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New procedure to investigate external sulphate attack on cementitious materials

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ABSTRACT

In order to predict the durability of concrete structures in contact with sulphate rich environments, laboratory tests have to be sufficiently representative of the field conditions. This study is focused on the degradation of cement-based materials submitted to external sulphate attack. To be close to the natural degradation process, a new experimental procedure has been developed on the basis of two studies. The first was conducted on concrete and showed the deficiencies of common experimental programs only based on the measurement of the expansion. The second investigated the relation between the specimen size and the resistance to sulphate attack. As a result, the basic points of a representative procedure were identified. The mass and length of the specimens were monitored. The pH of the sodium sulphate solution was controlled, which allowed the assessment of leaching kinetics.

This procedure was then validated by analysing, at macroscopic and microscopic scales, the influence of the water sulphate concentration on the behaviour of cementitious materials. The accelerating effect of high sulphate concentrations is due to the increase in leaching kinetics rather than the type of expansive products. Whatever the concentration, the same critical proportion of the cross section is required to generate global swelling.

1. Introduction

External sulphate attack (ESA), which could be defined as a reaction between sulphate ions and hydration products of cement causing damages, is a real threat for concrete durability in an aggressive environment. Sulphate ions are present in ground water, sea water, soils and waste waters [1,2]. The reaction mentioned above leads to formation of gypsum, ettringite and/or thaumasite and may cause cracking, spalling, softening, expansion, loss of strength and other forms of damage [3]. Physical and chemical factors, such as the pH of the solution and its concentration, the associated cation, the binder type, the water-to-binder ratio and the curing conditions, can influence the ESA.

To investigate the effect of ESA, tests have been developed in order to correlate the risk of expansion (time of initiation or expansion at a given time) and the composition of the cementitious materials, namely: water-to-cement ratio (W/C), cement content, compound composition of cement, type and proportion of mineral additions. The resulting correlations can be found in prescriptive requirements on cement [4,5] and concrete [6] or they can be deduced from performance-based tests [7–9].

Currently, the effect of ESA is assessed through several indicators: length variations, loss or increase of mass, strength and elastic modulus decrease. In most of the studies, two stages are identified from length measurements: The first stage is a dormant period where no or little expansion can be measured, and then significant expansion occurs. More information is needed to explain the phenomena occurring during the first stage of sulphate attack. However, the expansion data do not provide enough information to assess the chemical reactions and understand the mechanisms causing damage. Models based on expansion and visible damage depths are not reliable to predict the long term performance of concrete although these two parameters are the most common to evaluate the attack [10,11]. The time for a global expansion of the specimens caused by local formation of gypsum and ettringite may exceed several years [12,13]. A well-known way to accelerate the test is to increase the sulphate concentration. As a consequence, tests [14,15] are often made at relatively high concentrations (more than 20 g/L) whereas concentrations are lower in natural exposures which could interfere with the obtained results. The sulphate concentration of seawater is about 2800 mg/L [16]; and chemically aggressive environments refer to concentrations between 600 and 6000 mg/L [6], or between 150 and 10,000 mg/L [17,18]. Moreover, some of the tests are led at constant pH while others not, whereas pH can be assumed to be constant in natural exposures. Studies have shown that the response to ESA could be different according to the pH of the solution [19]. Furthermore, the measurements are generally done at a macroscopic scale whereas the disorders take place in the microstructure at the scale of the cement paste phases. These phenomena are not

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identified with the common measurements methods. Detailed analyses at the microscopic scale correlated with traditional measurements at the macroscopic scale are necessary.

As a consequence, there is a need to develop an experimental procedure sufficiently representative of the degradation process in field conditions and to define a relevant scale for the study in order to monitor the degradation at the same scale as the disorders.

The experimental program, presented in this study, has been designed in order to provide data to establish a representative test or method to understand the mechanisms of external sulphate attack on cementitious materials. Two experimental studies have been conducted. Firstly, the base point of the program has consisted in a pilot study on several concrete mixtures exposed to sodium sulphate at constant pH. The tests were made at two sulphate concentrations: 3 g/L and 30 g/L. Mass and length measurements were analysed. Secondly, concrete samples have been replaced by mortar samples with different volume-to-surface ratios to choose the appropriate specimen size. In the light of these two studies, a new experimental procedure has been designed. Tests were led at constant pH in controlled conditions. Hence, clarifications about the dormant period (first stage) during the degradation process could be made when correlating mass and length measurements with kinetics of leached calcium and hydroxide ions. The last part of this investigation shows the relevance of the new method to understand the influence of sulphate concentration on the degradation kinetics and mechanisms using chemical analysis and microscopic observations.

2. Experimental program

2.1. Pilot study

Three different cements have been used in the experimental tests:

- Sulphate-resisting Portland Cement (low C_3A content: 2%).
- Normal Portland Cement (1) (C_3A content: 7.6%).
- Normal Portland Cement (2) (C_3A content: 11.4%).

Their compositions are given in Table 1.

Four concrete mixtures were designed (Table 2). Two concrete mixtures C2 and C3 comply with two levels of prescriptive requirements (XA2 and XA3 exposure classes) from standard NF EN 206-1 [6] for exposure to ESA, in terms of minimum Equivalent Binder content (Eq. Binder) and maximum W_e/Eq. Binder[1] ratio. The binder of C3 concrete mixture is not sulphate-resisting, because its C_3A content is higher than 5%. C1 concrete mixture was made of the same cement as C3 and has a higher W_e/Eq. Binder ratio. Such mixture often lead to poor resistance to sulphate attack [20], but C1 was designed to investigate the sensitivity of the test. C1B derived from the C1 composition, by replacing the cement (1) by the cement (2) with higher C_3A content.

Table 1
Cement composition.

	Sulphate-resisting Portland cement	Normal Portland Cement (1)	Normal Portland Cement (2)
Chemical analysis (%)			
CaO	64.95	64.10	65.4
SiO_2	21.25	20.40	20.43
Al_2O_3	3.47	4.70	4.9
Fe_2O_3	4.23	2.95	1.83
SO_3	2.65	3.60	3.6
Compound composition of clinker (%)			
C_3S	73.5	63.6	66.4
C_2S	5.5	13.4	5.6
C_3A	2.0	7.6	11.4
C_4AF	12.9	9.4	5.4
Physical properties			
Blaine fineness (cm^2/g)	3650	4130	3750
Normal comp. strength (MPa)	62.6	65.5	69.1

Table 2
Concrete mixture compositions.

Exposure classes (kg/m^3)	X0	X0	XA2	XA3
	C1	C1B	C2	C3
Siliceous gravels				
12, 5/20	365	365	374	374
8/12	268	268	275	275
4/8	410	410	421	421
Siliceous sand				
0/4	741	741	760	760
Cement (C)				
Sulphate resisting Portland Cement	–		352	–
Normal Portland Cement (1)	280		–	385
Normal Portland Cement (2)	–	280	–	–
Effective water (W_e)	205	205	174	154
Superplasticizer	0	0	0.73	3.16
W_e/C	0.73	0.73	0.49	0.4
Volume of paste Vp (L/m^3)	296	296	286	278

$7 \times 7 \times 28\ cm^3$ concrete specimens were sealed cured until they reach 50% of required compressive strength (NF EN 206-1) for the corresponding exposure class (X0, XA2, XA3) and stored at 20 °C and 50% of relative humidity (RH). After 28 days, they were immersed in two sodium sulphate solutions at 3 g/L and 30 g/L of SO_4^{2-}. The pH of the bath is controlled [21].

2.2. New experimental procedure

2.2.1. Material and mixture characteristics

Mortar specimens were made with the Normal Portland Cement (2) and siliceous sand with maximum particle size of 2 mm. Siliceous sand and gravels were used to avoid the thaumasite formation because it was shown in previous studies [15,22] that the presence of limestone aggregates can promote its formation due to calcium carbonate. The W/C ratio was 0.60. The mortar composition is given in Table 3.

In order to study the size effect; mortar specimens were cast as $1 \times 1 \times 10$; $2 \times 2 \times 16$; $4 \times 4 \times 16$ and $7 \times 7 \times 28\ cm^3$ prisms. They were cured at 100% of RH for 24 h; then they were demolded and stored in limewater for 27 days. After this phase, the specimens were submerged in a sodium sulphate solution (3 g/L of SO_4^{2-}) with controlled pH. In parallel, control specimens were stored in tap water. Mass and expansion were measured periodically in order to follow the effect of the sulphate ingress.

In the study on concentration, specimens were cast as $2 \times 2 \times 16\ cm^3$ prisms and cured at 100% of RH for 24 h then they were demolded and stored after in limewater for 14 additional days. At day 15, specimens were immersed into three different sodium sulphate solutions $\left(SO_4^{2-}\right)$ at different concentrations (3 g/L; 10 g/L and 30 g/L of SO_4^{2-}). Two control specimens were stored in tap water.

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[1] W_e refers to effective water.

Table 3
Mortar mixture.

	Proportions (kg/m^3)
Siliceous sand 0/2 mm	1409
Normal Portland Cement (2)	495
Water	301
W/C	0.60
Volume of paste Vp (L/m^3)	460

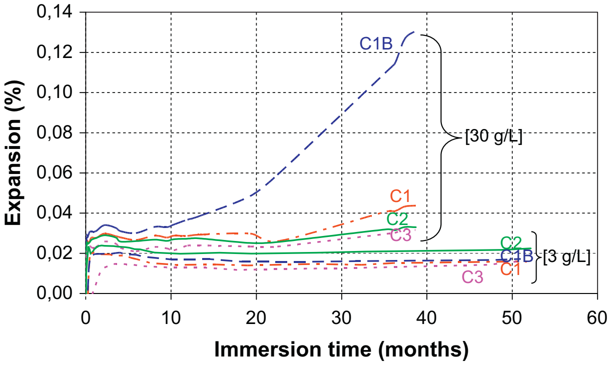


Fig. 2. Expansion of immersed concrete specimens.

2.2.2. Experimental setup

Tests have been realized in controlled conditions at constant pH and temperature, 7.5 and 20 °C respectively. This test procedure is adopted because if the pH is not controlled, after a short immersion period of samples, the pH of the solution is raised to 12 or above [19]. The pH was regulated by adding a nitric acid solution at 0.5 mol/L. The 1.8-L solutions were renewed every 15 mL of added acid. Hence, constant concentration of sulphates could be ensured during the test. This is meant to be representative of field conditions where concrete is exposed to continual supply of sulphate ions [23–25].

The device used for this test is presented in Fig. 1.

Mass and length were measured each time the solution was renewed. The prisms had gauge studs at both ends in order to measure expansion with length change comparators, with an Invar cylinder as a reference length to zero the comparator. The mass of the samples was measured using a digital balance (×0.01 g). Before each mass and length measurement, the samples were wiped to remove the water from the sample surface. The samples remained saturated with dry surface.

In parallel, the quantity of leached hydroxide ions was assessed through the added volumes of nitric acid HNO_3 solution and that of leached calcium by the titration of the solutions.

Microscopic analysis were realised beside these measurements (Scanning Electron Microscopy (SEM) observations and energy dispersive spectroscopy analysis). The specimens used for SEM were embedded in an epoxy layer then cut to expose the cross-section of the specimen. Polishing was then performed using abrasive polishing discs of different grit in order to have a flat polished surface.

3. Results and discussion

3.1. Pilot study on concrete

3.1.1. Mass and length measurements

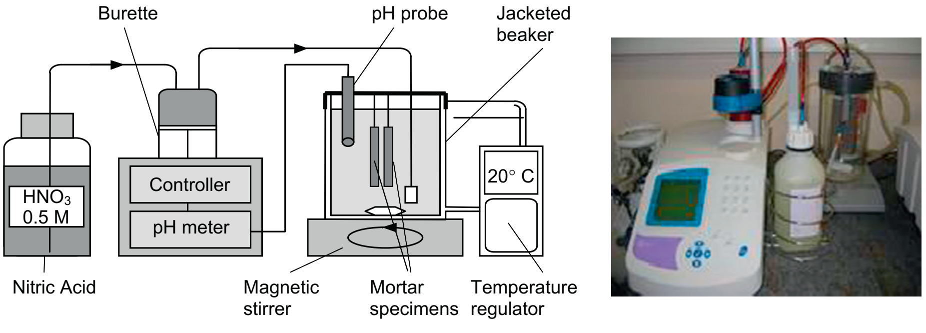
Length measurements of concrete samples are presented on Fig. 2. Whatever the concentration was, an expansion was detected after immersion. This is due to absorption of water and depends on the magnitude of drying shrinkage before the test. After this length increase, the sulphate exposure did not induce a global expansion except for the C1B composition exposed to 30 g/L of SO_4^{2-} for which swelling was detected after 1 year of exposure due to the relatively high C_3A content and W/C ratio.

Referring C2 exposed to 3 g/L, the difference between the deformation of the specimen exposed to sulphate attack and the control was less than 0.01% after 50 months of immersion, which does not represent a significant expansion. These results show why relatively high sulphate concentrations are often preferred for testing: a significant expansion appears earlier.

It should be noted that the expansion is likely to correspond to localized swelling on some specimens due to the cracks that appear on the ends of the specimens around the studs. Hence, the length measurements were affected by these cracks and may not reflect a global swelling of the entire specimen.

The remaining concrete mixtures will probably behave similar to C1B but shifted in time, as these compositions are potentially more resistant to sulphates. A study made by Kosbab and Kurtis [13], based on an investigation conducted over a 40 years period, actually showed that for different concrete mixtures exposed to a sodium sulphate solution at 21.3 g/L, little or no expansion was measured in the first 5 to 10 years.

The length monitoring was not sufficient. Further information is actually needed for a better understanding of the attack. The monitoring of the concrete specimens included mass measurements. Control specimens stored in tap water showed a slight mass increase, which corresponds to hydration, whereas specimens stored in sulphate solution showed much lower increase or slight loss of mass. In order to separate the effect of sulphate attack from the phenomena of absorption and hydration, differences of relative mass variations are plotted against square root of time in Fig. 3. This figure shows that leaching is prevalent in the degradation pro-



(a) (b)

Fig. 1. Schematic diagram (a) and device (b) used for sulphate test.

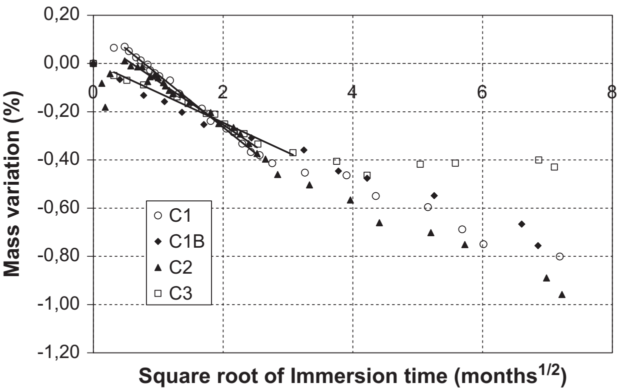


Fig. 3. Variations of relative mass referring to the last measurement before immersion (3 g/L): difference between specimens exposed to sulphates and the controls.

cess and overrides the penetration of sulphates during the first stage. The mass varied linearly as a function of the square root of time up to approximately 260 days what suggests that the phenomenon is diffusive. The mass of the specimens C1 and C2 continued to decrease with time (over a three years period). This loss of mass is the result of leaching and spalling. The decrease of curve slopes for the other compositions is probably due to an artefact in the experimental procedure since the solution is renewed less frequently. Leaching actually generated a loss of mass while the sulphate penetration caused mass increase.

3.1.2. Deficiencies of expansion-based studies

On the one hand, the four-year study on concrete confirmed that the duration of the first stage, when no significant expansion can be measured, may exceed several years. Thus expansion-based tests did not make it possible to understand the degradation mechanism and subsequently to assess the potential durability of the concrete mixtures. On the other hand, this study highlighted that sulphates penetrated mainly by diffusion [26]. That is why it is useful to keep the gradient of concentrations constant and quantify the leaching of calcium and hydroxide ions from the samples to the surrounding solution: a point of improvement of this test that should be taken into account. Mass measurements appeared to be a reliable indicator of resistance to sulphates.

Inaccurate values of length measurements might be registered because of the cracks that could be observed around gauge studs. In order to avoid this artefact and protect the studs, a resin layer was applied to both ends of the specimens and to the first 5 mm of the other sides, in the following studies. This type of protection prevented sulphate solution from penetrating by the ends. If the ends of the specimens are not coated, sulphate ions penetrate from multiple directions and the measured expansion is more affected by the ends than the mid-length portions of the bar as shown in the previous study. This protection helps to get more representative and reproducible results for the length change [27,28].

The following experiments were made at constant pH on mortars; because studies on paste may not be representative of performance of concrete, and studies on concrete take several years [12,21]. The behaviour of mortar is relevant to evaluate the binder. It takes into account the effect of aggregates without the disadvantage of the study on concrete. Another way to reduce the test duration without affecting the mechanism of degradation is to change the scale because the phenomenon is diffusive. As a consequence; the second investigation (§ 3.2) was led on different sizes of mortar specimens in order to select the appropriate scale. Then the developed experimental procedure was used to understand the accelerating effect of increasing the sulphate concentration (§ 3.3).

3.2. Size effect

The length variations are presented on Fig. 4. Independently of the specimen size, two phases were observed (separated by a vertical line on the expansion curve of the 4 × 4 × 16 cm^3 specimens as an example): a latency period when the length did not vary considerably, followed by an expansion period. Depending on the sample size, cracking and expansion started at different times but the shapes of the curves were similar for all the specimens. The smaller samples were the first to show expansion and were followed by the others in the order of increasing sizes.

The time of initiation of global expansion defined on the curve of expansion is plotted against Volume/Surface (V/S) ratio in Fig. 5.

The time of initiation of expansion increased with the specimen size. This behaviour could be explained as follow. To generate an expansion of the cores of the specimens, a minimum proportion of the section must be affected by the formation of expansive products. For small samples, this surface is reached quickly. While for larger samples, the minimum degraded area, necessary to generate global swelling, has not been reached yet at this time. The time of initiation of expansion is directly related to the diffusion of sulphate ions.

It may be noted also that the difference between the time of initiation and the end of the test (time for which the deformation is no longer measurable) was approximately the same for the 1 × 1 × 10 cm^3 and the 2 × 2 × 16 cm^3.

According to the results of this experimental campaign, the test procedure was improved. The cross section of the specimens was decreased. Nevertheless, the dimensions of the specimens must be consistent with the mortar mixtures. Using 2 × 2 × 16 cm^3 mor-

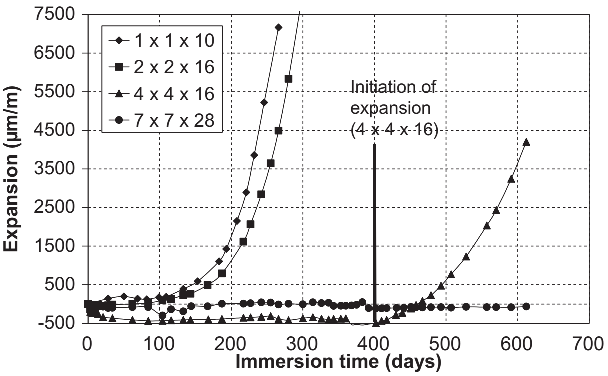


Fig. 4. Expansion of immersed mortars specimens, $\left[SO_4^{2-}\right] = 3$ g/L.

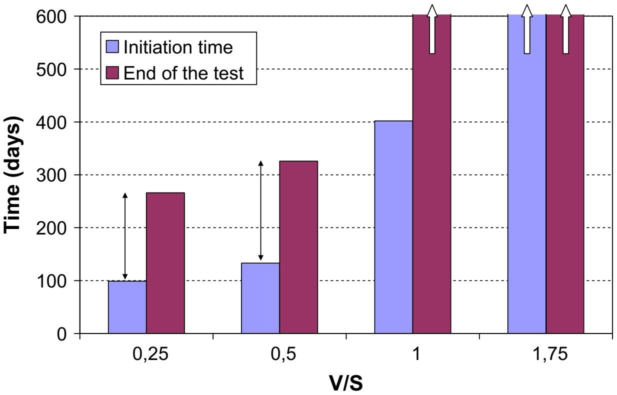


Fig. 5. Initiation time for expansion and time end of the test.

tar specimens seemed to be a good compromise between the representativeness of the specimens and the duration of the test. The specimens dimensions were actually consistent with the 0/2 mm sand used in the mortar mixtures: the smaller side of the specimen is ten times larger than the maximum particle size. Moreover, in order to validate the assumption of a minimum proportion of the section affected by the formation of expansive products, the procedure was adapted to assess the leaching kinetics.

3.3. Evaluation of the new procedure

The results of the first studies on concrete and mortar have shown that the relative mass decrease of the specimens exposed to sulphate is a global consequence of the ingress of sulphate and the leaching of hydration products of cement. The first stage where no expansion was measured hid the phenomena that occurred during this phase. Thus, it is necessary to follow a procedure that allows to keep the boundary conditions constant and to quantify the species involved in leaching. The experimental procedure detailed in (§ 2.2) is then adopted.

A strong influence of the sulphate concentration on the kinetics of degradation has been reported in previous studies [21,29,30]: higher is the concentration, faster is the attack. In order to validate the experimental procedure and investigate the effect of this parameter, an experimental study was designed.

On Fig. 6, the amount of leached calcium (Ca^{2+}) is plotted vs. the amount of leached hydroxide (OH^-). An increase in sulphate concentration led to an increase in leaching of calcium hydroxide out of the cement matrix. The ratio between the leached calcium and the leached hydroxide is close to 0.5 for the 3 and 10 g/L sulphate concentrations. This ratio can also been found in the following equation describing the leaching of portlandite:

$$Ca(OH)_2 + 2H_3O^+ \rightarrow Ca^{2+} + 4H_2O \tag{1}$$

The ratio is different for the concentration of 30 g/L because in addition to leaching of portlandite, an additional quantity of calcium ions could be released by the dissolution of gypsum, according to Eq. (2), probably due to the high concentration of sodium; as sodium sulphate solution is used.

$$CaSO_4 \cdot 2H_2O \rightarrow SO_4^{2-} + Ca^{2+} + 2H_2O \tag{2}$$

Mass differences between the exposed and the control specimens are plotted on Fig. 7. It can be seen that for any sulphate concentrations, mortar specimens had approximately the same response to the attack but with a delay in time at low concentrations. Two stages could be identified in the evolution of the mass and the length (identified on Figs. 7 and 8 with arrows on the 3 g/L of SO_4^{2-} curve), namely:

- a first stage corresponding to the latency period, when no significant expansion can be measured with a relative mass decrease caused by leaching,
- a second step corresponding to a mass and length increase due to swelling of damaged mortar specimens.

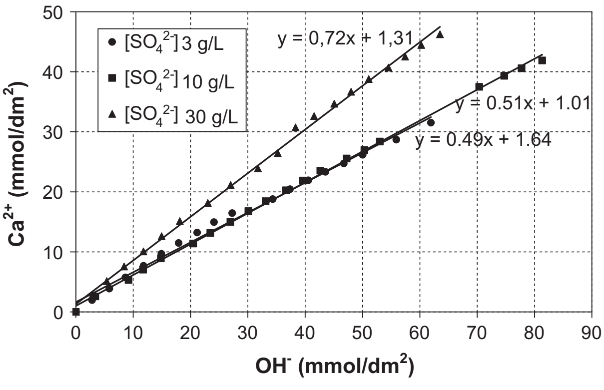


Fig. 6. Leached calcium vs. leached hydroxide.

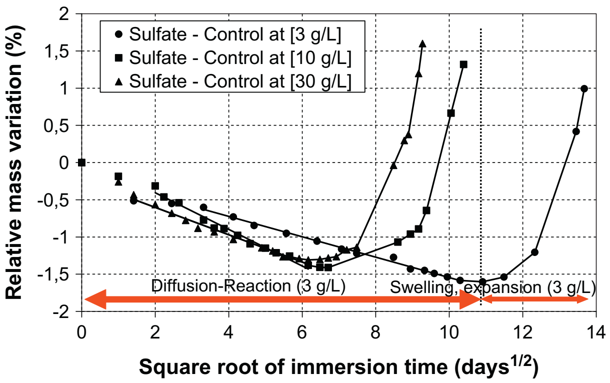


Fig. 7. Difference of mass variation between attacked specimens and control.

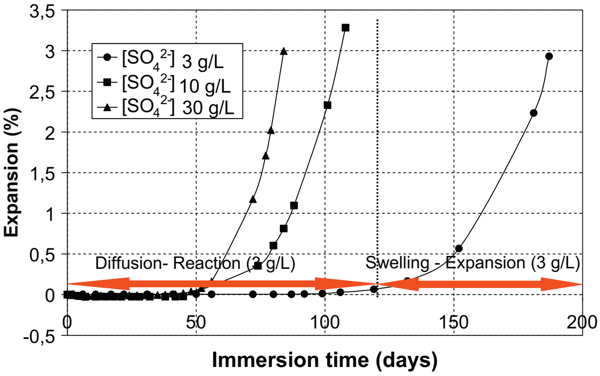


Fig. 8. Expansion data for 3, 10 and 30 g/L of Na_2SO_4 solutions.

These results are quite similar to those obtained in other studies where these two phases were also distinguished on mortars immersed in sodium sulphate solutions at different concentrations [14,15,31].

It should be noted that these measurements were made on saturated specimens. Thus the mass increase can be explained by the swelling of the specimens.

Taking into consideration the correlation of the mass and length variations, we notice that at the first stage, leaching was the dominant phenomenon which explains this relative mass decrease. At the second stage, the formation of expansive products induced expansion and mass increase.

The kinetics of leaching can be described through linear functions of square root of time:

$$n_{Ca^{2+}}(t) = k_{Ca^{2+}} \cdot \sqrt{t} \quad \text{and} \quad n_{OH^-}(t) = k_{OH^-} \cdot \sqrt{t} \tag{3}$$

On Fig. 9, the kinetics of leaching are correlated with the time of initiation of expansion t_i presented on Fig. 8. Higher the kinetics coefficients of leaching were, shorter the initiation time of swelling was. This confirmed the predominant part of diffusion and leaching on ESA. Fig. 10 shows the length measurements as a function of leached ions. At early exposure age, an important quantity of calcium and hydroxides was leached but no expansion was measured. Afterwards, expansion increases with a moderate rate of leached ions. Fig. 10 shows also a common threshold required to trigger

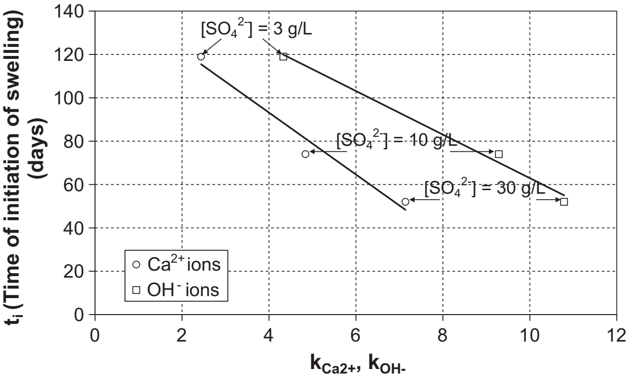


Fig. 9. Time of initiation of swelling vs. kinetics of leaching of Ca^{2+} and OH^-.

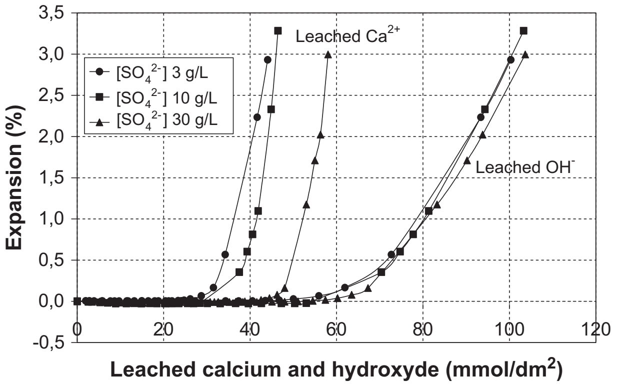


Fig. 10. Strain vs. leached ions (calcium and hydroxide).

the expansion. Whatever the concentration was, the expansion began at the same level of leached ions. Since the mixtures were the same for the three tests, the calcium hydroxide content was the same at the beginning of the tests. So the time of initiation of expansion would correspond to the same amount of leached material and ingress of sulphate ions, which could be linked to a critical proportion of the cross section of the specimens required to generate global swelling (Fig. 11).

Microscopic and macroscopic analyses were realised beside these measurements. As for example, Fig. 12 shows an energy dispersive spectroscopy analysis on an attacked mortar sample for the three different concentrations. The results consist in maps of calcium, silica, sulphur, and aluminium relative concentrations. The high calcium concentrations correspond to the cement paste and the highest silica concentrations refer to the aggregates as siliceous sand was used. Relatively high sulphur concentrations could be observed around aggregates. They were associated with high calcium concentrations. This reveals the presence of gypsum ($CaSO_4$, $2H_2O$). If ettringite $C_3A.3C\overline{S}.H_{32}$ was present, a higher localised concentration of aluminium should be detected; which is not the case. Gypsum crystals were mainly formed in the interfacial transition zone (ITZ) which can be associated with cracking, spalling and cohesion decrease. However, it is not clear if the formation of gypsum is the cause or the consequence of swelling. Mehta [32] explained the presence of gypsum in the ITZ by microcracks existing previously to sulphate exposure. These microcracks, larger than capillary pores, are a preferential path for the creation of interconnections leading to a more permeable system. So the diffusion of sulphate ions is preponderant in this zone. The effect of cracking and spalling on the transport processes is also mentioned in the modelling of ESA as for example in the meso-scale model developed by Idiart et al. [33]. Bonen and Sarkar [34] have also studied the replacement of CH by gypsum in the ITZ and they found thick deposits of gypsum up to 50 μm precipitating in this zone by a through-solution mechanism. These observations showed the important role of the ITZ that would be absent if the investigation was led on paste samples.

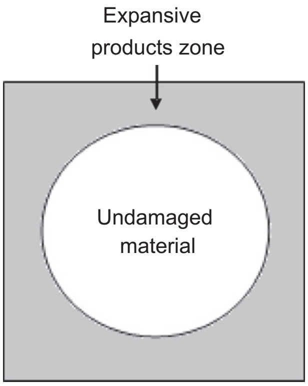


Fig. 11. Required critical section to generate swelling.

This study on the influence of sulphate concentration validated the method of investigating the ESA by showing the accelerator effect of high concentrations and highlighting the role of leaching that explains the mass loss. This method may be used in future studies to evaluate several parameters that could influence the attack in order to explain the degradation mechanism.

4. Conclusion

This paper presents the results of a study which aimed to define a new method to study the external sulphate attack (ESA). The investigation deals with the scale of the study, the exposure conditions, and the assessment of the degradation indicators. The new procedure provided results which help to understand the effect of the sulphate concentration.

Several concrete mixtures cast as 7 × 7 × 28 cm^3 prisms were exposed to two different sodium sulphate concentrations (3 g/L and 30 g/L of SO_4^{2-}). Tests were performed at constant pH and the solution was renewed regularly. The results of these tests confirmed that the time when global swelling can be detected through length monitoring may exceed several years and the measurement of expansion is not sufficient to assess the potential durability of concrete and understand the degradation mechanism. The monitoring of mass showed the influence of diffusion and leaching.

Another experimental campaign was led to choose the appropriate specimens size. Mortar specimens of different sizes were tested (1 × 1 × 10; 2 × 2 × 16; 4 × 4 × 16 and 7 × 7 × 28 cm^3). The 2 × 2 × 16 cm^3 prisms were found to be a good compromise between the test duration and its representativeness.

The experimental results of the two previous investigations underlined the part of leaching in the sulphate attack. Thus a new procedure was designed to keep the boundary conditions constant and to quantify the leached ions to estimate their impact on the degradation mechanism.

This procedure helped in validating the effect of the concentration of the sulphate solution in the range 3–30 g/L SO_4^{2-}. As expected, increasing sulphate concentration leads to faster degradation and increases the leaching of portlandite. The assessment of the leaching kinetics of calcium and hydroxides highlights the main role of leaching in ESA: leaching is the driving phenomenon that triggers the sulphate attack. The initiation time of swelling de-

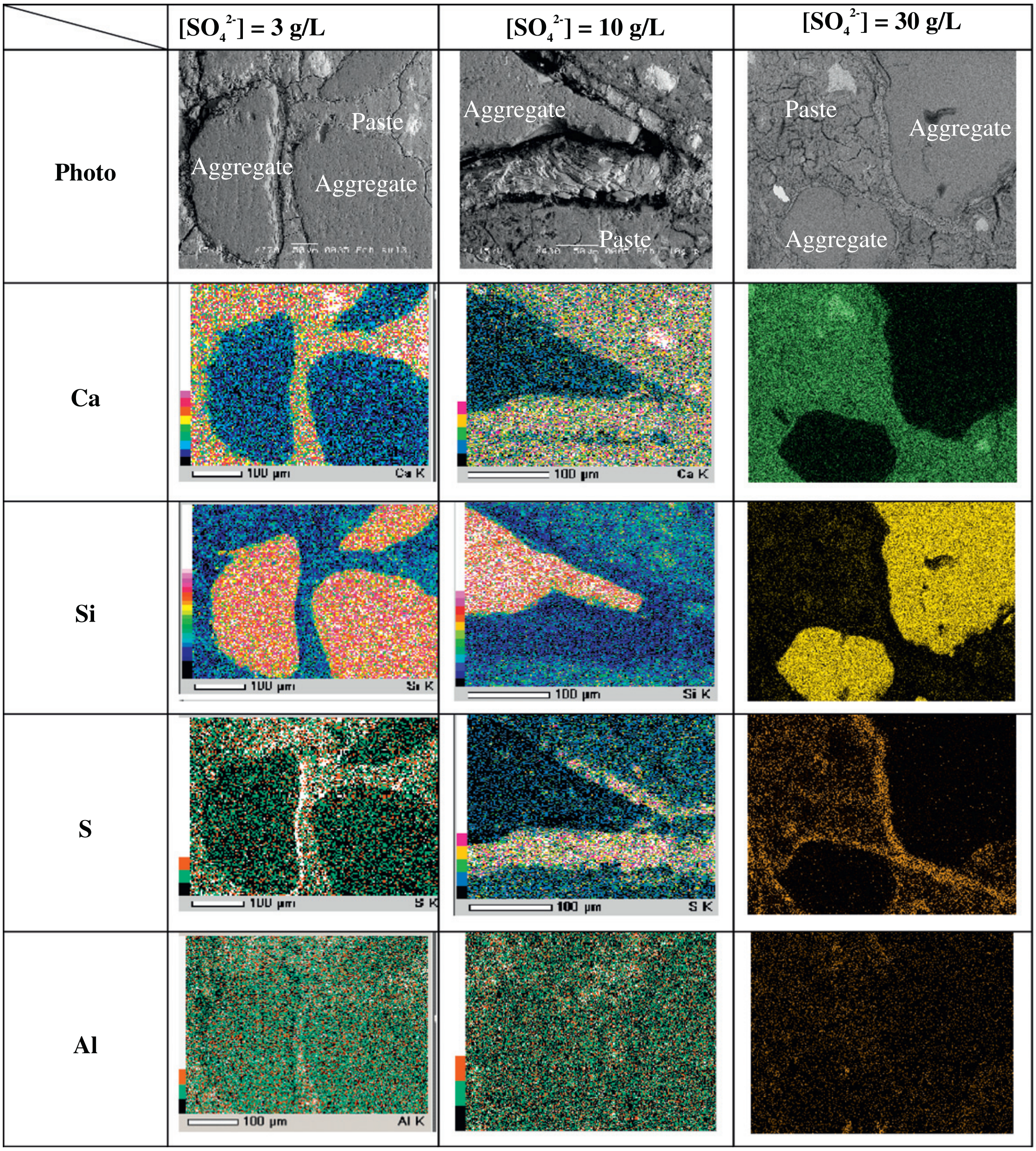


Fig. 12. Energy dispersive spectroscopy analysis on an attacked mortar sample.

pends of the kinetics of leaching. Furthermore, correlating the expansion and the amounts of leached ions showed that in order to generate a global swelling, a critical proportion of the cross section of the specimens should be degraded. No significant expansion could be measured if this section is not reached.

As the new procedure is likely to provide reliable data to assess the causes and consequences of ESA, other studies dealing with the effect of the composition of cementitious materials could be useful to understand the mechanism of degradation and the influence of the composition or curing parameters.

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