile tests showed the DEF effect on the mechanical properties of the cement paste-aggregate interface. The dispersion of the tests results explained by non-repeatability of ettringite formation at the interface observed at two scales. The results showed two zones: The first zone would correspond to the samples with less ettringite (major contact between cement paste and aggregate). In this zone, the strength is close to that of the sound sample and decreases with sample swelling. The second zone would correspond to composites with ettringite throughout the interface. In this case, the initial strength is low compared to the first zone and also decreases during the degradation. To confirm these results, non-destructive visualization techniques such as tomography would be necessary during the pathology progress.

5. Conclusion

The main objective of this work is the understanding of the DEF phenomenon at the local scale (interface between the cement paste and the aggregate). Given the size and shape of the composite samples, the implementation of the experimental protocol for the fabrication and DEF triggering required many preliminary tests (choice of the temperature to be applied, rate of rise in temperature, heating time, ...). This study explains the DEF development by the late crystallization of ettringite at the cement paste-aggregate interface which is characterized by a high porosity due to the concrete wall effect. The composites study showed higher speed expansion at cement paste-aggregate interface than on cement paste. These results based on SEM observations showed the inhomogeneous presence of ettringite around the aggregates. However, crystallization of ettringite at the interface does not lead to the development of high crystallization pressures, since ettringite locally precipitates as parallel needles. The inhomogeneous formation of ettringite at the interface influences the results of the tensile test that mechanically characterizes the interface: a dispersion of the results is observed.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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