Pergamon

CEMENT AND CONCRETE RESEARCH

Cement and Concrete Research 30 (2000) 83–90

Modelling of leaching in pure cement paste and mortar

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Received 28 May 1999; accepted 21 September 1999

Abstract

The leaching of cement-based materials is analysed through experimental and numerical results. From the experimental point of view, the leaching processes of a pure cement paste and a mortar are characterised by the degraded depths and the cumulative amount of leached calcium at different times. From the mathematical point of view, the leaching is modelled with the mass balance equation of calcium in the liquid phase. Material properties useful for a numerical resolution of this equation are presented and the finite volume method is applied for the numerical simulations. Comparisons between experimental and predicted results show the validity of the simple model used to describe the leaching phenomenon and the ability of the finite volume method to support high nonlinearities.

Keywords: Waste management; Cement paste; Mortar; Degradation; Finite volume analysis

1. Introduction

Predicting the long-term behaviour of concrete, particularly concrete used for radioactive waste disposal, requires a sound knowledge of the various deterioration mechanisms that will affect the structures over their lifetimes and the extensive use of simulation tools. The object of this paper is to describe a model that predicts the extent of chemical degradation by the action of pure water on a cement paste or a mortar. Indeed, constantly renewed water appears to be one of the aggressive agents that attack the chemistry of concrete.

Deionized water in contact with cement-based materials create concentration gradients that lead to the diffusion of ions contained in the interstitial solution. These transfers modify chemical balances and induce a total leaching of Portlandite and a progressive decalcification of C-S-H.

The two-dimensional model DIFFU-Ca was developed for calculating the variation of the degraded depth and the leached calcium flux with time for cement-based materials. In what follows, DIFFU-Ca is described and validated only for one-dimensional problems by comparing its results with experimental data obtained with both cement pastes and mortars.

2. Experimental program and results

2.1. Materials

Two different cement-based materials were investigated: a pure Portland cement paste and a mortar made with the same Portland cement and a quartz sand. The volumetric proportion of paste in the mortar was approximately 50% in volume. The sand was not attacked by leaching on the timescale considered. All the paste and mortar samples were made with the same ordinary Portland cement (OPC). The chemical composition is given in Table 1. The water/cement (w/c) ratio of both materials was 0.4.

The mixtures were poured into cylindrical moulds (7 × 8 cm for paste and 11 × 22 cm for mortar). All the samples were demoulded after 24 h and then were cured for 6 months under lime-saturated water at 20°C. These curing conditions led to maximum hydration and to partial leaching of alkalis.

2.2. Leaching process

The samples, protected from atmospheric CO_2, were immersed in water at a constant temperature of 20°C, and kept at pH 7 by small additions of nitric acid. The leaching process resulted from the concentration gradients between the pore solution of the cement-based material and the deionised water that surrounded the sample. These gradients induced a diffusion process of all species present in the pore

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0008-8846/00/$ – see front matter
PII: S0008-8846(99)00208-2

Table 1
Chemical composition of the cement

CaO	SiO_2	Al_2O_3	SO_3
62.9	20.6	5.8	3.1

solution of the material toward pure water, and they broke the initial local equilibrium between the solid and the liquid phase. Consequently, a partial dissolution of the cement-based matrix occurred to restore the local equilibrium between solid and liquid phases. The solution in which the samples were immersed was frequently renewed to maintain a constant boundary condition and was analysed to monitor the leaching kinetics of calcium.

2.3. Experimental results

The altered section of both materials that were immersed in deionised water consisted of a solid phase assemblage of which each zone, with constant mineralogy, was separated by a dissolution or precipitation front (Fig. 1). These clearly defined fronts show that as soon as a solid phase is under- or supersaturated with respect to the contacting solution, it immediately precipitates or dissolves [1].

The evolution of the degraded depth, delimited from the core by the Portlandite dissolution front, is proportional to the square root of time. For the pure paste and for the mortar, these evolutions are very close, with about 1.4 mm of degradation after 3 months and 2.1 mm after 7 months for both materials.

The outgoing leaching fluxes of calcium are also proportional to the square root of time. The kinetics for the mortar can be deduced from that of pure paste using the proportion of paste in the mortar as a reducing factor [2] (Fig. 2). Indeed, for a 50% proportion of cement paste in the mortar, the effects of tortuosity due to sand and interfacial transition zone (ITZ) complement one another.

3. Modelling of pure cement paste leaching

The existence of a solid phase assemblage with clearly defined dissolution fronts can only be explained by assuming local equilibrium (i.e., instantaneous dissolution). Since the diffusion rates are much slower than those of the chemical reactions, the kinetics of degradation are governed by a diffusion process.

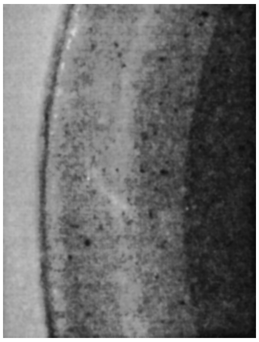


Fig. 1. Degradation fronts in a cement paste sample (from [1]).

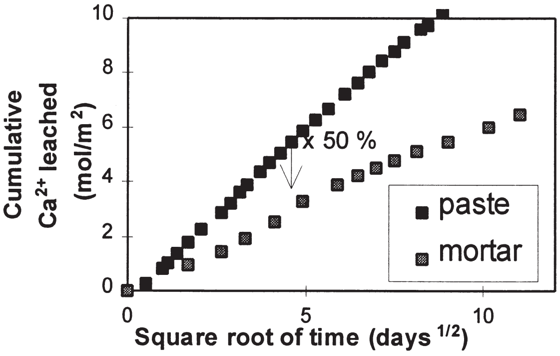


Fig. 2. Experimental cumulative leached calcium (from [1,2]).

Adenot [1] has developed a model called DIFFUZON by combining diffusion transport and local chemical equilibrium for all species of a cement material. The kinetics of degradation are proportional to the square root of the time, as long as there is an unaltered zone (infinite medium) and an aggressive solution with a constant composition. The model can account for the main solid phases of the hydrated cement in equilibrium with the pore solution. The influence on the porosity and diffusion coefficient of the dissolution and precipitation in the degraded portion is taken into account. However, the model, which requires a Boltzmann variable change (relying on a single, variable time and space), can only be applied for a unidirectional problem with constant limiting conditions.

The previous model had to be simplified to develop a three-dimensional model for studying the influence of cracks and to investigate real structures with variable-limiting conditions. The simplification assumes that the single evolution of calcium describes the leaching of cement-based materials since all hydrates contain calcium [3–5]. Therefore, the model DIFFU-Ca is based on the resolution of the mass balance equation for calcium only [see Eq. (1)].

$$\frac{\partial(\phi \cdot C_{\mathrm{Ca}})}{\partial t} = \mathrm{Div}[D_e \cdot \mathrm{Grad}(C_{\mathrm{Ca}})] - \frac{\partial S_{\mathrm{Ca}}}{\partial t} \qquad (1)$$

Where t is the time, C_{Ca} is the calcium concentration in pore solution, S_{Ca} is the calcium concentration in the solid phase, ϕ is the porosity, and D_e is the diffusivity of calcium in the porous media. In Eq. (1), the first term on the right-hand side stands for the diffusion process of the calcium in the liquid phase, which is assumed to be governed by Fick's law. The second term of the right-hand side of Eq. (1) accounts for the dissolution process, which leads to an arrival of calcium in the liquid phase. It can be noted that whatever the functions into Eq. (1) are (linear or not), the Boltzmann variable change is valid, so the degradation is proportional to the square root of the time as long as there is an unaltered zone and the aggressive solution composition is constant.

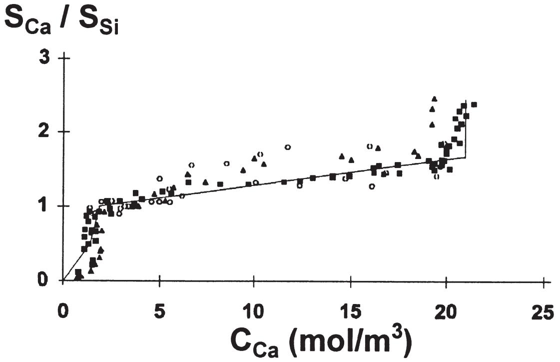


Fig. 3. Evolution of ratio S_{Ca}/S_{Si} in solid phase as a function of calcium concentration in solution C_{Ca}: experimental results compiled [9] in CaO-SiO$_2$-H$_2$O system and line used in the present model for cement-based materials.

However, this variable change will not be applied in what follows, so that we can modellize two-dimensional problems or problems with variable-limiting conditions. The numerical predictions will emphasise proportionality between degradation and square root of time.

Eq. (1) is a simplified representation of the leaching process since numerous ionic species interfere during the leaching of cement-based materials. Furthermore, the proposed model does not account for any precipitation that may also occur in the material [6–8]. However, it can be viewed as an initial approach that make a good prediction of the leaching process possible, in term of degraded depth and cumulative amount of leached calcium.

The equilibrium between the solid calcium phases (Portlandite CH and C-S-H) and the calcium concentration in pore solution is now well identified and leads to a relation between the calcium in the solid phase S_{Ca} and in the pore solution C_{Ca} [9] (see Fig. 3 where S_{Si} denotes the silicium concentration in the solid phase).

The evolution of S_{Ca}/S_{Si} ratio as a function of calcium concentration in solution (from the right to the left) roughly corresponds to the alteration from the core to the external zone of a cement material that contain mainly Portlandite and C-S-H, but also ettringite and monosulfoaluminate.

The dissolution of Portlandite occurs suddenly for a threshold calcium concentration of $\approx$21 mol/m^3 and explains the steep drop at this value. The decalcification of C-S-H is progressive due to the various forms of C-S-H having an S_{Ca}/S_{Si} ratio ranging from 1.65 to 1. This explains the decreasing calcium concentration in pore solution between 21 and 2 mol/m^3. A progressive dissolution of ettringite and monosulphoaluminate also occurs in that zone. For calcium concentrations under 2 mol/m^3, the solid phase corresponds to a silica gel.

3.1. Evolution of physical and chemical variables of the model DIFFU-Ca for cement paste

The initial values of S_{Ca} and S_{Si} (i.e., values for sound paste) were determined by chemical analysis. As the silicon concentration in solid phase is virtually constant, whatever the state of degradation, the relation between the calcium concentration in the solid phase S_{Ca} and in the pore solution C_{Ca} (or the reverse relation) follows from the equilibrium relation (see Fig. 4).

The porosity is directly linked to the mineralogy (type and proportions of the existing solid phases). The porosity created by C-S-H decalcification is extremely fine (less than 3 nm) and remains negligible compared with the already existing microporosity and capillary porosity. Furthermore, diffusivity in that kind of porosity is very low [10]. Therefore, the increase in porosity resulting from the decalcification of C-S-H is ignored in calculations. The porosity is thus calculated as a function of the calcium concentration by assuming a rapid dissolution of Portlandite, followed by a progressive dissolution of ettringite and monosulphoaluminate. The relations between porosity and calcium concentration in pore solution or in solid phase are given in Fig. 5.

The evolution of the effective diffusion coefficient (i.e., coefficient of global diffusion through porous material) is determined from experimental results obtained with a two-compartment diffusion cell on OPC cement pastes with different w/c ratios and different porosities (see Fig. 6). An empirical relationship between the effective diffusion coefficient and the porosity of sound materials was then proposed [11]. The extrapolation of this formula to high porosities relies on the fact that the diffusion coefficient of a

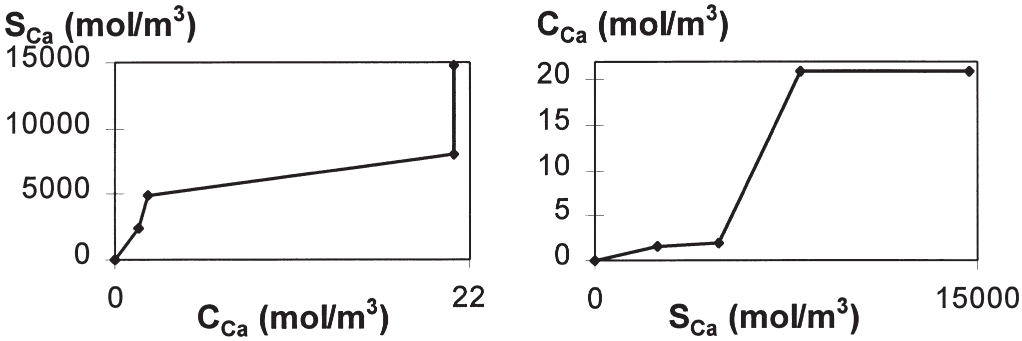


Fig. 4. Relation between calcium concentration in pore solution C_{Ca} and in solid phase S_{Ca}.

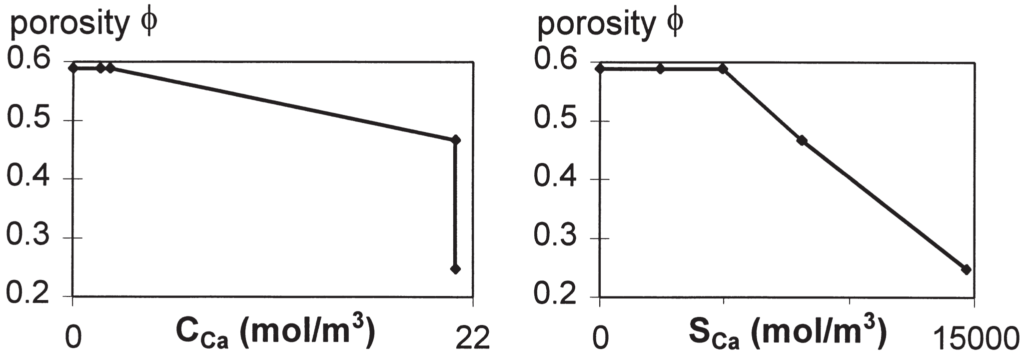


Fig. 5. Relation between porosity and calcium concentration in pore solution or in solid phase.

porous material cannot exceed that of pure water. Therefore, for $\phi < 0.92$, we have [see Eq. (2)]:

$$D_e = e^{(9.95\phi - 29.08)} \tag{2}$$

where D_e is given in m^2/s. In the other case, D_e is equal to the free diffusion coefficient of calcium in water (i.e., 2.2×10^{-9} m^2/s for a temperature of 20°C).

Fig. 7 shows the relations between effective diffusion coefficient and calcium concentrations deduced from Fig. 5 and Eq. (2), used for degraded materials.

3.2. Numerical resolution and comparison with the experiment

The choice of the main state variable for modelling the leaching process with Eq. (1) is of great importance. One approach is to solve Eq. (1) with the calcium concentration in the liquid phase (i.e., C_{Ca}) as proposed previously [3–5,11]. However, the dissolution of the Portlandite occurs at a fixed value of the calcium liquid concentration, so that the calcium concentration in the solid phase (i.e., S_{Ca}) is not a unique function of the calcium liquid concentration (see Fig. 4). On the contrary, the liquid calcium concentration can always be deduced from the solid calcium concentration. Thus, it appears more suitable to use the calcium concentration in the solid phase as the main state variable. Eq. (1) is numerically handled in the form [see Eq. (3)]:

$$\frac{\partial[\phi(S_{Ca}) \cdot C_{Ca}(S_{Ca})]}{\partial t} = \mathrm{Div}\{D_e(S_{Ca}) \cdot \mathrm{Grad}[C_{Ca}(S_{Ca})]\} - \frac{\partial S_{Ca}}{\partial t} \tag{3}$$

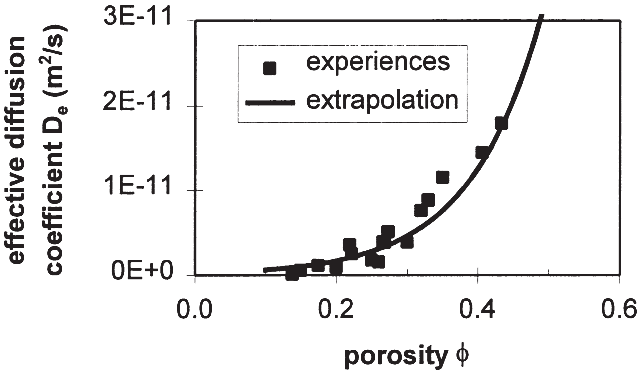


Fig. 6. Experimental results and extrapolation for the relation between effective diffusion coefficient and porosity.

In the unattacked region, calcium concentration in the liquid phase is constant and greater than or equal to the threshold one (21 mol/m^3) and the S_{Ca}/S_{Si} ratio is greater than or equal to 1.65. Thus, no diffusion of calcium occurs and local concentration gradient is null. Therefore, the mathematical problem is said to be degenerated. A sharp front associated with the dissolution of the Portlandite evolves at a finite velocity. Due to a high nonlinearity of variables, this dissolution front is difficult to capture numerically and requires a fine discretisation of the media. The numerical method used in this analysis is the finite volume (FV) method. This is a robust and simple method for approximating the nonlinear conservation equations and for precisely locating the dissolution front [12–14]. In these conditions, the FV method does not suffer from the problems that can occur when using the finite element method (divergence or oscillations) [15].

The leaching process of cylindrical samples immersed in pure water can be modelled with the one-dimensional expression of Eq. (3). The boundary condition corresponding to the aggressive water is expressed as $C_{Ca} = 0$ and a null diffusion flux of calcium is imposed on the other boundary since the Portlandite dissolution front evolves at finite velocity. The complete finite volume scheme used for solving Eq. (3) is given elsewhere [16]. Based on the data presented in Fig. 4 [i.e., $C_{Ca}(S_{Ca})$], Fig. 5 [i.e., $\phi(S_{Ca})$], and Fig. 6 [i.e., $D_e(\phi)$], this scheme allows the prediction of the degraded depth and the outgoing flux of leached calcium according to Fick's law. The numerical results are compared with experimental data below.

Numerical and experimental values of degraded depths after 3 and 6 months of leaching are compared in Table 2. Globally, the predicted values given by the finite volume method are very close to experimental data even though

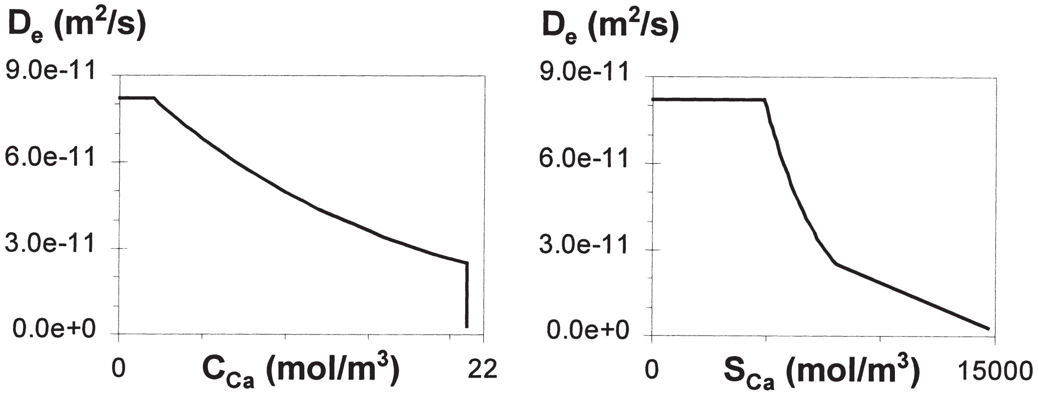


Fig. 7. Relation between effective diffusion coefficient and calcium concentration in pore solution or in solid phase.

they slightly underestimate the experimental degraded depths.

Fig. 8 shows the experimental [1] and the predictive cumulative quantity of leached calcium by the sample as a function of the square root of time. The numerical prediction is derived from the discretisation of Fick's law at the aggressive boundary of the sample using the finite volume approximation.

The experimental and numerical results presented in Fig. 8 are very close, though the numerical prediction slightly overestimates the experimental results.

The quality of the numerical prediction seems sensitive to the variables of the model [i.e., $C_{Ca}(S_{Ca})$, $\phi(S_{Ca})$ and $D_e(\phi)$]. Thus, the influence of these parameters on the results of the model is studied in the following section.

3.3. Sensibility of the modelling to the material properties

This section analyses the influence of the material properties on the modelling of the leaching process using Eq. (1). Four parameters were selected to study the sensitivity of the numerical results because significant uncertainties exist on the reference values chosen in the previous parts:

parameter 1: the threshold calcium concentration in liquid phase [i.e., the calcium concentration for which the Portlandite dissolves, noted as C_{Ca} (Portlandite)];

parameter 2: total porosity according to the solid calcium concentration, noted as $\phi(S_{Ca})$;

parameter 3: the diffusion coefficient according to the total porosity, noted as $D_e(\phi)$; and

parameter 4: the initial value of the solid calcium concentration, noted as S_{Ca} (initial).

Table 2
Comparison between experimental and predicted degraded depths

	3 months (mm)	6 months (mm)
Experiment	1.45	2.1
Finite volume prediction	1.4	2.0

The threshold calcium concentration in the liquid phase can vary with the ionic force of the solution: it is higher if ionic force increases. It also varies with the presence of alkali in solution; in this case, it is less important. Lastly, it depends on the temperature that makes variations in the two directions. For parameter 2 or 4, uncertainties come from the assumption chosen to estimate the quantity of calcium in the various solid phases. For example, all calcium is assumed to be contained on Portlandite, C-S-H, ettringite, and monosulfoaluminate of calcium, and the proportion of calcium under an inert phase is ignored [1]. Moreover, for parameter 2, additional uncertainties exist because of the assumptions made on the description of chemical degradation (dissolution of ettringite and monosulfoaluminate of calcium and nondissolution of C-S-H). Lastly, parameter 3 comes from interpolation of experimental data with extrapolation for high porosities, which create a strong uncertainty on the final value.

The reference values were given in Figs. 4, 5, and 6 with 21 mol/m³ for the threshold calcium concentration in the liquid phase, and 14.7×10^3 mol/m³ for the initial calcium concentration in the solid phase. A 10% uncertainty is assumed on these data. The effects of these parameters are

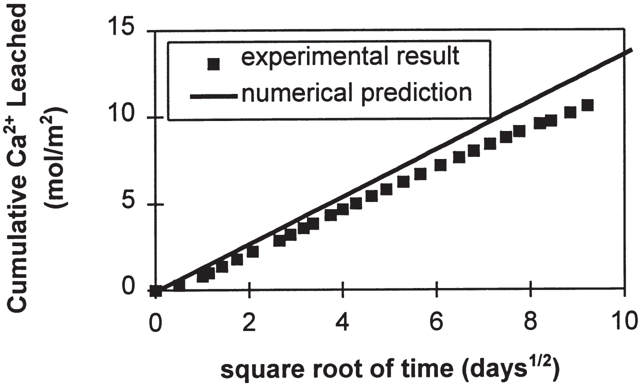


Fig. 8. Comparison between experimental and predicted cumulative quantity of leached calcium for cement paste.

Table 3
Comparison between experimental and predicted degraded depths

Parameter	Experiment (mm)	Finite volume prediction (mm) With reference value	With reference value −10%	Variation with reference value −10%	With reference value +10%	Variation with reference value +10%
1: C_{Ca} (Portlandite)	2.1	2.03	1.92	−5.4%	−2.12	+4.4%
2: $\phi(S_{Ca})$			1.55	−23.6%	−2.66	+31.0%
3: D_e (ϕ)			1.92	−5.4%	−2.12	+4.4%
4: S_{Ca} (initial)			2.24	+9.1%	+1.87	−7.9%

studied independently, which means that only one parameter changes at a time and the three others remain fixed. The effect of these parameters is observed on the degraded depth at 6 months and on the cumulative amount of leached calcium at 85 days. The results of these variations are presented in Table 3 for degraded depth and in Table 4 for leached quantity.

The results in Tables 3 and 4 show that parameters 1, 3, and 4 have a small influence on the results of the mathematical model. In fact, variations of 10% of parameter 1, 3, or 4 induce variations of the numerical prediction lower than 10%, around the prediction obtained with the reference parameters. The variations are even lower than 5.5% for parameters 1 and 3. However, parameter 4 plays an important role in the results. Unlike others parameters, it has distinct effects on the degraded depth and on the cumulative leached calcium. The most important parameter is the expression of the porosity as a function of the solid calcium concentration (parameter 2). This can be easily explained by the fact that the diffusion coefficient is an exponential function of the porosity. Consequently, a variation of 10% in porosity induces a large variation in the diffusion coefficient.

The discrepancy observed between the experimental data and the numerical prediction of the degraded depth and of the leached fluxes can be explain by an over estimation of the initial value of the calcium concentration in solid phase (parameter 4). In fact, reducing the initial value of the solid calcium concentration will lead to a lower outgoing quantity of calcium and a faster advance of the dissolution front of Portlandite since there is a lower amount of calcium to be dissolved.

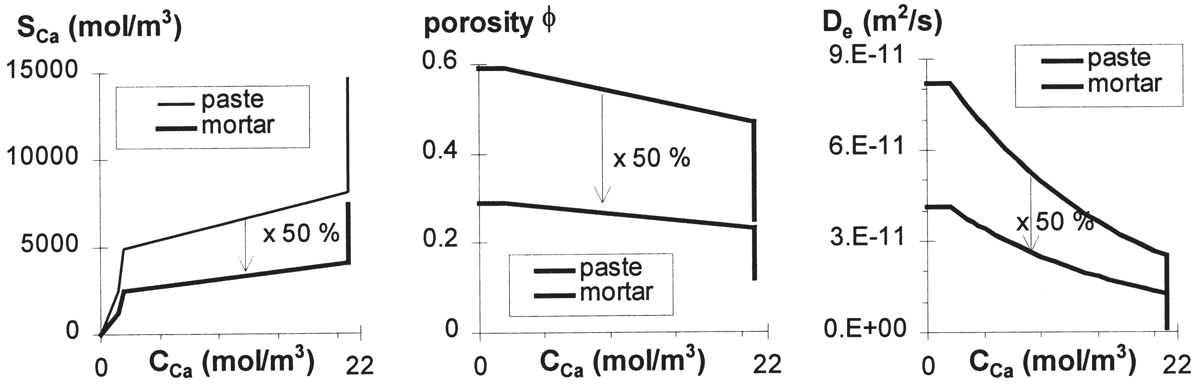
In any case, as the parameters of the model have been determined totally independently of degradation experiments, the numerical simulation appears to be in good accordance with experimental results. Therefore, the engineer can use the numerical resolution of Eq. (1), including the evolution of physical and chemical variables of the model for cement paste (subsection 3.1) as a predictive tool for the modelling of leaching. In the next section, the model of the leaching process of a mortar is extended from the model developed for paste.

4. Modelling of mortar leaching

As for pure cement paste, modelling the chemical degradation of mortar consists of resolving the mass balance equation for calcium only. Experimental results show that the kinetics of mortar degradation can be deduced from that of pure paste: evolutions of degraded depth of paste and mortar are very close, and the leached calcium fluxes are proportional to the paste content in the material [2] (see Fig. 2). For mortars, evolution of physical and chemical variables for the resolution of the mass balance Eq. (1) are then deduced from that of the pure cement paste using the proportion of paste as a dilution factor. Fig. 9 shows data for a pure paste and a mortar containing 50% paste. For other proportions of cement paste, it would be useful to adjust the reducing factor to take into account the tortuosity due to sand and the influence of the ITZ with higher diffusivity, using previous experimental results [17] or modelled calculations [18]. However, using the proportion of paste as a dilution factor appears to be valid. It gives good data for sound mortar porosity (see previous experimental data [2,11]), and also for the effective diffusion coefficient (a value of 1.4×10^{-12} m^2/s is determined using the reducing factor to be compared to 1.7×10^{-12} m^2/s measured experimentally on mortar [11]). In the following text, the comparison between experimental data and predicted results for mortar degradation will show the validity of the model extended from paste to mortar.

Table 4
Comparison between experimental and predicted cumulative quantity of leached calcium

Parameter	Experiment (mol/m^2)	Finite volume prediction (mol/m^2) With reference value	With reference value −10%	Variation with reference value −10%	With reference value +10%	Variation with reference value +10%
1: C_{Ca} (Portlandite)	10.62	12.53	11.98	−4.4%	13.05	+4.2%
2: $\phi(S_{Ca})$			9.49	−24.3%	16.54	+32.0%
3: D_e (ϕ)			11.88	−5.2%	13.14	+4.9%
4: S_{Ca} (initial)			11.58	−7.6%	13.41	+7.0%

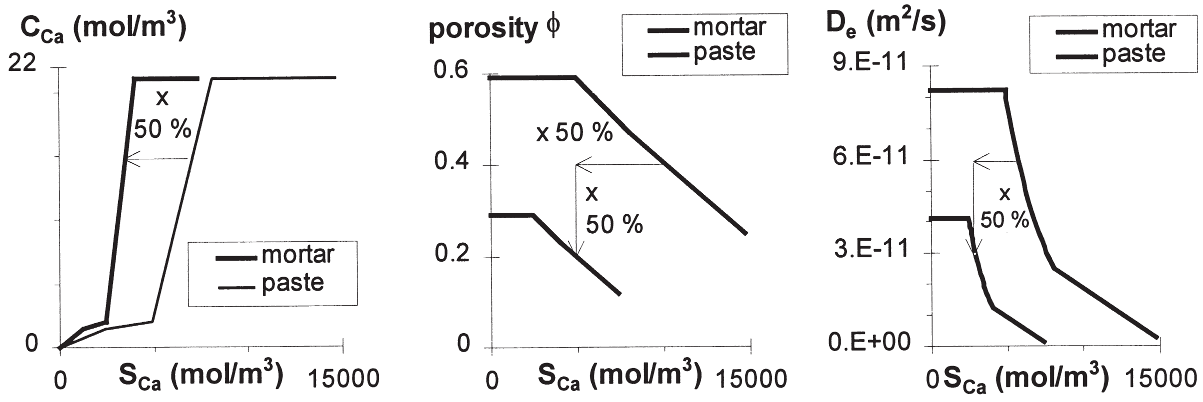


for calcium concentration in solid phase

for porosity

for diffusion coefficient

a) Evolutions in function of calcium concentration in pore solution C_{Ca}



for calcium concentration in solid phase

for porosity

for diffusion coefficient

b) Evolutions in function of calcium concentration in solid phase S_{Ca}

Fig. 9. Data for modelling.

The same program used previously has been used to obtain the predicted degraded depth and the cumulative quantity of leached calcium as a function of time. The material properties of this modelling are given in Fig. 9. After 7 months of chemical attack, the predicted degraded depth (2.18 mm) and the experimental depth (2 mm) are very close. The experimental and numerical results of cumulative quantity of leached calcium are presented in Fig. 10. Again, the experimental and numerical prediction are in good agreement. As shown in Fig. 8, the numerical prediction slightly overestimates the experimental results.

The extension of the modelling from cement pastes to mortars appears to be valid since the experimental results for mortar samples have been accurately predicted. This confirms the validity of the simple diffusion Eq. (1) used to describe and predict the leaching of cement-based material. This equation can be applied to different media, using the proportion of paste and data from the same pure cement paste. These results suggest that the modelling of the leaching process of a concrete sample should be possible by using a similar approach.

5. Conclusions

To predict the long-term evolution of concrete used for waste disposals in aggressive conditions, a modelling of the calcium leaching in cementitious materials has been presented. This modelling is based on a single equation accounting for the mass balance equation of calcium in the

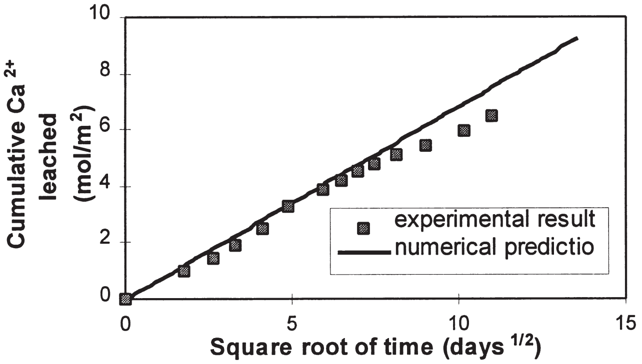


Fig. 10. Comparison between experimental and predicted cumulative quantity of leached calcium for mortar.

liquid phase. Its resolution with a numerical method requires the knowledge of the equilibrium relation between the solid and the liquid phase, the evolution of the porosity with the dissolution process, and the variation of the effective diffusion coefficient with the porosity.

Such parameters have been presented for a cement paste. Then, the finite volumes method has been used for the numerical resolution with the calcium concentration in solid phase S_{Ca} as the main variable. Due to its conservativity properties, this method supports high nonlinearities and ensures a good positioning of the dissolution front of Portlandite. Comparisons between experimental and numerical results in terms of degraded depth and leached calcium fluxes give the value of simple modelling in predicting the leaching phenomenon with time. Furthermore, it has been noted that the evolution of the porosity with the calcium concentration plays the most important role in the accuracy of the modelling. As the parameters of the model have been determined totally independently of degradation experiments and as numerical simulations are in good accordance with experimental results, the model appears to be of worth.

Finally, it has been extended with success to the leaching of a mortar using the proportion of paste in mortar as a reducing factor to determine data for the mortar from those for the paste. The modelling of the leaching process of concrete should be possible by using a similar approach.

Now, the model DIFFU-Ca may be used to analyse the effects of cracking on the degradation process by studying two-dimensional problems [11] or to predict the degradation of cementitious materials attacked by aggressive solution varying with time [5].

Acknowledgments

The present investigation was carried out with financial support from the French Agence Nationale pour la Gestion des Déchets Radiacatifs (ANDRA) and Electricité de France (EDF) in a GEO project [19].

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