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Impact of Internal Sulfate Reaction (ISR) expansion on concrete transfer and containment properties: detection and modification of properties.

Pichelin A.⁽¹⁾, Carcassès M.⁽²⁾, Cassagnabère F.⁽³⁾, Multon S.⁽⁴⁾, Nahas G.⁽⁵⁾

- (1) *Laboratoire Matériaux et Durabilité des Constructions (LMDC), Toulouse, France, pichelin@insa-toulouse.fr*
- (2) *Laboratoire Matériaux et Durabilité des Constructions (LMDC), Toulouse, France, myriam.carcasses@iut-tlse3.fr*
- (3) *Laboratoire Matériaux et Durabilité des Constructions (LMDC), Toulouse, France, cassagna@insa-toulouse.fr*
- (4) *Laboratoire Matériaux et Durabilité des Constructions (LMDC), Toulouse, France, multon@insa-toulouse.fr*
- (5) *Institut de Radioprotection et de Sécurité Nucléaire (IRSN), Fontenay aux Roses, France, georges.nahas@irsn.fr*

Abstract

Internal swelling reaction in concrete can be attributed to two types of chemical reactions: Internal Sulfate Reaction (ISR) and Alkali-Aggregate Reaction (AAR). These reactions cause tensile stresses in the concrete leading to the creation of cracks in the structure.

This study focuses on research of the correlations between the evolution of the different physicochemical and mechanical parameters according to the degree of ISR pathology developed in concrete. In order to detect this pathology, the characterization of physicochemical and mechanical properties was made for six times in accordance to the level of expansion. The first characterization was made at the initial state, two during the latent period, two during the accelerating phase and one at the end of expansion. Numerous properties were performed, only relevant tests were introduced in this paper. Scanning Electronic Microscopy was employed to observe the initiation and the presence of Delayed Ettringite Formation (DEF). Moreover, physical tests linked to transfer properties such as gas permeability and electrical resistivity were performed to detect micro-cracks generated by the DEF advancement. Finally, to observe the impact of micro-cracks on the mechanical properties, compressive strength and dynamic moduli were experimentally determined. Furthermore, the measurement of longitudinal and transversal expansion with time was ensured by micrometers. It was observed that few tests can be used to detect these pathologies earlier, during the latent period. In fact, gas permeability, electrical resistivity and static moduli results were promising. These tests are very sensitive to the development of micro-cracks generated in the concrete when the threshold of tensile strength, due to the development of internal swelling reaction, is reached.

Keywords: Delayed Ettringite Formation (DEF); Sustainability indicators; Internal Sulfate Attack (ISA); Swelling; Sulfate.

1. Introduction

The formation of delayed ettringite (DEF) in cementitious material, after concrete hardening and without external sulfate supply is an Internal Sulfate Attack (ISA). Many engineering structures are susceptible to develop these pathologies. This reaction requires the presence of three main factors: high sulfate and aluminate contents, water, and a rise in temperature during the life of the structures. The reaction mechanisms of DEF are complex but Brunetaud has proposed a global mechanism, described in four phases, grouping many theories [1]. Several parameters have an influence on the development of DEF, and can be divided into two groups: those related to the formulation of concrete and those related to the environment. The main consequences of DEF are expansion and cracks. They affect the physicochemical and mechanical properties of the concrete. Cracks lead to an increase of transfer properties and a decrease of mechanical properties and may lead to a loss of containment, which is harmful for nuclear power plants. Few data can be found in the literature on the evolution of these

properties according to the degree of advancement of DEF. The present study had two objectives: first, to find a sensitive test allowing DEF to be detected before cracking can be observed visually and, second, to evaluate the evolution of the properties that impact the containment and also the sustainability of concrete. A test protocol to accelerate DEF development was carried out and provided a good representation of the reality on site [2]. A large number of samples were used to characterize the impact of DEF on concrete properties at different levels of expansion. Chemical tests were performed to observe the initiation and the presence of DEF, while physical and mechanical tests evaluated the impact of DEF on mechanical and transfer properties.

2. Experimental program

2.1. Concrete

2.1.1. Cement and aggregates

In this study, two cements and two types of aggregates were used. The choice of cements focused on a CEMI and a CEMII, the main characteristics of which are summarized in Table 1. The aggregates used were based on silica or limestone according to the formulations. These two cements have been used in previous studies and show a high swelling potential in the case of DEF, in particular because of their high sulfate, alkali and aluminate contents [2, 3].

Mass content (%)	CEM I 52.5 N	CEMII/A-LL 42.5 R
SiO ₂	19.3	19.7
CaO	63.2	63.5
Al ₂ O ₃	5.3	4.6
Fe ₂ O ₃	2.6	3.2
K ₂ O	0.94	1.36
Na ₂ O	0.08	0.11
SO ₃	3.5	2.7
Total	94.92	95,17

Table 1: Cement composition

2.1.2. Mix designs

The mix designs have already been studied in other experimental works, which make it possible to complete the database on the behavior of these concretes [2, 4]. However, some modifications were made in the nature of aggregates used. The formulations are detailed in Table 2.

(a) CEMII-Si		(b) CEMI-Ca	
Material	Content (kg/m ³)	Material	Content (kg/m ³)
CEMII/A-LL 42.5 R	350	CEM I 52.5 N	400
0/0.315	264	0/4	718
0.315/1	151	4/11.2	289
1/4	254	11.2/22.5	813
4/8	145	Effective water	185
8/12	399		
12/20	616		
Effective water	188		

Table 2: Mix designs of “CEMII-Si” (a) and “CEMI-Ca” (b) concrete

The CEMII-Si and CEMI-Ca concretes contained siliceous and limestone aggregates, respectively, to show the impact of the nature of aggregates on DEF. The concrete casting procedure used in this study followed the protocol of French standards [4, 5].

2.2. Conditioning

IFSTTAR Test N° 66 was adopted, with some modifications [6] to allow rapid development of DEF. The conditioning is in four stages: concrete mixing, heat treatment, dry/wet cycles, and final immersion of samples in water at 20°C. The thermal treatment at young age corresponded to that in Al Shamaa's study [3].

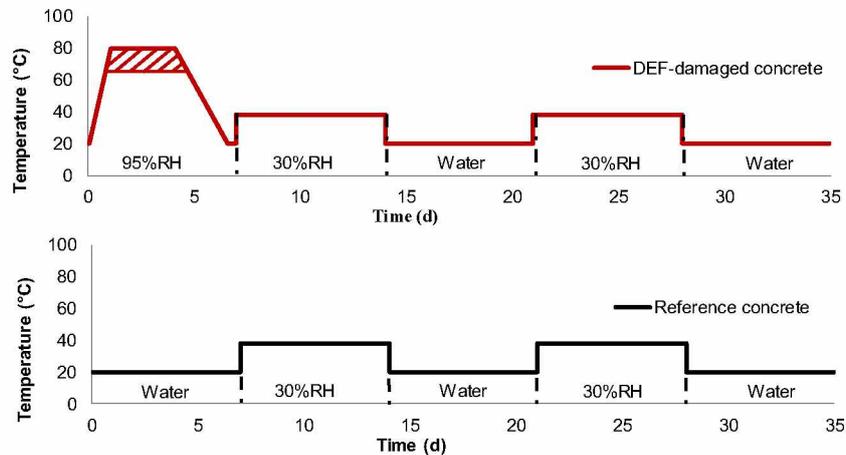


Figure 1: Thermal profile of concretes developing DEF and wet/dry cycles for both concretes

Once cast, specimens were placed in a climate chamber at controlled temperature and relative humidity. The heat treatment was applied for 7 days and comprised four stages: a hold at 20°C, 95% RH for 2h, followed by a rise in temperature from 20°C to 80°C, 95% RH at a rate of 2.5°C/h. Stabilization at 80°C, 95% RH for 72h and a temperature decrease from 80°C to 20°C, 95% RH at a rate of 1°C/h (Figure 1).

The selected dry/wet cycles correspond to the accelerated protocol of IFSTTAR Method N° 66 [6]. They are composed of two 14-day cycles: drying at 38°C, HR < 30% for 7 days and humidification in water at 20°C for 7 days (Figure 1). These cycles allow the initiation of micro-cracking in the concrete and thus accelerate the kinetics of DEF appearance within the concrete without increasing the amplitude of the deformations [7]. The beginning of the degradation is considered to occur at 35 days, at the end of the heat treatment and wet/dry cycles, in this case. During the first 35 days, the concrete is considered to be in a state of hydration where no expansion due to DEF develops. The storage water is not renewed.

When the wet/dry cycles had been completed, the samples were stored in isothermal containers at 20°C, equipped with a pump to homogenize the temperature and the species present in the water. The reference specimens underwent the same elaboration process but were not subjected to the heat treatment. During the first 7 days, the reference specimens were stored in water at 20°C instead of being heated. Wet/dry cycles were also applied (Figure 1).

The mass and expansion monitoring was performed on three cylindrical test pieces (11 x 22 cm). The shrinkage plots were fixed directly on the mold and were therefore embedded in the concrete. Longitudinal expansion was measured by a digital micrometer having an accuracy of $\pm 1 \mu\text{m}$. The transversal expansion was measured by a micrometer having an accuracy of $\pm 5 \mu\text{m}$.

2.3. Protocols

Chemical analyses were performed to ensure the presence of DEF in the concrete. When the swelling was initiated (expansion of 0.04%), measurement of the physical properties revealed the impact of the micro-cracking generated on the transfer properties. Finally, measuring the mechanical properties gave some indications on the state of damage of the concrete subjected to these pathologies. Measurements of all the properties were made at different degrees of expansion.

2.3.1. Physical tests

Two physical tests were performed: electrical resistivity and gas permeability.

The electrical resistivity test was performed according to the standard, on three 11 x 5 cm cylindrical samples obtained from an 11 x 22 cm one [8]. The saturated concrete solution of the tests was replaced by a NaOH concentration of 0.1 mol/l according to a new test protocol (PerfDub).

Gas permeability test was performed according to the standards [9] but the drying temperature was adapted to this study and did not correspond to that proposed in the standard (105°C). It was set at 40°C to avoid destabilizing the delayed ettringite formed in hardened concrete and it allowed the micro-cracking generated during drying to be reduced.

2.3.2. Mechanical tests

Two mechanical properties were measured: the compressive strength and the static moduli. These properties were measured on three 11 x 22 cm cylindrical specimens according to the standards [10, 11].

3. Results

3.1. Expansion

Figure 2 shows the evolution of expansion as a function of time for the two concretes and their corresponding references concrete. IFSTTAR method n° 66 refers to a swelling limit of 0.04% in an expansion test [6]. Beyond this threshold, there is a risk of the concrete being damaged by DEF. The reference formulations did not exceed the limit threshold and showed a maximum swelling of 0.02%.

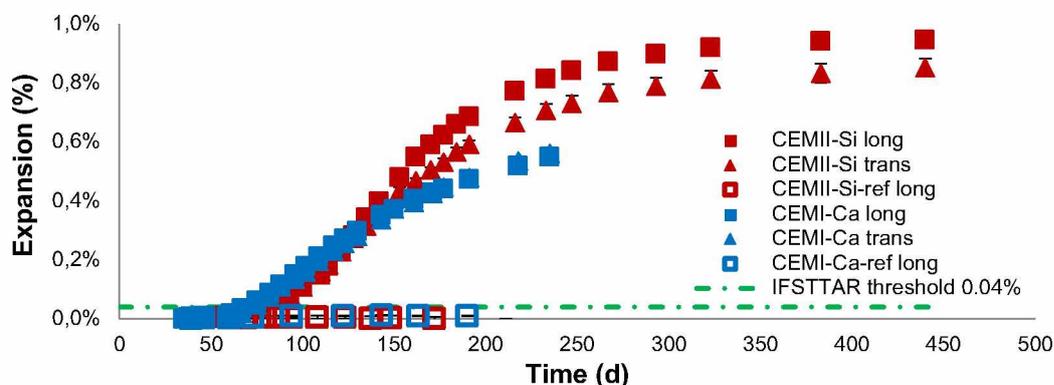


Figure 2: Evolution of expansion (transversal and longitudinal) versus time

Specimens developing DEF showed significant swelling, around 0.9%, at the end of the expansion for CEMII-Si. The latent period began at around 70 days after casting, or 35 days after curing. For the CEMI-Ca design, this period started at 60 days and ended quickly. The first point of inflection was at 80 days for the CEMI-Ca and 110 days for the CEMII-Si concrete. CEMI-Ca contained large limestone aggregates that decreased the kinetics of the latent period [12]. The final expansion of CEMI-Ca showed a swelling of around 0.6%. This lower swelling was attributed to the presence of limestone aggregates, which allowed the bond between aggregate and cement paste to be increased by the formation of calcium hydrates around the aggregates [13, 14, 15]. The cracks generated by DEF at the Interfacial Transition Zone (ITZ) were also reduced, allowing a lower final expansion [15]. The longitudinal and transversal expansion measurements are proportional, confirming the isotropy of the swelling already observed in stress free conditions [16]. However, the transversal swelling is slightly lower on the CEMII-Si concrete at the end of expansion.

3.2. Microscopic observations

The presence of delayed ettringite was observed by SEM for each degree of expansion.

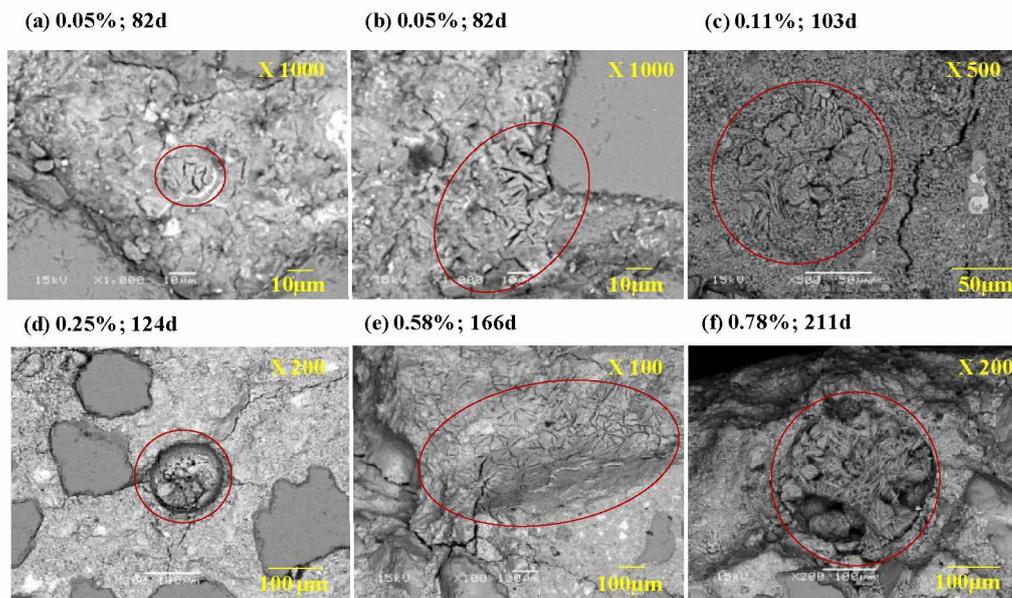


Figure 3: SEM images of the CEMII-Si concrete

Figure 3 shows the presence of delayed ettringite in massive and compressed form for different degrees of swelling. In the CEMII-Si formulation, siliceous aggregates were used. The SEM images show that the initiation of swelling was induced by the formation of delayed ettringite in Hadley grains (Figure 3a) and at the ITZ (Figure 3b). The swelling measured at this degree was 0.05%. As expansion continued, delayed ettringite continued to develop in these areas and in the porosity (Figure 3c and 3d). Then there was a separation from the cement paste all around the aggregates (Figure 3d). At the time corresponding to 0.25% of swelling, the cracks propagated from this interface to the porosity, causing an acceleration of swelling. When expansion continued, the presence of delayed ettringite became increasingly significant, both in the cement paste (Figure 3e) and in the porosity (Figure 3f).

The SEM observations on the CEMI-Ca were consistent with those observed on the CEMII-Si concrete. However, cracking generated at the ITZ in the case of limestone aggregates did not propagate all around the aggregate, thus confirming the observations of Yang et al. [15].

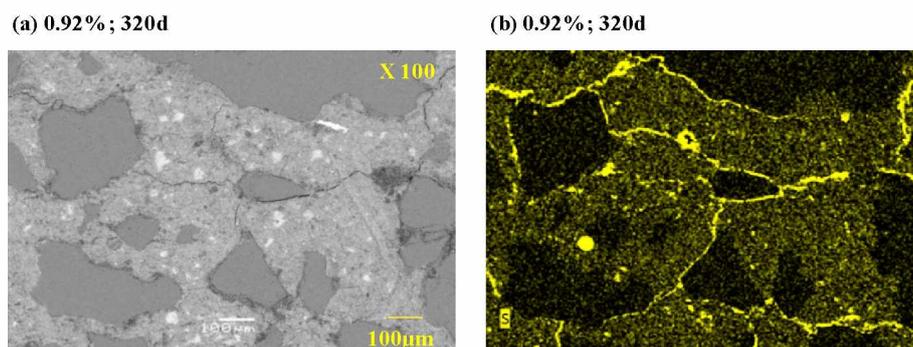


Figure 4: CEMII-Si SEM images (a) initial image and (b) sulfur image (mapping)

For each formulation, the presence of delayed ettringite is found first at the ITZ in the Hadley grains and in the porosity. Ettringite then propagates in and between these preferential zones, generating more and more cracking. This cracking marks the acceleration of the expansion. As mentioned above, the cracking generated at the ITZ depends on the nature of the aggregates [15]. In the case of siliceous aggregates, cracking is generated all around the aggregate but it only partially surrounds limestone aggregates. Ettringite then spreads in existing fissures. Figure 4b shows the presence of sulfur in the sample (yellow in Figure 4b), proving the presence of delayed ettringite in concrete cracks. These observations confirm that the cracks caused by the DEF are filled with ettringite during the expansion. These results are in agreement with the theory of swelling proposed by Brunetaud [1].

3.3. Detection

One of the first aims of this experimental program was to find a sensitive test allowing DEF detection. DEF can be detected if the evolution of one of the properties measured in this work is significant (sufficiently higher than the scatter on results) and only due to DEF expansion (not to concrete hydration). In this aim, a DEF detection criterion for physicomechanical properties was developed, taking the effect of hydration and experimental scatter into account (equation 1):

$$X_{i\ threshold}(t) = X_0 \left(1 + \frac{X_{i\ ref}(t) - X_{0\ ref}}{X_{0\ ref}} \right) \pm 2\overline{\sigma}_{ref} \quad (1)$$

with $X_{i\ threshold}(t)$ the threshold value of detection of the property measured in time, X_0 the initial value of the pathological concrete, $X_{0\ ref}$ the initial value of the reference concrete, $X_{i\ ref}(t)$ the value of the property of the reference concrete in time and $\overline{\sigma}_{ref}$ the average of the standard deviations obtained on the reference specimens.

3.4. Physical measurements

The electric resistivity tests (Figure 5a) showed a great sensitivity. Reference concretes CEMII-Si-ref and CEMI-Ca-ref had a resistivity of 45 $\Omega\cdot m$ at 35 days. The concretes developing the pathology had a lower resistivity, around 35 $\Omega\cdot m$, at the same time, the heat treatment having caused additional damage.

In Figure 5a, a progressive decrease in the resistivity is observed during the damage. A threshold of around 25 $\Omega\cdot m$ is reached at 0.3% of expansion. This threshold of resistivity could be due to the cracking caused by the pathology being gradually filled by ettringite. In contrast, for the reference concretes, the electrical resistivity increased significantly between 35 and 90 days. A resistivity gain of 25% was observed from day 35 to day 180, due to the evolution of the hydration reactions.

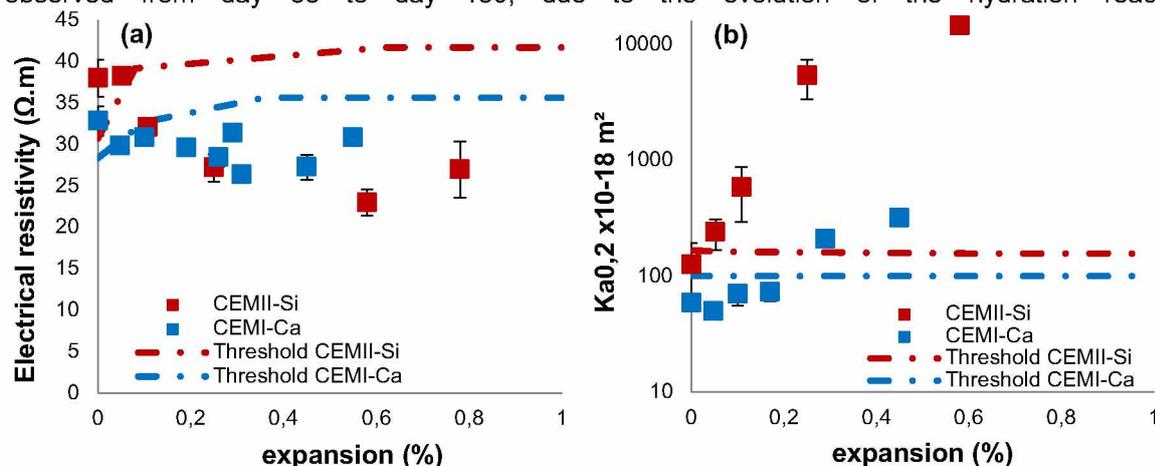


Figure 5: Electrical resistivity (a) and apparent gas permeability (b) according to the expansion

With the chosen criterion, the detection of DEF by the electrical resistivity took place between 0.05 and 0.1% of expansion, during the latent period. Between these two expansion levels, the decrease of resistivity was sufficient to point out significant damage.

Figure 5b shows the evolution of the apparent air permeability coefficient according to the expansion generated by DEF. The reference concretes CEMII-Si-ref and CEMI-Ca-ref show no change in their coefficients of permeability over time of $110 \times 10^{-18} m^2$.

The CEMII-Si and CEMI-Ca concretes have similar apparent gas permeability, respectively equal to $125 \times 10^{-18} m^2$ and $60 \times 10^{-18} m^2$ before expansion. During expansion, the CEMII-Si concrete shows a strong increase in the coefficient of permeability at the beginning of expansion (permeability at 0.05% is double that before expansion). The CEMI-Ca also shows an increase but it occurs later and is smaller. This difference could be due to the mineralogical nature of the aggregates used. The ITZ of concretes containing limestone aggregates seems to be improved [12, 13, 15]. Cracks due to DEF passing through this interface are reduced in the case of limestone aggregates [15]. There may be fewer preferential paths in the CEMI-Ca concrete, causing a smaller increase of permeability at early expansion. When the acceleration phase of the pathology is reached, at around 0.2% of swelling, the permeability is significantly impacted. This drastic increase can be attributed to the formation of more and more

percolating paths within the material, generating a large increase of gas flow during the test. These results are therefore in agreement with those of Al Shamaa et al. [17].

Depending on the criterion chosen, the detection of DEF by the measurement of gas permeability takes place during the latent period before 0.05% expansion for CEMII-Si concrete and at the beginning of the acceleration phase, between 0.17 and 0.29%, for CEMI-Ca concrete.

3.5. Mechanical properties

The compressive strength measurements show the impact of the heat treatment on the mechanical properties. The reference concretes CEMII-Si-ref and CEMI-Ca-ref have compressive strengths of 34 MPa and 44 MPa, respectively, at 35 days.

The compressive strengths of CEMII-Si and CEMI-Ca are lower, at 23 MPa and 29 MPa, respectively. The reduction of the compressive strength by the heat treatment can be explained by the occurrence of damage in concrete and by the modification of hydrates [12, 18, 19]. The compressive strength of CEMI-Ca and CEMI-Ca-ref concretes remains higher than those of other concretes and increases with time following the hydration process. This difference is explained by the fact that these concretes contain a larger amount of cement and a lower W/C ratio, and also because the presence of limestone aggregates in the formulation improves the ITZ. The CEMII-Si and CEMI-Ca show compressive strength that remains stable during the initiation of the pathology. However, during the acceleration phase, the strength decreases spontaneously and then stabilizes at the end of the acceleration period (Figure 6a). The compressive strength drop is 30% and 20% for the CEMII-Si and CEMI-Ca concretes, respectively, at the middle of expansion.

Depending on the criterion chosen, the detection of DEF by the measurement of compressive strength takes place between 0.17 and 0.25% of expansion, at the beginning of the acceleration phase.

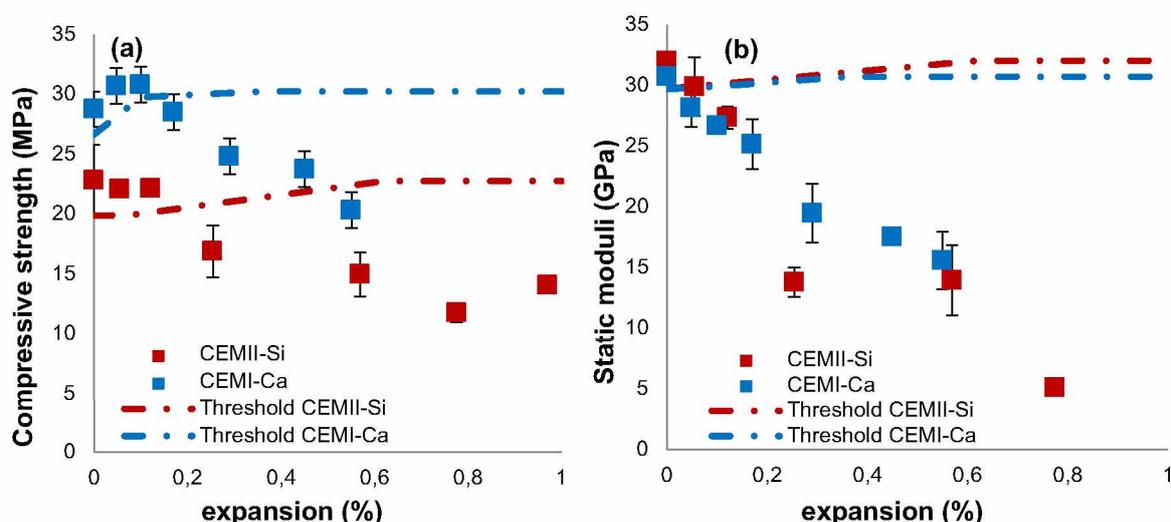


Figure 6: Compressive strength (a) and static moduli (b) according to the expansion

The static moduli of CEMII-Si-ref and CEMI-Ca-ref reference concretes shows small changes between 35 and 190 days. The moduli is still higher for CEMII-Si-ref concrete (equal to 40 GPa) than for CEMI-Ca-ref concrete (31 GPa) at 35 days.

CEMII-Si and CEMI-Ca concretes with DEF reveal that the moduli decreases progressively during the expansion. A significant decrease in the moduli is observed for swelling of around 0.2% during the acceleration phase of the pathology (Figure 6b). At this time, the moduli decreases by 40% and 50% for the CEMI-Ca and CEMII-Ca-ref concretes, respectively. The loss of static moduli seems to be proportional to the expansion rate caused by the pathology. These results are not in agreement with those of Al Shamaa et al., who show no decrease of static moduli during the latent period [20].

Depending on the criterion chosen, the detection of DEF by the measurement of static moduli takes place between 0.05 and 0.1% of expansion, during the latent period.

4. Discussion

Only particular tests make it possible to detect DEF at the beginning of the expansion, during the latent phase. Table 3 summarizes all the expansion values at the time of detection of pathologies presented in the experimental results.

	Expansion at detection (%)		DEF phase
	CEMII-Si	CEMI-Ca	
Electrical resistivity	0.05-0.11	0.05-0.11	Latent phase
Gas permeability	0.05	0.17-0.29	Latent phase
Static moduli	0.05 / 0.11	0.05-0.11	Latent phase
Compressive strength	0.11 / 0.25	0.17-0.29	Acceleration phase

Table 3: Summary of the evolution of the properties at detection for the CEMII-Si and CEMI-Ca concretes

Microscopic observations showed the evolution of ettringite formation during expansion in the material. Some physical measurements related to the transfer properties allowed DEF to be detected during the latent phase and measurements of the gas permeability and electrical resistivity appear to provide a good sustainability indicator for the detection of DEF. CEMI-Ca concrete showed a later increase in permeability than CEMII-Si. The improvement of ITZ in the case of limestone aggregates seems to reduce the cracks generated at this interface during DEF damage [15]. Thus allowing the cracks in the concrete to be less percolating.

The electrical resistivity tests showed significant sensitivity, the detection being effective from the latent phase. The electrical resistivity decreased by 16% and 10% for the CEMII-Si and CEMI-Ca concretes, respectively, for expansions of 0.12% and 0.19%. Since the electrical resistivity of the reference concretes increased substantially from 35 days to 180 days, the decrease in the resistivity associated with the development of the pathology seems to be significant. However, its sensitivity to water content could make electrical resistivity difficult to use for damaged structures [21].

Static moduli measurements also showed significant sensitivity. A continuous decrease was observed throughout the development of the pathology. A 15% loss of moduli associated with a swelling of 0.11% was observed for each concrete. The compressive strength of the CEMII-Si and CEMI-Ca concretes damaged by DEF decreased by 26% and 14% respectively for expansions of 0.25% and 0.29%. This loss of compressive strength in the middle phase of acceleration delayed the detection of the pathology. Three sustainability indicators made it possible to detect DEF earlier, during the latent phase for laboratory specimens, the gas permeability, the electrical resistivity and the static moduli.

Figure 7a shows the evolution of the damage as a function of the expansion for the two concretes damage by DEF. The damage was proportional to the expansion due to DEF. The damage was calculated from the static moduli according to Equation 2:

$$D = 1 - \frac{E_i}{E_0} \quad (2)$$

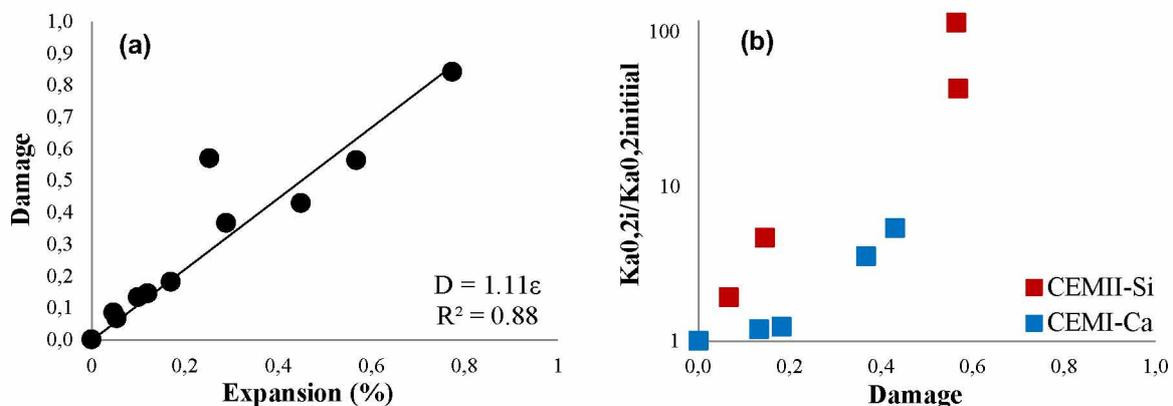


Figure 7: Evolution of the damage as a function of expansion (a) and of the apparent permeability rate as a function of damage (b)

A study by Martin et al. on coupled AAR and DEF expansion on concrete shows an almost linear trend. The loss of damage according to the expansion is in accordance with these results [22].

The increase in permeability seems to be an exponential function of the damage. The percolating paths in CEMII-Ca concrete specimens would appear at around 20% of damage, at the beginning of the acceleration phase of the pathology. For the CEMII-Si concrete, the percolating paths appear for slight damage, around 7%, causing a considerable increase of the air flow through the material. The greater bonding at the ITZ in the case of limestone aggregates seems to allow the containment properties to be maintained during the beginning of the DEF damage, when it is lower than 20%.

Table 4 shows the evolution of the potential durability of concretes during the development of the pathology. The “low” classification at the initial state of concretes is mainly due to the effects of heat treatment and wet/dry cycles as mentioned previously [12, 18, 19].

Sustainability indicators	Concretes	Initial state	Acceleration phase
Expansion (%)	CEMII-Si	0	0.15-0.7
	CEMI-Ca	0	0.1-0.45
Electrical resistivity	CEMII-Si	Very low	Very low
	CEMI-Ca	Very low	Very low
Gas permeability (40°C)	CEMII-Si	Medium	Very low
	CEMI-Ca	High	Medium

Table 4: Classification of concrete damaged by DEF relative to limit values for the potential sustainability and properties of concrete [23]

During the acceleration phase of the pathology, the potential sustainability of concretes becomes greatly affected. This could accelerate the kinetics of appearance of other pathologies such as AAR, carbonation and corrosion, causing premature aging of the structures.

5. Conclusion

An experimental program has been realized to obtain a sensitive measurement of DEF. The second objective was to evaluate the evolution of the properties that have an impact on the containment of concrete. Several physicochemical and mechanical properties were measured by the use of a large quantity of samples. These tests were performed on two concretes showing a high swelling potential. Original results have been obtained:

- Electrical resistivity and gas permeability are sensitive to the expansion generated by DEF. However, the electrical resistivity could be difficult to use for damaged structures [21].
- For a same expansion, the permeability increase is greater for concrete containing silica aggregate than for concrete with limestone aggregate.
- Only the measurement of the static moduli allows detection of DEF expansion during the latent period.
- Three sustainability indicators seem to allow the detection of pathology in the short term during the latent period: gas permeability, electrical resistivity, and static moduli.
- The loss of containment related to the presence of DEF depends of the nature of the aggregates and occurs for less than 20% of damage.

This study was performed in stress free swelling and fully saturated conditions. It is therefore necessary to pursue the study to evaluate the influence of the presence of reinforcement and in case of moisture gradient. In the case of nuclear power plants, it is necessary to develop nondestructive methods to detect DEF in order to maintain the containment property of concrete. In this context, a test platform for the measurement of DEF on concrete structure is being developed in the ODOBA (Observatoire de la Durabilité des Ouvrages en Béton Armé) project.

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References

- [1] Brunetaud X., 2005. Etude de l'influence de différents paramètres et de leurs interactions sur la cinétique et l'amplitude de la réaction sulfatique interne au béton. PhD thesis, Ecole Centrale de Paris.
- [2] Jabbour J., 2018. Méthodes d'essais de vieillissement accéléré des bétons à l'échelle des ouvrages. PhD thesis, Université Paris-Saclay.
- [3] Al Shamaa M., 2012. Etude du risque de développement d'une réaction sulfatique interne et de ses conséquences dans les bétons de structure des ouvrages nucléaires. PhD thesis, Université Paris-Est.
- [4] NF P18-404, 1981. Bétons - Essais d'étude, de convenue et de contrôle - Confection et conservation des éprouvettes.
- [5] NF P18-400, 1981. Bétons - Moules pour éprouvettes cylindriques et prismatiques.
- [6] Méthode d'essai n°66, 2007. Réactivité d'un béton vis-à-vis d'une réaction sulfatique interne. Laboratoire Central des Ponts et Chaussées.
- [7] Leklou N., Aubert J-E., Escadeillas G., 2013. Influence of various parameters on heat-induced internal sulphate attack. *European journal of environmental and civil engineering*. Vol. 17:3, p. 141-153. <https://doi.org/10.1080/19648189.2012.755338>.
- [8] PerfDuB, 2017. Determination of the resistivity of saturated concrete.
- [9] XP P18-463, 2011. Bétons - Essai de perméabilité aux gaz sur béton durci.
- [10] NF EN 12390-3, 2003. Essais pour béton durci - Partie 3: résistance à la compression des éprouvettes.
- [11] NF EN 12390-13, 2014. Essai pour béton durci - Partie 13: détermination du module sécant d'élasticité en compression.
- [12] Al Shamaa M., Lavaud S., Divet L., Colliat J.-B., Nahas G., Torrenti J.-M., 2016. Influence of limestone filler and of the size of the aggregates on DEF. *Cement and Concrete Composites*. Vol 71, p. 175-180. <https://doi.org/10.1016/j.cemconcomp.2016.05.007>
- [13] Grattan-Bellew P.E., Beaudoin J.J., Vallée V.G., 1998. Effect of aggregate particle size and composition on expansion of mortar bars due to delayed ettringite formation. *Cement and Concrete Research*. vol. 28, n° 8, p. 1147-1156. [https://doi.org/10.1016/S0008-8846\(98\)00084-2](https://doi.org/10.1016/S0008-8846(98)00084-2).
- [14] Heinz D., Kalde M., Ludwig U., Ruediger I., 1999. Present state of investigation on damaging late ettringite formation (DLEF) in mortars and concretes. *ACI International*. Vol. 177-1, p. 1-14.
- [15] Yang R., Lawrence C. D., Sharp J.H., 1999. Effect of type of aggregate on delayed ettringite formation. *Advances in Cement Research*. Vol. 11, n°3, p. 119-132. <https://doi.org/10.1680/adcr.1999.11.3.119>.
- [16] Bouzabata H., Multon S., Sellier A., Houari H., 2012. Effects of restraint on expansion due to delayed ettringite formation. *Cement and Concrete Research*. Vol. 42, p. 1024–1031. <https://doi.org/10.1016/j.cemconres.2012.04.001>.
- [17] Gallucci. E., Zhang. X. and Scrivener. K. L., (2013). Effect of temperature on the microstructure of calcium silicate hydrate (C-S-H). *Cement and Concrete Research*. Vol. 53, p. 185-195. <https://doi.org/10.1016/j.cemconres.2013.06.008>.
- [18] Cagnon. H., Vidal. T., Sellier. A., Bourbon. X. and Camps, G., 2015. Drying creep in cyclic humidity conditions. *Cement and Concrete Research*. vol. 76, p. 91-97. <https://doi.org/10.1016/j.cemconres.2015.05.015>.
- [19] Al Shamaa M., Lavaud S., Divet L., Nahas G., Torrenti J.-M., 2015. Influence of relative humidity on delayed ettringite formation. *Cement and Concrete Composites*. Vol. 58, p. 14-22. <https://doi.org/10.1016/j.cemconcomp.2014.12.013>.
- [20] Al Shamaa M., Lavaud S., Divet L., Nahas G., Torrenti J.-M., 2014. Coupling between mechanical and transfer properties and expansion due to DEF in a concrete of a nuclear power plant. *Nuclear Engineering and Design*. Vol. 266, p. 70-77. <https://doi.org/10.1016/j.nucengdes.2013.10.014>.

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Pichelin A.; Carcassès M.; Cassagnabère F.; Multon S. ; Nahas G.

[21] Rivard P., Saint-Pierre F., 2009. Assessing alkali-silica reaction damage to concrete with non-destructive methods: From the lab to the field. *Construction and Building Materials*. Vol. 23, p. 902–909. <https://doi.org/10.1016/j.conbuildmat.2008.04.013>.

[22] Martin R.-P., Sanchez L., Fournier B., Toutlemonde F., 2017. Evaluation of different techniques for the diagnosis & prognosis of Internal Swelling Reaction (ISR) mechanisms in concrete. *Construction and Building Materials*. Vol. 156, p. 956–964. <https://doi.org/10.1016/j.conbuildmat.2017.09.047>.

[23] Baroghel-Bouny V. et al., 2004. Conception des bétons pour une durée de vie donnée des ouvrages. Maîtrise de la durabilité vis-à-vis de la corrosion des armatures et de l'alcali-réaction. Etat de l'art et guide pour la mise en œuvre d'une approche performantielle et prédictive sur la base d'indicateurs de durabilité. Guide AFGC.