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Sulfate transfer through concrete: Migration and diffusion results

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ABSTRACT

This paper presents a study on the effect of sulfate ions on concrete. The sulfate attack is approached in two different ways: results in natural diffusion are compared to migration results. Both sodium sulfate and magnesium sulfate are used as test agents. The comparison is presented in terms of concentration profiles and amounts of sulfate reacting with the cementitious matrix, together with mechanical resistances. It is shown that the electrical field has impact on the transfer of sulfate: when Mg^{2+} is used as the counter-ion, the penetration depth proves to be larger in migration than the depth obtained with sodium sulfate. It is the opposite case in natural diffusion. A possible explanation could be found in the kinetics of brucite formation. Finally, the combination of magnesium sulfate with the electrokinetic technique results in significant mechanical strength losses after 6 months of test, while natural diffusion attacks lead to a much smaller loss.

1. Introduction

Sulfate attack is one of the major threats for cement-based materials durability. When concrete is in contact with salts containing sulfate, interactions between the solid phase and the sulfate solutions occur, leading to chemical reactions within the material. Such "reaction fronts", as mentioned by Lothenbach et al. [1], can lead in time to the deterioration of the material.

The sources of sulfate in the environment of concrete are numerous: concrete may be exposed to groundwaters or soils. The same can be said for seawaters [2]. Other types of sulfate attack exist. See for example Ref. [3] on internal sulfate attack, or Refs. [4–6] on thaumasite formation. Ettringite and gypsum are observed as reaction products depending on the level of the sulfate concentration and on the cement composition. Ettringite is formed in high C_3A cements while gypsum is formed in low C_3A cements. [7–10]. The interactions of sulfate with the cement matrix result in disruptive expansion of the material, and significant loss of mechanical strength [11,12].

Whereas the research community dedicated much effort during the past decades to the understanding of sulfate attack to concrete with the aim of preventing it, the objective of this study is, on the opposite, to understand better the interactions between sulfate and concrete in order to enhance the attack. The reason of this choice is to be found in an issue that the civil engineering community will have to face in the very near future: the deconstruction of concrete-based structures and its economical and societal costs. The challenge is to propose original methods for weakening the structures so that the time spent on using more classical methods of deconstruction (such as mechanical systems: hammering etc...) is shortened. Furthermore, we are looking for alternative methods that do not require the continuous presence of workers on site. Thus the idea of employing and enhancing phenomena which are famous for being the main threats of cement-based materials: it was decided to act at the level of the cement matrix by forcing the transfer of an external species whose deleterious effects are well-known, sulfate. By forcing, we mean controlling the direction of transfer, accelerating the transfer and the kinetics of reaction. For all these reasons, and since sulfate is an ionic species, an external electrical field was employed within a set of migration procedures.

Because most concrete structures are reinforced structures, a first part of the work consisted in developing a technique that would destroy the continuity of the steel reinforcement in order to avoid their Faraday's cage effect [13]. These results are not presented here. This paper focuses on the material aspects only. We present a comparison between the sulfate transfer through concrete under an electrical field and in natural diffusion conditions in terms of mainly concentration profiles, amount of reacting sulfate, and mechanical resistances.

2. Materials and procedures

The tests were performed on a CEM-I concrete based on a type I cement with a water to cement ratio by mass of 0.48. Table 1 presents the chemical analysis of the cement while Table 2 gives the concrete composition and properties. The water porosity and

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Nomenclature

c	concentration of one species per unit of volume of solution, mol m^{-3}
D	diffusion coefficient, m^2 s^{-1}
F	Faraday constant, 9.648×10^4 J V^{-1} mol^{-1}
j	current density, A/m^2
J	diffusive flux, mol m^{-2} s^{-1}
L	thickness, m
p	porosity
R	universal gas constant, 8.314 J K^{-1} mol^{-1}
t	time, s
T	temperature, K
U	external electrical potential, V
x	distance across the material, m
z	charge number
Greek symbol	
φ	electrical potential, V
Subscript	
T	total

Table 1
Chemical analysis of the cement.

Chemical composition (%)	
SiO_2	20.29
$A1_2O_3$	5.56
Fe_2O_3	2.32
CaO	64.22
MgO	2
SO_3	3.17
K_2O	0.62
Na_2O	0.56
Loss of ignition	1.54
Bogue composition (%)	
C_3A	10.8
C_3S	65.5
C_2S	10.8
C_4AF	7
Gypsum	3

Table 2
Concrete composition and properties.

CEM I 52.5R, Martre Tolosane (Lafarge)	375 kg/m^3
Aggregates 4/10	1038 kg/m^3
Sand 0/4	692 kg/m^3
Water	180 kg/m^3
Limestone filler	50 kg/m^3
Superplasticizer (in %mass of cement)	1.88 l/m^3
W/C	0.48
Apparent density	2296 kg/m^3
Water porosity	13%

apparent density were measured following the classical AFREM protocol in which the samples are first of all vacuum saturated in order to reach the open porosity. The cement is a standard one with a regular C_3A and sulfate content in order to highlight at best the external sulfate attack that might concern regular buildings that are candidates for deconstruction. The concrete was cast into cylindrical moulds of 22 cm height and 11 cm diameter, and immediately stored in a wet room at controlled temperature (20 °C). Twenty-four hours after casting, the samples were demolded and stored for at least 3 months in the same wet chamber prior to testing. The choice of a minimum of a 3 month curing period was made in order to approach complete hydration of the CEM-I concrete samples.

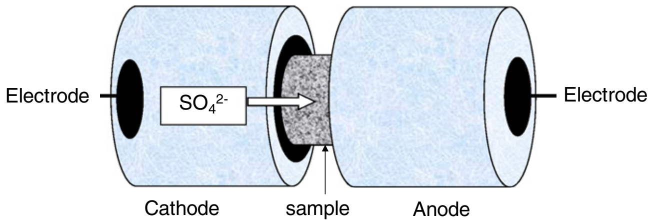

Fig. 1. Migration test schematic.

The migration cells design was inspired from classical chloride migration cells: samples of concrete were sandwiched between two electrolyte compartments, each of them containing 2.5 l of electrolyte solution. Two round perforated stainless steel plates were used as electrodes for applying an external electrical field, and placed on the left-hand side of the cathodic compartment and on the right-hand side of the anodic compartment respectively (see Fig. 1). Two other stainless steel electrodes were placed in contact with the two surfaces of the tested samples in order to measure the actual electrical potential difference. Different configurations were tested, each of them corresponding to a sulfate concentration of 40 g/l on the cathodic side. The associated cation was either sodium or magnesium. The anodic compartment was filled with a solution of 50 g/l of magnesium sulfate. The duration of the tests varied from 4 days to 9 months. The applied electrical potential difference was 60 V. The 11 cm diameter cylindrical concrete samples were 11 cm thick. The lateral surface of the sample was coated with a resin to prevent from transfer with the environment.

In addition, natural diffusion tests were performed using the same type of experimental set-up for which the samples were in contact with a solution of either Na_2SO_4 or $MgSO_4$. In each case, the sulfate concentration was 40 g/l. The test duration was 4 months.

The sulfate concentration profiles were obtained by grinding the core of the concrete samples with a constant grinding step of 2 mm. The extraction of the sulfates from the powder was achieved by acidification with 14 g/l of hydrochloric acid. The sulfate content was then measured by ionic chromatography. Note that this procedure leads to the measurement of the total sulfate content.

3. Results and discussion on sulfate transfer

3.1. Kinetics of sulfate transfer

When a sulfate solution is in contact with a saturated cementitious material, two processes occur. First, due to the concentration gradient created between the outside and the interior pore network, sulfate ions enter the material and diffuse through the pores from the high concentration zone to the low concentration zones. Second, sulfate ions react with the cement matrix [14]. What is commonly called external sulfate attack [7,15] is the sum of two processes: diffusion through the materials, and chemical reactions leading to disorders of the materials.

The migration tests were applied on 11 cm thick samples, the diameter of the cylinders remaining 11 cm. A preliminary issue was to make sure that the pore solution of the 11 cm thick samples could be saturated in sulfate. For this purpose, tests were performed in which the cathodic solution contained Na_2SO_4 (60 g/l), while the anodic compartment was filled with 10 g/l of Mg^{2+} in a solution of $MgCl_2$. A constant electrical potential difference of 60 V was applied for 12 days which corresponded to an actual electrical field of 530 V/m between the two faces of the material.

In order to check whether the applied electrical field could create a heat transfer during the experiment, the temperatures of the solutions in the two compartments were monitored. The two probes indicated a constant temperature of 23 °C during the test. The volumes of solutions in the two compartments were also checked. None of them varied during the test, which means that we were not able to detect any electro-osmotic effect.

After 12 days of test, the experiment was stopped and the sample was evaluated for its total sulfate content. The objective was not to determine a concentration profile at this stage of preliminary tests, but to verify that the sample can be saturated in sulfate ions. The average initial amount of sulfate in the material was both calculated from the cement composition and measured by ion chromatography from a reference sample of concrete, leading to the same value of 5.6 g/kg of concrete.

From the values of the sulfate concentration in the cathodic compartment, the water porosity and the apparent density, the amount of free sulfate that the pore network may contain could be calculated as 2.3 g/kg of concrete.

The measurements of the total sulfate content in the material gave a value of 9 g/kg of concrete which is a concentration at least equal to the sum of the initial sulfate content and the free sulfate content on both sides of the sample. Furthermore, sulfate was detected in the cathodic compartment (by ion chromatography) at the end of the test in a concentration of 0.3 g/l.

From the continuity equation (Eq. (1)), the time to cross the sample can be predicted, in an order of magnitude sense. Indeed, assuming a saturated material in the absence of a pressure gradient, the continuity equation becomes, in the case of a one-dimensional transfer

$$\frac{\partial c_T}{\partial t} = D\left(\frac{\partial^2 c}{\partial x^2} + z\frac{F}{RT}\frac{\partial c}{\partial x}\frac{\partial \varphi}{\partial x}\right) \tag{1}$$

where c_T is the total concentration of sulfate, c is the free concentration in the pore solution, D is the diffusion coefficient, z is the charge number, F is the Faraday constant, R is the gas constant, T is the temperature, and φ is the electrical potential. In the case of a natural diffusion process, the electrical potential is the membrane potential, i.e. the potential created between the different ionic species in the pore solution [16]. When an external electrical field is applied, φ is the sum of the external electrical potential and the membrane potential.

In an order of magnitude sense, it is assumed that $\frac{\partial \varphi}{\partial x} \sim \frac{\Delta U}{L}$, where ΔU is the electrical potential difference, and L is the material thickness. Furthermore, neglecting the ionic interactions with the material, $c_T = pc$, where p is the porosity.

The orders of magnitude are $z \sim 1$, $c \sim \Delta c$, and $x \sim L$. Assuming that the electrical effects are much faster than diffusion, one has

$$p\frac{\Delta c}{t} \sim D\frac{F}{RT}\frac{\Delta c}{L}\frac{\Delta U}{L} \tag{2}$$

and

$$t \sim p\frac{RTL^2}{DF\Delta U} \tag{3}$$

which in our case means, for a diffusion coefficient with an order of magnitude of 10^{-12} m^2/s, that the time to cross an 11 cm thick sample is about 6 days.

This analysis neglects the sulfate reaction with the cementitious matrix which slows down the sulfate transfer from one side to the opposite one. Accounting for this, the detection of sulfate in the anodic compartment after 12 days of tests is in accordance with the predictions. Added to the measurements inside the material, it can be concluded that the migration test allows saturating the concrete samples even though the products created by the sulfate reaction tend to obstruct the free sulfate path in the pore network.

Even more convincing are the following results: an 11 cm thick sample was placed in a migration cell, while a 60 V electrical potential difference was applied for 3 weeks. Each compartment this time contained 50 g/l of $MgSO_4$ ($[SO_4^{2-}] = 40$ g/l) which means that the boundary conditions are identical. Fig. 2 presents the total sulfate concentration profile obtained. The procedure leading to the total sulfate concentration profile, namely grinding the core of concrete, treating the obtained powders in order to extract the sulfate ions, and analyzing the sulfate content with ion chromatography, was applied 3 weeks after stopping the electrical potential difference. Plotted in Fig. 2 is also the average initial sulfate content, together with the amount of sulfate expected to be found in the saturated pore network by our calculations. From the measurements, the pore network appears to be entirely saturated with sulfate ions at the end of the experiment. In addition, at the two ends of the sample, the total amount of sulfate is greater than the sum of the amount in solution and the initial sulfate content, indicating that the sulfate reaction occurred already. The lack of symmetry at mid-distance in the concentration profile can be explained by the influence of the external electrical field. A concentration gradient in sulfate is created between the outside of the material and the pore solution, leading to the penetration of the sulfate ions from both sides. Being an anion, sulfate will be attracted by the anodic side when the electrical potential difference is applied. This means that in the cathodic compartment the sulfate concentration gradient and the electrical field are two driving forces acting in the same direction. In the anodic compartment the same two driving forces are competing: the sulfate concentration gradient forces the sulfate ions to leave the anodic compartment and diffuse through the concrete sample, while the electrical field tends to prevent the same ions from entering the material and proceeding towards the cathodic side. From this competition, arises a smaller amount of total sulfate in the vicinity of the material face in contact with the anodic compartment, compared to the face by the cathodic side.

Fig. 3 presents the total concentration profiles of sulfate measured through the samples of concrete when the electrical field was applied for 4 days, 7 days, 3 weeks, 6 weeks, and 9 months. The cathodic compartment contained 60 g/l of a sodium sulfate solution, while the anodic compartment was filled with 50 g/l of

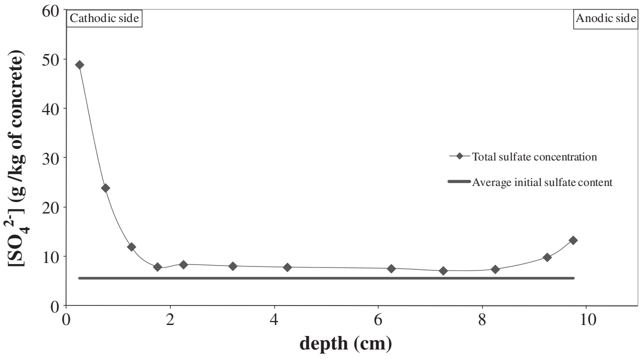

Fig. 2. Preliminary test: sulfate concentration profile through the sample after 3 weeks of migration ($MgSO_4$ is used in both compartments of the cell).

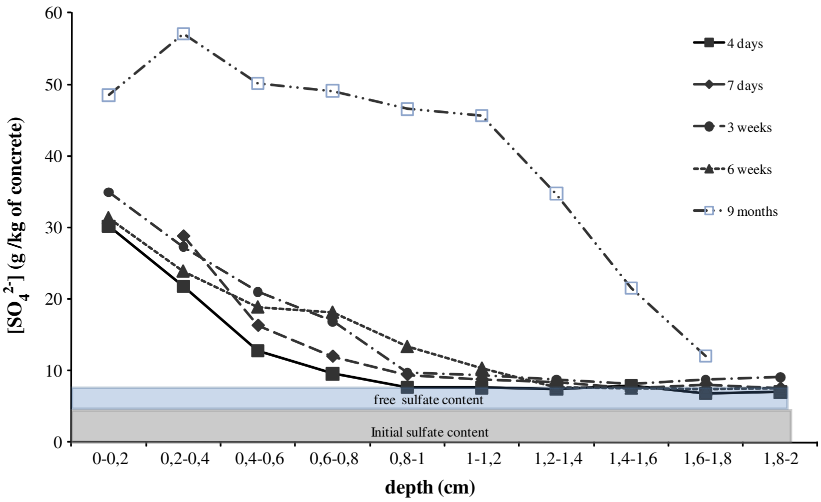

Fig. 3. Kinetics of sulfate concentration profiles during a migration test (Na_2SO_4 in the cathodic compartment, $MgSO_4$ in the anodic side).

magnesium sulfate. The analysis was carried out from the face in contact with the cathodic compartment. The first 2 cm were saturated with sulfate after 4 days of electrical field, while the sulfate ions already reacted with the cementitious matrix at this stage. Plotting the concentration profiles in time allows following the kinetics of sulfate reaction under migration. The amount of sulfate that reacted on the first mms of the sample face in contact with the cathodic solution does not vary much from the first 4 days of the migration test to 6 weeks. As time progresses, the amount of sulfate that reacted increased significantly (see the 9 months electrical field concentration profile). Fig. 3 indicates that during the first weeks of the test, the sulfate reaction proceeds from the cathodic face of the material towards the core of the sample. Such results tend to teach that after an immediate reaction with the cement-based matrix, the sulfate reaction propagates towards the core of the material. Then, in time, additional sulfate reaction occurs, leading to a greater amount of total sulfate.

The surface delimited by the horizontal curve of the sum of the average initial sulfate content and the free sulfate content, and any concentration profile, represents the entire sulfate amount that reacted with the cement matrix. Plotted in Fig. 4 is the variation of this content in time. During the first weeks, a constant increase in the bound amount of sulfate is noticed. Then, the content of bound sulfate seems to stagnate from 3 weeks to 6 weeks of applied electrical field. Finally, Fig. 4 exhibits a dramatic change in slope between the first weeks of migration tests and the results obtained after 9 months of test.

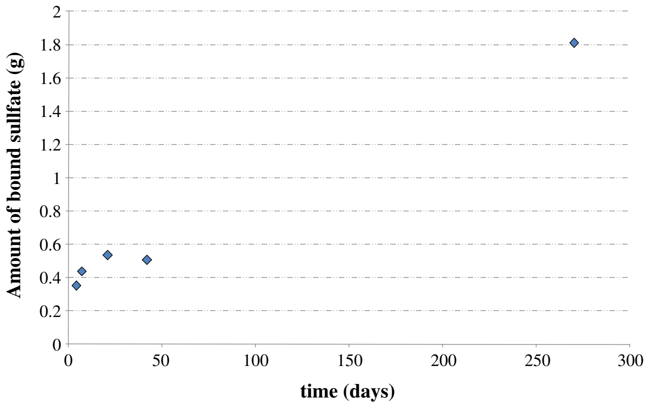

Fig. 4. Amount of bound sulfate vs. time during a migration test (boundary conditions are identical to Fig. 3).

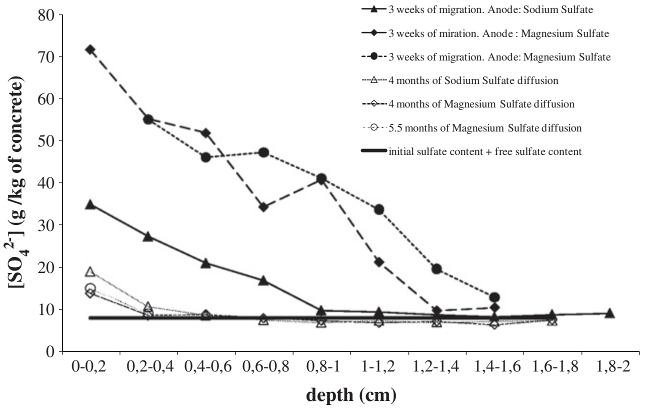

Fig. 5. Effect of the associated cation on the sulfate concentration profile in diffusion and migration.

Effect of the associated cation: Fig. 5 shows the total sulfate concentration profiles obtained after applying the 600 V/m electrical field to the concrete samples. The samples were analyzed immediately after stopping the electrical field. The only difference in the results remains in the boundary conditions. The cathodic compartment was filled either by Na_2SO_4 or $MgSO_4$. In each case the sulfate concentration was 40 g/l. The concentration profiles were measured from the cathodic side. Because the concentration profile corresponding to sodium as the associated cation indicated that the depth of sulfate reaction was about 5 mm, only the first cm is presented in the figure. Fig. 5 indicates that the kinetics of reaction is greater when the cation associated to sulfate ions is Mg^{2+}. Indeed, in the case of Na_2SO_4 the concentration in sulfate is greater than the amount corresponding to the sum of the initial content and the pore solution content, from the surface to a depth of 5 mm. When magnesium is used in association with sulfate, the depth of sulfate reaction is 8 mm which corresponds to an increase of 60%. Furthermore, the amount of sulfate that reacts with the cementitious matrix is much greater (about three times) with Mg^{2+} than the reaction products obtained with Na^{2+}.

3.2. Sulfate concentration profiles in diffusion

Fig. 5 presents also the total sulfate concentration profiles measured during a diffusion test of 4 months when associating magnesium or sodium to 40 g/l of sulfate. An additional concentration profile is plotted corresponding to a second diffusion test for $MgSO_4$ that lasted 5.5 months. The samples were analyzed in their first centimeter in contact with the aggressive solution of sulfate. Noteworthy is the fact that the pore solution is saturated with sulfate in this thickness. From 3 to 10 mm indeed, the amount of sulfate measured is 7.9 g/l, close also to the sum of the initial sulfate content from the matrix and the free sulfate content from the pore solution.

The amount of sulfate that reacted with the material is slightly greater in the case when sodium is added to sulfate. This tendency is confirmed by the second concentration profile that corresponds to a diffusion test with $MgSO_4$ during 5.5 months. Yet the depths of sulfate reaction are similar for the two associated cations. Comparing the concentration profiles obtained during the migration tests with the concentration profiles measured in diffusion, one sees immediately the impact of the electrical field on the sulfate transfer. Previous works on other ions [17–20] led to the conclusion that applying an external electrical field accelerates the transfer of ionic species through the pore system of cementitious materials. This is the case also here. The electrical field, just like in chloride transport [21], also accelerates the depth of reaction whatever the cation associated to sulfate, sodium or magnesium. Fig. 5 demonstrates also that the electrical field has a strong impact on the kinetics of reaction: the amount of sulfate that reacted with the cement-based matrix is much greater than in the diffusion case. More surprising is the fact that the amount of reaction product is greater with $MgSO_4$ under an electrical field, while it is the opposite (more sulfate reaction) with Na_2SO_4 in diffusion. This tendency is confirmed by Fig. 6 that plots the results of the bound amount of sulfate both for $MgSO_4$ and Na_2SO_4. The data presented in Fig. 6 were obtained as follows: considering that the amount of total sulfate measured within the first mm of the samples represents with a good accuracy the surface total sulfate concentration, we deduced that the difference between this concentration and the boundary sulfate concentration (40 g/l) would provide the trend of the bound amount of sulfate, even though this calculation does not account for time effects. Fig. 6 indicates that the combination of an electrical field together with $MgSO_4$ rather than Na_2SO_4 leads to a greater amount of sulfate products in the material.

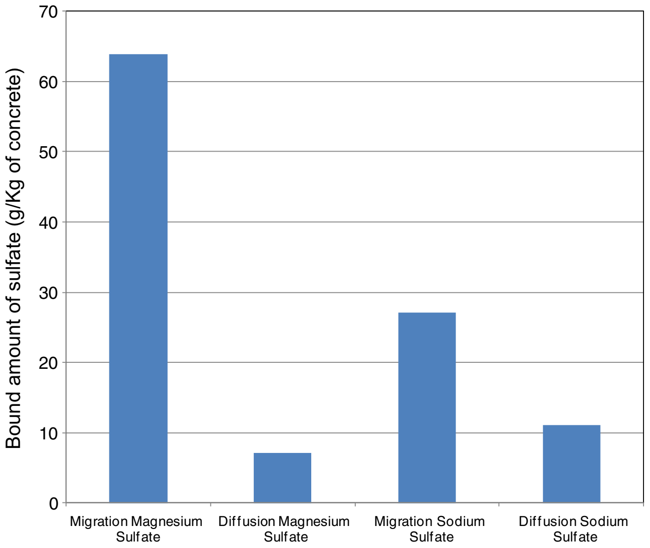

Fig. 6. Bound sulfate from migration and diffusion tests.

During a diffusion process of $MgSO_4$, the dissolution of portlandite allows the formation of gypsum together with brucite through the following chemical reactions [14]

$$[Ca(OH)_2] \rightarrow Ca^{2+} + 2OH^- \tag{4}$$

$$Ca^{2+} + 2OH^- + Mg^{2+} + SO_4^{2-} + 2H_2O \rightarrow CaOSO_4 2H_2O + Mg(OH)2 \tag{5}$$

Brucite is commonly observed through SEM investigations as reported for example by [22,23]. According to [24,25], brucite would be the first product created, leading to the precipitation of gypsum. A direct consequence of such a process is that the pore network is blocked by brucite which lowers and delays the sulfate reactions. This explains why the amount of bound sulfate is greater in the case of Na_2SO_4 diffusion than when $MgSO_4$ diffuses through the concrete samples.

From the results obtained in migration tests, to the contrary, the sulfate reaction is greater when associating magnesium to sulfate. Fig. 7 presents an example of SEM observations for a piece of concrete taken close to the $MgSO_4$ diffusing side during a migration test. It is noteworthy to emphasize that Fig. 7 is one photograph among many that were taken. No brucite was ever observed. Even though more investigations need to be pursued, a first hypothesis could be that the electrical field prevents or delays the combination of magnesium ions with hydroxide to brucite. Therefore in the absence of pore obstruction, secondary gypsum is free to form in strong amounts. In the case of a magnesium sulfate attack, magnesium sulfate hydrates are expected to replace C–S–H resulting in a loss of cohesion properties. More investigations will have to be done in order to visualize it through SEM observations. It is assumed that this was the case here due to the loss in mechanical resistance that was measured.

3.3. Compressive strength

Compressive strength tests were performed on concrete samples after both the migration sulfate attack and the diffusion sulfate attack. Because the geometry of the specimens was 11 cm in height, and 11 cm in diameter, the reference tests on concrete not submitted to the sulfate attack were done on the same geometry. The length to diameter ratio of 1 does not allow a comparison with results available in the literature since the restraining effect of the platens tends to mask the effects of cracking. Yet all the samples were cast, cured, and conditioned in the same manner which

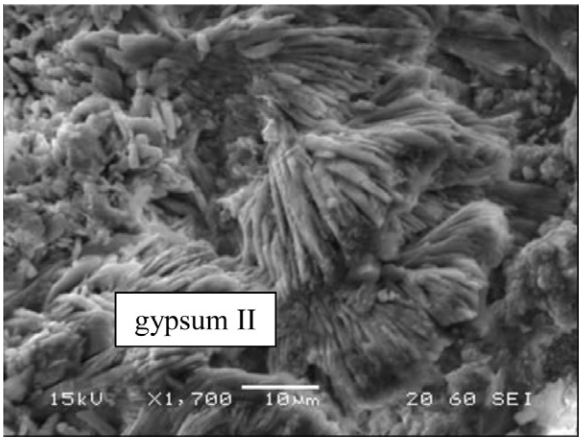

Fig. 7. Example of an SEM picture obtained during the migration of $MgSO_4$.

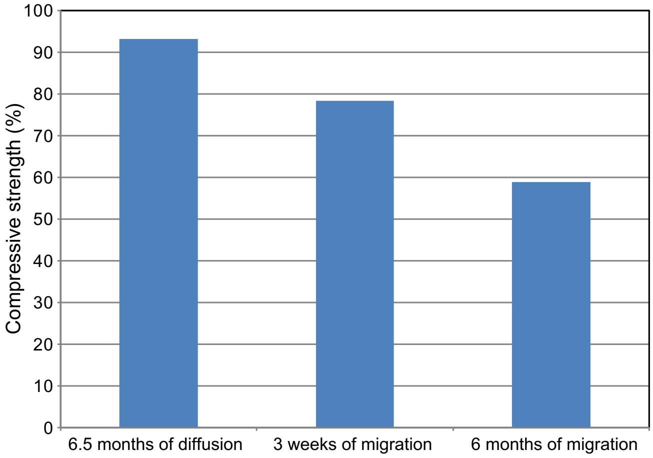

Fig. 8. Loss in compressive strength after $MgSO_4$ attack in diffusion and migration.

allows a relative comparison between them. In each case, three samples were tested with a standard deviation in the results ranging from 2 to 4 MPa (after 6 months of $MgSO_4$ migration for the latter). In each case also, samples not submitted to the sulfate attack were tested in compression at the same time as the attacked samples. In the absence of attack, the average mechanical resistance was on average 50.2 MPa; it was 46.6 MPa in diffusion, 41 MPa after 3 weeks of migration and 28.5 MPa after 6 months of migration.

Plotted in Fig. 8 are the relative results obtained when magnesium is the cation associated to sulfate after 6.5 months of immersion (diffusion process), 3 weeks of migration, and 6 months of migration. The loss in compressive strength is 7% after 6.5 months of diffusion while the same duration of tests leads to a decrease of 40% when an electrical field is applied. From the literature the loss in mechanical resistance can be explained by the local replacement of C–S–H by M–S–H. The impact of the electrical field on the sulfate attack and therefore on the macroscopic behavior, is such that after only 3 weeks of migration, the loss in compressive strength is 20%. One to 2 years are necessary to obtain similar results in natural diffusion, see for examples the works of [11,26,27]. Still, it is important to note that this loss in mechanical performance is not linear in time since multiplying the application time of the electrical field by 9 leads to only double the loss in compressive strength. Such a result is based in the link with the kinetics of the bound amount of sulfate, in other words the evolution in time of the sulfate reaction with the cementitious matrix as highlighted in Fig. 4.

4. Concluding remarks

This paper documented a study of sulfate attack through CEM I concrete by means of two different driving forces: (i) a concentration difference, i.e. natural diffusion, and (ii) an external electrical potential difference together with a concentration difference, i.e. migration.

Concentration profiles in (total) sulfate are presented both in migration and in diffusion. They show the impact of the electrical field on the kinetics of sulfate penetration through the pore network. The use of an electrical field allows one to decouple the penetration of sulfate from the sulfate reaction with the cementitious matrix. It was shown that the amount of sulfate reacting with the matrix does not increase linearly in time.

The effect of the associated cation was also investigated, an effect which is linked to the type of driving force. In migration, the penetration depth of sulfate is greater with $MgSO_4$ together with the amount of reacting sulfate. It is the opposite in diffusion where the reaction products are greater when Na^+ is the counter-ion. It is suggested that this could be due to the kinetics of brucite formation which is slower than the penetration time of sulfate under an electrical field. During a diffusion process, which is much slower, enough time is devoted to the brucite formation and the pores can be obstructed, preventing further sulfate from diffusing and reacting.

Finally, it was shown that using an electrical field to enhance the sulfate attack results in a dramatic reduction in the time necessary to decrease the mechanical performance: a loss of 40% in compressive strength is obtained after 3 weeks, while the loss is only 7% after 6.5 months of diffusion.

Even though more investigations need to be implemented in the future, the above results are extremely encouraging in the pursuit of a deconstruction objective.

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