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Modelling the leaching kinetics of cement-based materials—influence of materials and environment

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Abstract

The leaching of cement-based materials in aggressive water is studied according to several protocols of accelerated or non-accelerated tests. The paper analyses international experimental published data on leaching concerned with several parameters, like material parameters as water-to-binder ratio and silica fume content, and environmental parameters, like pH and temperature. Then, a simplified model based on this analysis is developed to predict the kinetics of leaching. This model assumes that the rate of leaching follows a square root of time. Each parameter is introduced as a weight function. Particularly, the paper introduces the coupling between the built database and the model.
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1. Introduction

Leaching of concrete by water may severely damage structures like dams or radioactive waste depositories. The kinetics of leaching is very slow but can be a risk at long term.

When a cement-based material is in contact with water, ionic transfers occur between the external water and the pore solution of the material. These transfers modify the chemical equilibrium of the medium and more particularly that of hydrates. First the equilibrium is restored as long as dissolution and/or precipitation of hydrates are possible [1–5]. However, the porosity in degraded zones increases and, consequently the physicochemical [3–6,9–13] and mechanical performances [3–8,10,14–17] decrease. A succession of dissolution fronts is characteristic of the chemical degradation. The portlandite (CH) is the first mineral to be dissolved, then there is a progressive decalcification of C–S–H. The propagation kinetics of the front, also called kinetics of leaching, follows a square root of time [1–5,11,13].

It is clear, according to leaching mechanisms, that the kinetics is closely related to the characteristics of cement-based materials and environmental conditions as well. Indeed, the greatest the porosity, temperature and gradient of pH between the pore solution and aggressive water, the more accelerated is the ionic diffusion. This results in higher leaching kinetics.

Several researchers have modelled the leaching kinetics. Adenot [2] has proposed a one-dimensional model based on the resolution of diffusional transport equations and local chemical equilibrium. The model taking into account the main solid phases and main pore solution ions was simplified by Gérard [4] in order to develop a three dimensional one, coupled with mechanical damage. This new model consists of using the calcium concentration in pore solution as the unique state variable that determines the evolution of the degradation. Gérard [4] resolved this equation numerically by using finite element software. Mainguy et al. [18] then proposed a numerical solution by using finite volume method.

In this paper, a different approach to model the leaching kinetics is proposed. The new approach is a practical and simplified one-dimensional model based on a laboratory data bank. This database is built with the published experimental data dealing with the effect of material parameters and environmental conditions on the kinetics of leaching. The model assumes that the rate of leaching follows a square root of time. Each

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parameter is introduced as a weight function. Firstly, the database is presented. Secondly, the simplified model of leaching is proposed. Then a coupling between the built database and simplified model is developed. The leaching kinetics of various materials in different environmental conditions was studied during the last decade. However, in the absence of a standardised test, several laboratory protocols were developed which include accelerated and non-accelerated tests. In this paper, the non-accelerated tests correspond to degradations due to a controlled pH of the aggressive water [1–3,5]. The accelerated tests are either a forced convection through an electric field [4,19–22] or a water pressure [23], or a very reactive media such as an ammonium nitrate solution [3,7,8,10,17].

2. Parameters influencing the leaching kinetics of cement-based materials

The kinetics of diffusion–dissolution mechanisms during leaching is closely related to the characteristics of the material and environmental conditions. It is influenced by several parameters. However, this study is restricted to the influence of the following parameters:

- water-to-binder ratio (w/b) of the cement-based material,
- addition of supplementary minerals, more particularly the addition of silica fume,
- pH of the aggressive water,
- temperature,
- test protocol.

2.1. Water-to-binder ratio and leaching kinetics

The kinetics of leaching increases with the ionic diffusivity through the material. This ionic diffusivity increases with the capillary porosity of the material, which itself increases with the water-to-binder ratio. Fig. 1a introduces experimental leached depths L_d of cement-based materials versus w/b according to several authors [3,4,11,19]. Four sets are presented. Each set corresponds to the same binder at various w/b and in given conditions at the same leaching time. The different sets show that the leaching kinetics increases with the w/b.

2.2. Cement addition and leaching kinetics

The additions to cement play a significant role in the improvement in the rheology, mechanical behaviour and resistance to leaching. The addition of silica fume or fly ash to cement decreases capillary porosity. By pozzolanic effect, these additions decrease portlandite content and form denser and more stable C–S–H than the portland C–S–H. Thus, the ionic diffusion and dissolution of hydrates are slowed down. In this study, only the effect of the silica fume is considered. Fig. 1b shows experimental leached depth versus percentage of silica fume content according to several authors [11,13,22]. Four sets are illustrated. Each set corresponds to a material with various silica fume content, in given conditions and at the same leaching time like sets 1, 2, 3 or at different leaching times like set 4. For each set, only the given data corresponding to the same leaching time can be compared. According to set 1, set 2 and set 3 data, the leaching kinetics decreases with increasing silica fume content.

2.3. Aggressive water pH and leaching kinetics

The pH of the pore solution of traditional cement-based materials is about 13. When these materials are in contact with water with pH lower than 13, hydroxyl ions diffuse towards aggressive water under gradient of concentration. The lower the pH of the aggressive water and higher the concentration gradient, the diffusion of hydroxyl ions will be significant. The solid hydrates are more or less soluble, consequently, the lower the pH, the more significant the dissolution of hydrates. Fig. 1c presents published experimental leached depths versus pH of aggressive water [11,24,25]. Three sets are presented. Each set corresponds to different samples of a given cement paste, in water at different pH and at different leaching times. Only the given data at the same leaching time can be compared.

2.4. Temperature and leaching kinetics

Temperature accelerates diffusion and dissolution kinetics. This rule is often true but cannot be generalized. Portlandite belongs to the rare products of decreasing solubility with increasing temperature. Temperature also influences the stability of other hydrates, such as primary ettringite. This trisulfoaluminate disappears at temperatures higher than 70 °C and can recrystallise into expansive secondary ettringite when returning to room temperature [26]. The effect of temperature on the leaching kinetics is not largely studied. Fig. 1d presents the experimental leached depths, according to different temperatures, measured by Prené et al. [27]. These experimental data correspond to four different samples of a given cement paste. These samples were immersed in pure water with a flow of 1 l per day in controlled temperature media. The leaching times are different for the four samples. Fig. 1d shows that the leaching kinetics is influenced by the temperature, being higher at higher temperatures.

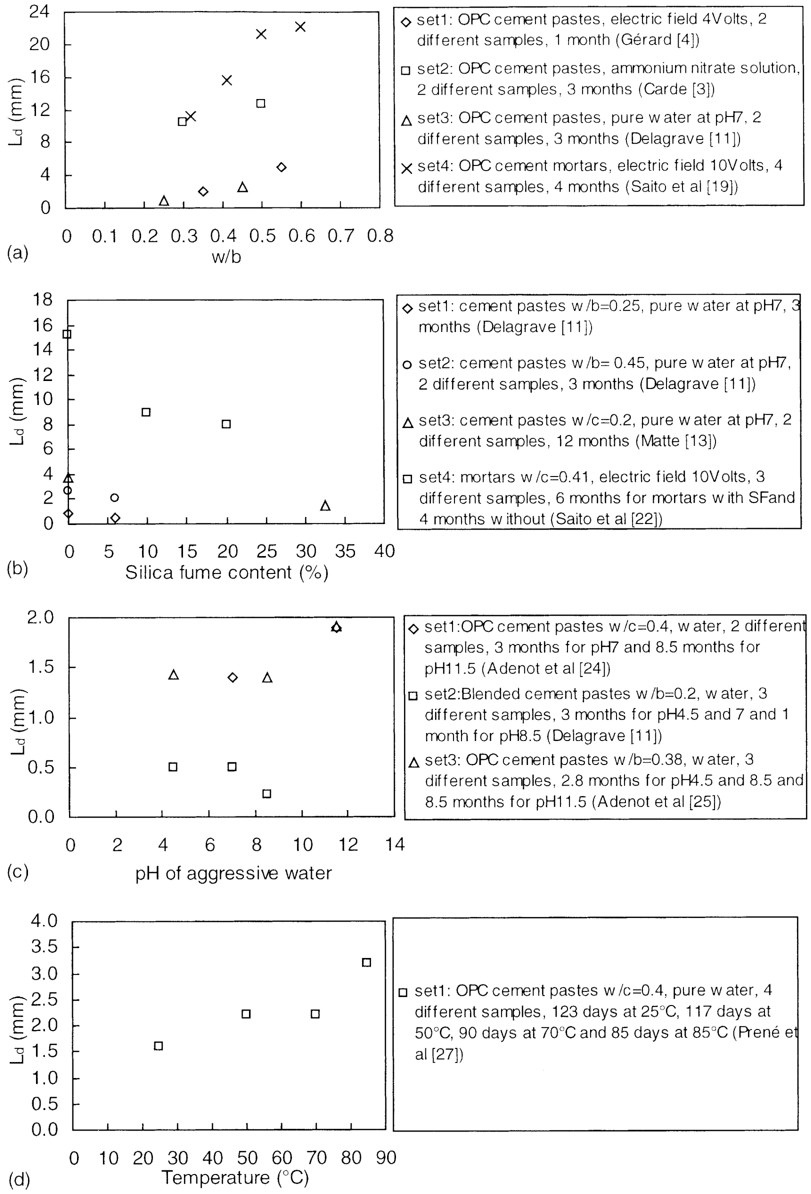

Fig. 1. (a) Experimental leached depth L_d versus w/b; (b) experimental leached depth L_d versus silica fume content (%); (c) experimental leached depth L_d versus pH of aggressive water; (d) experimental leached depth L_d versus temperature.

3. Simplified model for predicting leaching kinetics

A simplified model is proposed to predict the evolution of the leached depth with time as a function of material parameters (w/b, silica fume content) and environmental conditions (temperature, pH of aggressive solution, test protocol). The leached depth L_d in function of time is expressed as follows:

$$L_d = a \times t^{1/n} \tag{1}$$

with L_d (mm), t (day), a: constant parameter, $n = 2$ for degradation in absence of electric field and $n = 1$ in presence of electric field.

The parameter a is modelled as a product of weight independent functions f, g, h, i and j. Each function corresponds to the influence of one of the various parameters quoted above. The proposed expression of a is

$$a = e \times f(w/b) \times g(\mathrm{SF}) \times h(\mathrm{pH}) \times i(T) \times j(\mathrm{Prot}) \tag{2}$$

with e: constant, w/b: water-to-binder ratio, SF: percentage of silica fume content, pH: pH of aggressive water, T: temperature in °C, Prot: test protocol.

The various functions f, g, h and i are built using the experimental database from the literature previously presented.

In order to build the weight functions, a protocol of reference and a reference material are defined. In these reference conditions, the value of the various weight functions is equal to 1 (Eq. (3)). Thus, a is equal to e.

$$f((w/b)_{\text{reference}}) = 1 \quad g(\text{SF}_{\text{reference}}) = 1 \quad h(\text{pH}_{\text{reference}}) = 1$$
$$i(\text{T}_{\text{reference}}) = 1 \quad j(\text{Prot}_{\text{reference}}) = 1 \tag{3}$$

The procedure used by Adenot [2] was chosen as the reference protocol. This protocol is severe but remains relatively close to reality compared to other protocols found in the literature. Initial deionized water is maintained at pH 7 and 20 °C. The selected reference material is an OPC cement based paste with water-to-cement ratio w/c equal to 0.4. It is a traditional material that was studied by several authors in France. The leached depth of this material after three months of degradation by using the protocol of reference is of 1.4 mm. According to Eqs. (1) and (2), the parameter a is equal to the constant e itself equal to 0.147.

3.1. Modelling the influence of water-to-binder ratio

The construction of the function f is made by using the published experimental data of leached depths L_d. Fig. 1a contains four sets of data. In each set, only the parameter w/b varies. The procedure followed consists of recording, for each set, the L_d value at the point of reference, in this case $w/b = 0.4$. If for a set of data, this value is not measured, it is estimated using the other data. The set 4 of Fig. 1a shows that L_d versus w/b can be estimated as a linear regression. Consequently, the estimation of the degraded depth, at the point of reference $w/b = 0.4$ as per linear interpolation between the other available values, is correct. Then for a given set of data L_d is divided by corresponding reference L_d. Indeed, the L_d-to-reference L_d ratios make it possible to build the function f according to the following equation:

$$\frac{L_d(w/b)}{L_d(w/b=0.4)} = \frac{e \times f(w/b) \times g(\text{SF}) \times h(\text{pH}) \times i(T) \times j(\text{Prot}) \times t^{1/n}}{e \times f(w/b=0.4) \times g(\text{SF}) \times h(\text{pH}) \times i(T) \times j(\text{Prot}) \times t^{1/n}} = f(w/b) \tag{4}$$

The data of L_d-to-reference L_d built using the various experimental points according to the w/b of the Fig. 2 makes it possible to model the function f. The expression suggested for this function is the following one:

$$f(w/b) = 3.24 \times \frac{w}{b} - 0.296 \tag{5}$$

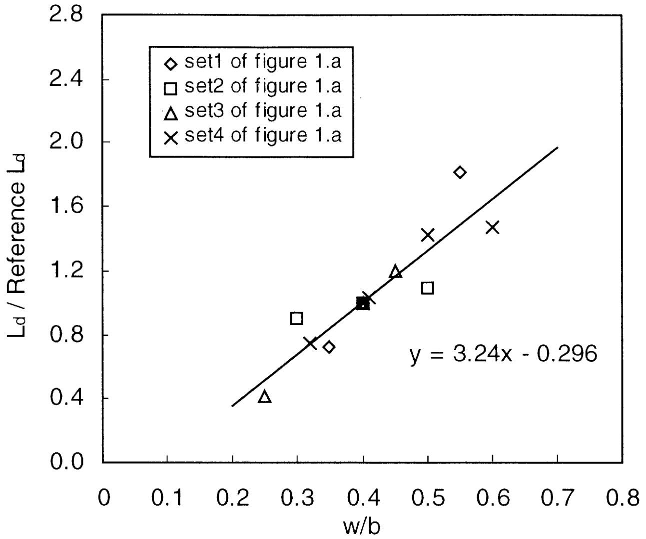

Fig. 2. L_d/Reference L_d versus w/b: function f.

3.2. Modelling the influence of silica fume content

The function g is built in the same way as the function f. In this case, the point of reference corresponds to SF = 0%. However, a preliminary stage is to be added. The leached depths of Fig. 1b (set 4) are given at different times of degradation contrary to the experimental data corresponding to the function f. However, to determine g (Eq. (6)), it is necessary to have, for each set of data, the L_d corresponding to the same duration of degradation. However, the degraded thickness L_d in a given time t_1 is easily calculable from the one at time t_0. When the degradation is due to water that is either more or less acidic, or to an ammonium nitrate solution, the kinetics of leaching is proportional to the square root of time. Consequently, L_d is calculated from Eq. (7). However, when degradation is accelerated by an electric field, the kinetics of leaching is proportional to time [19,22]. In this case, L_d is calculated according to Eq. (8).

$$\frac{L_d(\text{SF})}{L_d(\text{SF}=0\%)} = \frac{e \times f(w/b) \times g(\text{SF}) \times h(\text{pH}) \times i(T) \times j(\text{Prot}) \times t^{1/n}}{e \times f(w/b) \times g(\text{SF}=0\%) \times h(\text{pH}) \times i(T) \times j(\text{Prot}) \times t^{1/n}} = g(\text{SF}) \tag{6}$$

$$L_d(t_1) = \frac{L_d(t_0)}{t_0^{1/2}} \times t_1^{1/2} \tag{7}$$

$$L_d(t_1) = \frac{L_d(t_0)}{t_0} \times t_1 \tag{8}$$

Then, L_d of each set of data and for the same duration of degradation is divided by corresponding reference L_d. Fig. 3 presents the values of the L_d-to-reference L_d ratios calculated from the experimental data and according to the silica fume content. Thus this group of points is able to define a modelling of the function g as the following one:

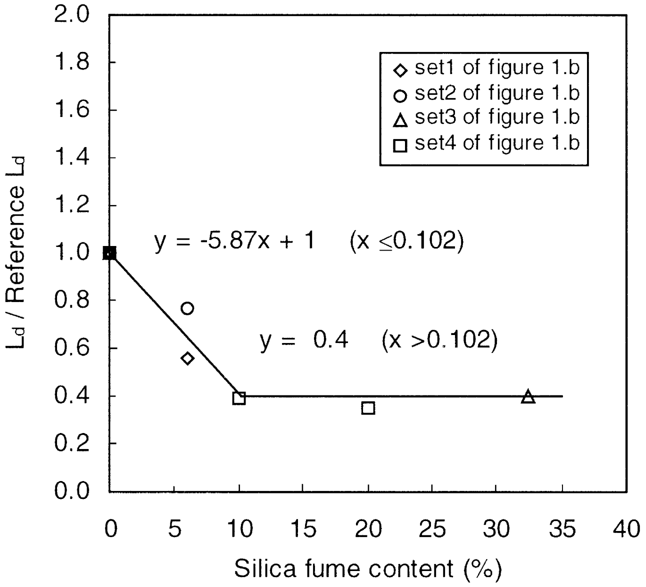

Fig. 3. L_d/Reference L_d versus silica fume content (%): function *g*.

$$g(\mathrm{SF}) = \begin{cases} -5.87 \times \mathrm{SF} + 1 & \text{if } \mathrm{SF} \leqslant 10.2\% \\ 0.4 & \text{if } \mathrm{SF} > 10.2\% \end{cases} \tag{9}$$

3.3. Modelling the influence of the pH of the aggressive water

In order to determine the function *h*, the experimental data of Fig. 1c are used. This function is built in the same way as the function *g*. In this case, the reference L_d corresponds to L_d with pH equal to 7. Fig. 4, presents the L_d-to-reference L_d ratio and generating the function *h* for pH going up to 12.4. For values higher than 12.4, the hydrates are stable and there is no degradation. Then, the function *h* can be modelled as follows:

$$h(\mathrm{pH}) = \begin{cases} -0.045 \times \mathrm{pH} + 1.311 & \text{if } \mathrm{pH} \leqslant 12.4 \\ 0 & \text{if } \mathrm{pH} > 12.4 \end{cases} \tag{10}$$

3.4. Modelling the influence of temperature

Function *i* is built from Fig. 1d using the same way as for the functions *g* and *h*. The value of reference L_d, L_d corresponding to the temperature 20 °C, is estimated by linear extrapolation, using the other available data of Fig. 1d. The values of L_d-to-reference L_d ratio make it possible to determine function *i* according to the following equation:

$$\frac{L_d(T)}{L_d(T = 20\ ^\circ\mathrm{C})} = \frac{e \times f(w/b) \times g(\mathrm{SF}) \times h(\mathrm{pH}) \times i(T) \times j(\mathrm{Prot}) \times t^{1/2}}{e \times f(w/b) \times g(\mathrm{SF}) \times h(\mathrm{pH}) \times i(T = 20\ ^\circ\mathrm{C}) \times j(\mathrm{Prot}) \times t^{1/2}} = i(T) \tag{11}$$

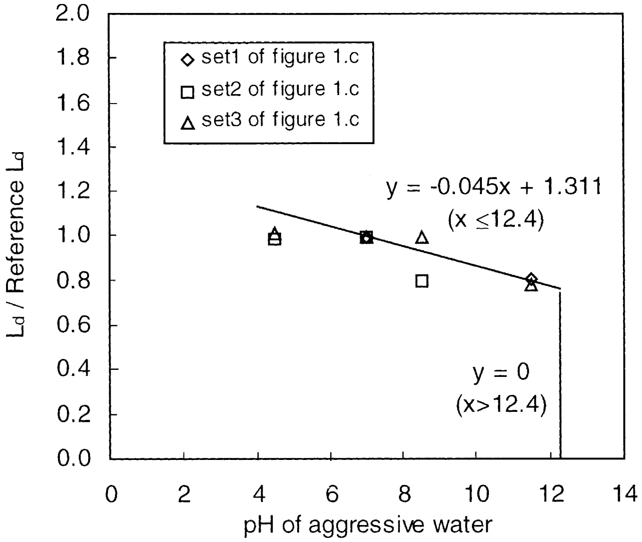

Fig. 4. L_d/Reference L_d versus pH of aggressive water: function *h*.

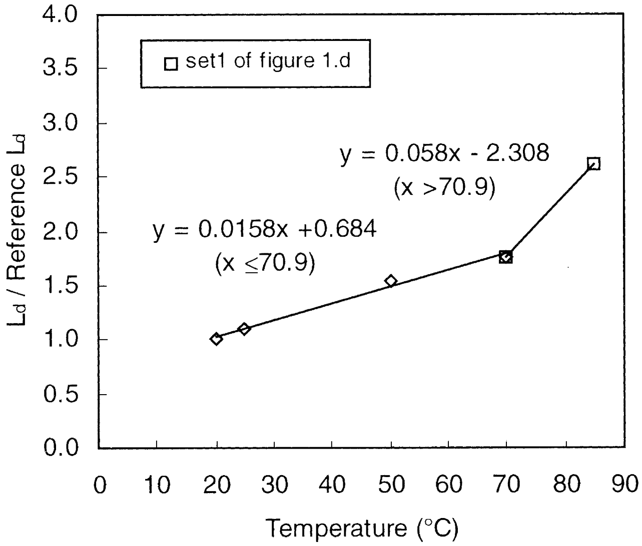

Fig. 5. L_d/Reference L_d versus temperature: function *i*.

Fig. 5, presents the values of L_d-to-reference L_d versus the temperature. According to the shape of this group of points, a linear extrapolation for the determination of L_d at 20 °C is correct. Function *i* can be modelled by two lines as follows:

$$i(T) = \begin{cases} 0.016 \times T + 0.684 & \text{if } T \leqslant 70.9 \\ 0.058 \times T - 2.308 & \text{if } T > 70.9 \end{cases} \tag{12}$$

3.5. Modelling the influence of the protocol test

The aim of this part is to model the influence of three protocol tests on the kinetics of leaching. These protocols were used by Tognazzi [28] for a comparative study:

1. Protocol of reference [2,28].
2. Prot2: a protocol of degradation accelerated by an ammonium nitrate solution at 6 mol/l at 20 °C with a variable pH between 4.5 and 8.5 during the test. The aggressive solution is agitated and renewed every day.
3. Prot3: a protocol of degradation accelerated by the application of an electric field at 5 V. Water is slightly basic with a pH around 9.2.

Table 1
Experimental leached depth L_d using three various protocols [28]

Protocol	Time of degradation (days)	Leached depth (mm)
Prot of reference	210	2
Prot2	4	4
Prot3	21	2.5

Table 1 shows the leached depths L_d of a mortar containing OPC with w/b 0.4, degraded 210 days according to the protocol of reference, four days according to Prot2 and 21 days according to Prot3. Knowing the expression of the leaching kinetics and according to these three protocols, the function j can be modelled in the following form:

$$j(\text{Prot}) = \begin{cases} 1 & \text{if Prot} = \text{Protocol of reference} \\ m & \text{if Prot} = \text{Prot2} \\ k & \text{if Prot} = \text{Prot3} \end{cases} \tag{13}$$

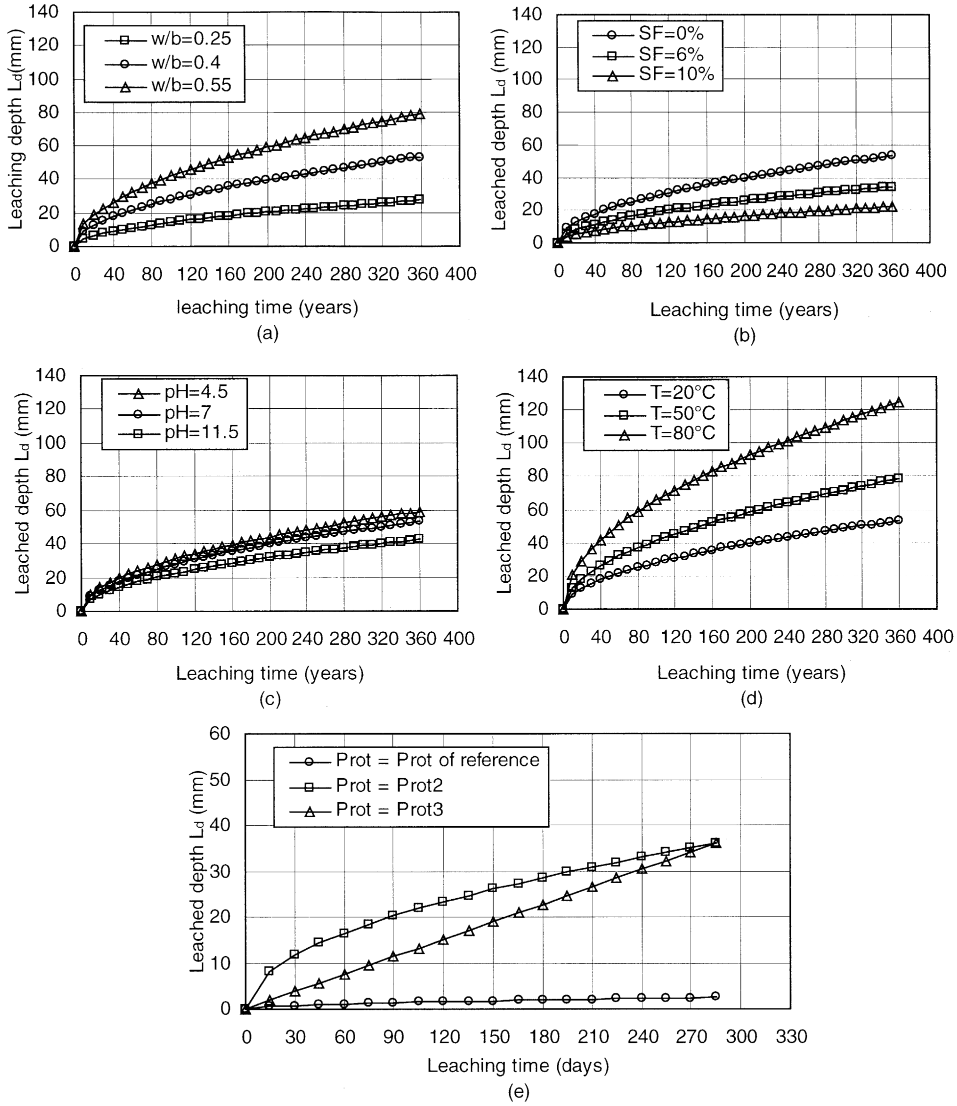

Fig. 6. Applications of the model: (a) leaching kinetics of OPC cement pastes with various w/b at 20 °C in a pure water pH7, (b) leaching kinetics of cement pastes with various silica fume contents at 20 °C in a pure water pH7, (c) leaching kinetics of OPC cement pastes with $w/b = 0.4$ at 20 °C in a water at various pH, (d) leaching kinetics of OPC cement pastes with $w/b = 0.4$ in a pure water pH7 at various temperature, (e) leaching kinetics of OPC cement pastes with $w/b = 0.4$ according to three protocols of degradation: Prot of reference, Prot2 and Prot3.

To determine the values of constants m and k, it is necessary to consider various degraded depths of Table 1 at the same time of degradation t.

The same methodology used for the construction of the function h is still applied. The found values are then divided by the value of L_d corresponding to the protocol of reference and according to the Eqs. (14) and (15):

$$\frac{L_d(\text{Prot2})}{L_d(\text{Prot of reference})} = \frac{e \times f(w/b) \times g(\text{SF}) \times h(\text{pH}) \times i(T) \times j(\text{Prot2}) \times t^{1/2}}{e \times f(w/b) \times g(\text{SF}) \times h(\text{pH}) \times i(T) \times j(\text{Prot of reference}) \times t^{1/2}} = m \qquad (14)$$

$$\frac{L_d(\text{Prot3})}{L_d(\text{Prot of reference})} = \frac{e \times f(w/b) \times g(\text{SF}) \times h(\text{pH}) \times i(T) \times j(\text{Prot3}) \times t}{e \times f(w/b) \times g(\text{SF}) \times h(\text{pH}) \times i(T) \times j(\text{Prot of reference}) \times t^{1/2}} = k \times \sqrt{t} \qquad (15)$$

According to Table 1 and Eqs. (14) and (15), the values of m and k are

$$m = 14.49 \quad \text{and} \quad k = 0.87$$

4. Discussion

The low dispersion of the results in Fig. 2 confirms the assumption of independence of the function f on the protocol of test used. According to this figure, the w/b ratio influences appreciably the kinetics of leaching. As an example, the leached depth of a cement paste with w/b equal to 0.6 is 2.5 times more significant than the one of a cement paste with w/b equal to 0.3.

The modelling of the influence of the silica fume content in concrete highlights and confirms the role of this addition in the improvement in the resistance of cement based materials to leaching [29]. According to the Fig. 3, there is a silica fume content threshold from which the improvement in the resistance to leaching is not significant. For silica fume addition until approximately 10.2%, the kinetics of leaching decreases appreciably. For example, the leached depth of a cement paste without silica fume is two times higher than the one with 10.2% of silica fume. However, for silica fume additions higher than 10.2%, the resistance to the leaching phenomenon remains unchanged.

Fig. 4 shows that the lower the pH of aggressive water, the higher the kinetics of leaching. However, the influence of the pH is less significant than the environmental temperature. Fig. 5 shows that the evolution of this kinetics is very sensitive to the temperature. The intersection of the two lines modelling the influence of the temperature corresponds to $T = 70.9$ °C. The variation of the slope of the second line corresponds to the transformation of cement hydrates (C–S–H, ettringite) resulting in a larger porosity and a higher diffusivity of aggressive ions. The coefficient of ions diffusivity changes dramatically at this temperature.

Five applications of the model are presented in Fig. 6 in order to highlight the influence of each parameter on the leaching kinetics of a cement paste studied in this paper. For example, Fig. 6a shows the influence of the water-to-binder ratio. Three w/b ratios are considered and the functions g, h, i and j are taken equal to 1 and the constant e remains equal to 0.147. Thus only the functions f (Eq. (2)) varies with the w/b ratios (Eq. (5)). For $w/b = 0.55, 0.40, 0.25$, the value of a (Eq. (1)) is, respectively, equal to 0.218, 0.147 and 0.076.

5. Conclusions

A model is developed by using experimental data from literature. It can predict the leaching kinetics of cement pastes according to material and environmental parameters. This kinetics is predicted according to three protocols of test: a severe protocol but relatively near to reality and two accelerated degradation protocols.

For the design of durable concrete structures, this model highlights the qualitative and quantitative influence of the water-to-binder ratio, silica fume, pH of the aggressive water, and temperature on the concrete resistance to leaching. This influence being very significant, it is necessary to take into account these parameters.

This model continues to be refined taking into account new data. Additional databases will be added to this model, considering the effects of other factors, such as fly ash or blast furnace slag addition to cement, sulphate and chloride content of the aggressive water and carbonation on long-term performance of the cement-based materials. A major step will be the coupling of the database with more physical models to increase the level of prediction.

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