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Cement and Concrete Research 37 (2007) 1145 – 1151
Cement and Concrete Research

Innovative study of non-steady-state tritiated water diffusion test

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Received 22 June 2005; accepted 12 February 2007

Abstract

The objective of this paper is to propose a new method for testing molecular diffusion in saturated cement materials. A saturated specimen of porous materials was placed between two compartments; the upstream compartment contained tritiated water (HTO) and the downstream compartment a solution without HTO. The activity gradient between the compartments induced a flux of tritiated water through the sample. Usually, the flux of HTO is repeatedly measured by analysing the content of the downstream cell in the steady state. In this work, a theoretical study based on Fick's laws is used to show that it is possible to determine the diffusion coefficient D_{Fick1} by considering only two measurements obtained in the non-steady state, in the downstream compartment of a diffusion cell. A significant saving of time (approximately half) can be obtained. An experimental study of this innovative method is presented.

Keywords: Characterization [B]; Diffusion [C]; Transport properties [C]; Concrete [E]

1. Introduction

It is becoming increasingly important, for maintenance cost and security reasons, to assess, the ability of concrete to resist the ingress of aggressive species. The effective diffusion coefficient can be used as a performance indicator to characterise this ability. Many experimental techniques have been proposed to measure this diffusion coefficient (see for example [1–11]). Among the different possibilities, the diffusion test used in this study (Fig. 1) and described by several researchers [12–15] is usually the most reliable because the transport mechanism involved in the measurement is similar to the one which takes place in real structures. Nevertheless this technique is time consuming and accelerated methods have been proposed. Acceleration is usually caused by an electrical field because the species of interest are generally ions. Due to a lack of understanding of the physics of ion transport in porous media when an electrical field is applied, the comparison between the experimental results obtained with the different methods is not easy at present. Many correlations between experimental results are presented in the literature but their usefulness is limited [2,5].

The description of ionic transport in concrete is frequently based on Fick's laws but this assumption has been recently discussed [16–19]. Due to interactions between ions in the pore solution, a description based on the Nernst Planck equation has been proposed. Following this more realistic approach, the

Fig. 1. Experimental set-up for diffusion experiments.

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0008-8846/$ - see front matter © 2007 Published by Elsevier Ltd.
doi:10.1016/j.cemconres.2007.02.012

measurement of the effective diffusion coefficient using the conventional test is not obvious. So, to simplify the characterisation of diffusion in these materials, some researchers suggest measuring the diffusion coefficient with tritiated water (HTO). HTO is also currently used in nuclear applications to characterize the ability of concrete to confine radioactive waste. In this case, the diffusing species is water in which some of the hydrogen atoms have been replaced by tritium, a hydrogen isotope. The diffusion is then purely molecular and Fick's laws can be applied. Nevertheless, pure diffusion tests are still time consuming. Our objective is to propose an improvement of this classical method in order to obtain the effective diffusion coefficient. The experimental results obtained in the conventional diffusion test are treated in the non-steady state in order to calculate the diffusion coefficient from a theoretical description based on Fick's equations.

The study is presented in two parts:

- An analytical study of diffusion process using Fick's laws.
- An experimental study.

2. Analytical study of diffusion by Fick's laws

2.1. Conventional study of diffusion cell test method

In the experimental set-up (Fig. 1) the specimen of interest was placed between two compartments containing an alkaline solution with sodium hydroxide [NaOH]=1 g/L and potassium hydroxide [KOH]=4.65 g/L. These two chemical products were chosen to limit the leaching of the pore solution alkalis. The diffusive species, tritiated water (HTO), was added to the upstream compartment. The concentration of HTO was characterised by its radioactivity. In the upstream compartment, the initial activity was $c_{\mathrm{up}}=37.10^9\ \mathrm{Bq\,m^{-3}}$. The activities of downstream (c_{down}) and upstream cells were analysed regularly by liquid phase scintigraphy [20].

The boundary conditions were maintained constant as follows: the downstream solution was renewed when its activity was one hundredth of the upstream activity and the upstream solution was changed or its activity was adjusted when there was a decrease in the activity equal to one tenth of the initial value. In the following, the boundary conditions will be considered as constant.

For $x \leq 0$, $c(0,t) = c_{\mathrm{up}} = \text{constant}$ for $t \geq 0$

For $x \geq L$, $c(x,t) = 0$ for $t \geq 0$

For $0 < x < L$, $c(x,0) = 0$ for $t = 0$

Two regimes are classically observed (Fig. 2): first a transient state and then a steady-state. The constant flow J_{e} in the steady-state makes it possible to calculate the effective diffusion coefficient ($D_{\mathrm{Fick\ 1}}$) by using Fick's first law.

$$J_{\mathrm{e}} = -D_{\mathrm{Fick\ 1}} \frac{\partial c}{\partial x} \tag{1}$$

$$D_{\mathrm{Fick\ 1}} = \frac{L}{c_{\mathrm{up}}} J_{\mathrm{e}} = \frac{L}{c_{\mathrm{up}}} \frac{\Delta Q}{S \Delta t} \tag{2}$$

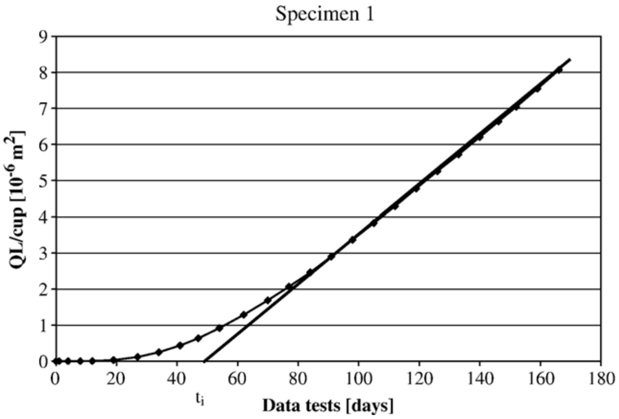

Fig. 2. Variation of QL/c_{up} with time. Q were measured in the downstream compartment.

where L is the thickness of the specimen, S, the cross sectional area of the exposed surface, and ΔQ, the quantity of HTO passing across the specimen during a period Δt in the steady-state. ΔQ is usually measured in the downstream compartment.

2.2. Theoretical determination of the diffusion coefficient from non-steady-state data acquired in the downstream compartment

In this study, we assume that:

- the effective diffusion coefficient is independent of the upstream concentration and of time (i.e. the microstructure of the cement-based material does not change during the diffusion test),
- the binding isotherm between the diffusing species and the cement-based material is linear [20],
- the temperature and the boundary conditions are constant during the diffusion test.

The non-steady state can be studied by solving Fick's second law:

$$\frac{\partial c}{\partial t} = \frac{D_{\mathrm{Fick1}}}{p + (1-p)\rho_{\mathrm{s}} K_{\mathrm{d}}} \frac{\partial^2 c}{\partial x^2} = D_{\mathrm{Fick2}} \frac{\partial^2 c}{\partial x^2} = \frac{D_{\mathrm{Fick1}}}{\alpha} \frac{\partial^2 c}{\partial x^2} \tag{3}$$

Where p is the open porosity, ρ_{s} is the density of the material, K_{d} is the distribution ratio [21] and α is defined by:

$$\alpha = p + (1-p)\rho_{\mathrm{s}} K_{\mathrm{d}} \tag{4}$$

With the boundary conditions of the experimental set-up, the analytical solution is [22]:

$$c(x,t) = c_{\mathrm{up}} - \frac{c_{\mathrm{up}} x}{L} - \frac{2c_{\mathrm{up}}}{\pi} \sum_{n=1}^{+\infty} \left[\frac{1}{n} \sin\left(\frac{n\pi x}{L}\right) \exp\left(-\frac{D_{\mathrm{Fick1}} n^2 \pi^2 t}{\alpha L^2}\right) \right] \tag{5}$$

The cumulative content of species $Q(x,t)$ coming from the upstream cell at x and t is obtained by integrating the flux given by Fick's first law between 0 and t:

$$Q(x,t)=\frac{D_{\mathrm{Fick1}}c_{\mathrm{up}}}{L}t+\frac{2c_{\mathrm{up}}\alpha L}{\pi^2}\sum_{n=1}^{+\infty}\left[\frac{1}{n^2}\cos\left(\frac{n\pi x}{L}\right)\left[1-\exp\left(-\frac{D_{\mathrm{Fick1}}n^2\pi^2t}{\alpha L^2}\right)\right]\right]\qquad(6)$$

For $x=L$, we obtain:

$$Q(L,t)=\frac{D_{\mathrm{Fick1}}c_{\mathrm{up}}}{L}t+\frac{2c_{\mathrm{up}}\alpha L}{\pi^2}\sum_{n=1}^{+\infty}\left[\frac{(-1)^n}{n^2}\left[1-\exp\left(-\frac{D_{\mathrm{Fick1}}n^2\pi^2t}{\alpha L^2}\right)\right]\right]\qquad(7)$$

or

$$Q(L,t)=\frac{D_{\mathrm{Fick1}}c_{\mathrm{up}}}{L}t-\frac{\alpha c_{\mathrm{up}}L}{6}-\frac{2c_{\mathrm{up}}\alpha L}{\pi^2}\sum_{n=1}^{+\infty}\left[\frac{(-1)^n}{n^2}\exp\left(-\frac{D_{\mathrm{Fick1}}n^2\pi^2t}{\alpha L^2}\right)\right]\qquad(8)$$

For $t\to\infty$, the last term of Eq. (8) tends toward 0. Then the asymptotic curve is given by:

$$Q(L,t)=\frac{D_{\mathrm{Fick1}}c_{\mathrm{up}}}{L}t-\frac{\alpha c_{\mathrm{up}}L}{6}\qquad(9)$$

The intercept of this asymptotic curve with the abscissa axis corresponds to the so-called time lag t_i (Fig. 2):

$$t_i=\frac{\alpha L^2}{6D_{\mathrm{Fick1}}}\qquad(10)$$

From Eq. (8), it is theoretically possible to obtain D_{Fick1} from pairs of experimental data $\{t_i, Q(L,t_i)\}$, α being considered as another unknown parameter. Practically, it is not easy to search for two parameters simultaneously. Nevertheless it is possible to solve this kind of problem by using optimisation functions.

Here we propose a simplified approach to find the two parameters D_{Fick1} and α. A term A is defined as follows:

$$A=\frac{D_{\mathrm{Fick1}}}{\alpha L^2}\qquad(11)$$

Eq. (8) can be rewritten:

$$Q(L,t)=c_{\mathrm{up}}\alpha L\left(\mathrm{At}-\frac{1}{6}-\frac{2}{\pi^2}\sum_{n=1}^{+\infty}\left[\frac{(-1)^n}{n^2}\exp\left(-\mathrm{An}^2\pi^2t\right)\right]\right)\qquad(12)$$

Let us consider two experimental values $Q_{\mathrm{a}}(L,t_{\mathrm{a}})$ and $Q_{\mathrm{b}}(L,t_{\mathrm{b}})$ corresponding to two data times t_{a} and t_{b} with $t_{\mathrm{b}}>t_{\mathrm{a}}\neq 0$.

Their ratio is given by:

$$\frac{Q_{\mathrm{a}}(L,t_{\mathrm{a}})}{Q_{\mathrm{b}}(L,t_{\mathrm{b}})}=\frac{\mathrm{At}_{\mathrm{a}}-\frac{1}{6}-\frac{2}{\pi^2}\sum_{n=1}^{+\infty}\left[\frac{(-1)^n}{n^2}\exp\left(-\mathrm{An}^2\pi^2t_{\mathrm{a}}\right)\right]}{\mathrm{At}_{\mathrm{b}}-\frac{1}{6}-\frac{2}{\pi^2}\sum_{n=1}^{+\infty}\left[\frac{(-1)^n}{n^2}\exp\left(-\mathrm{An}^2\pi^2t_{\mathrm{b}}\right)\right]}\qquad(13)$$

$Q_{\mathrm{a}}(L,t_{\mathrm{a}})/Q_{\mathrm{b}}(L,t_{\mathrm{b}})$ and t_{a} and t_{b} being known, it can be verified that Eq. (13) is monotonic when considered as a function of A. Consequently, this equation has only one solution, which can be calculated. Practically, the analytical study of this equation is difficult. Therefore, a numerical approach is proposed.

An example is presented in Fig. 3. The theoretical variation of A is plotted as a function of $Q_{\mathrm{a}}(L,t_{\mathrm{a}})/Q_{\mathrm{b}}(L,t_{\mathrm{b}})$ and t_{b} with $t_{\mathrm{a}}=50$ days. With a given t_{b} the figure shows that $\frac{Q_{\mathrm{a}}(L,t_{\mathrm{a}})}{Q_{\mathrm{b}}(L,t_{\mathrm{b}})}=f(A)$ is a strictly monotonic function.

For small values of A, Q_a and Q_b can be negative. In this case the curve of Fig. 3 presents a discontinuity. This mathematical result has no physical meaning.

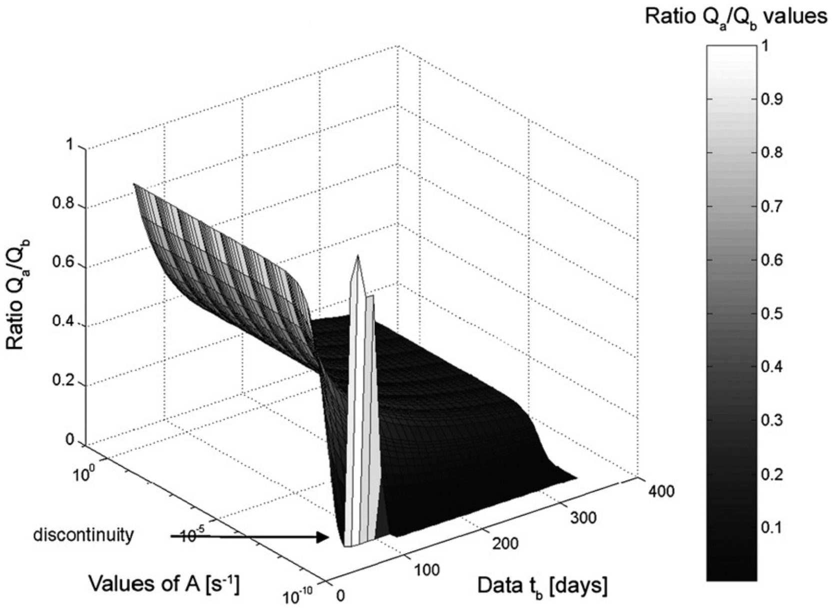

Fig. 3. A and $Q_{\mathrm{a}}/Q_{\mathrm{b}}(A,t_{\mathrm{a}},t_{\mathrm{b}})$ variations with t_{b} for $t_{\mathrm{a}}=50$ days ($t_{\mathrm{b}}>t_{\mathrm{a}}$). Downstream treatment.

Practically, the ratio $Q_a(L,t_a)/Q_b(L,t_b)$ and t_a and t_b are the experimental input of the problem. The expected output is the diffusion coefficient. To calculate D_{Fick1}, A and α have to be determined first and D_{Fick1} can then calculated from Eq. (11). A is calculated by an iteration process in order to verify Eq. (13).

Once A is known, α is calculated from Eq. (12) by using a pair of experimental data for Q and t. For example, with the pair $\{t_a, Q_a\}$, the result is given by:

$$\alpha = \frac{Q_a(L,t_a)}{c_{\mathrm{up}}L}\left(At_a - \frac{1}{6} - \frac{2}{\pi^2}\left(\sum_{n=1}^{+\infty}\frac{(-1)^n}{n^2}\exp\left(-\mathrm{A}n^2\pi^2t_a\right)\right)\right)^{-1} \tag{14}$$

Finally, the effective diffusion coefficient is calculated from Eq. (11).

3. Experimental study

This approach has been proposed to determine the diffusion coefficient before the steady state is reached. This theoretical analysis needs to be evaluated with respect to experimental work.

3.1. Experimental procedure

Tritiated water diffusion was studied on concrete specimens made with normal Portland cement (CEM I 52.5 R). The mix proportions of the concrete are given in Table 1. After mixing, the concrete was cast in cylindrical moulds (Φ=11 cm, H=22 cm). After 1 day, the cylinders were removed from the moulds and then cured in a plastic bag at 20 °C for 1 year. 15-mm-thick samples were then sawed from the cylinders for diffusion studies.

The experimental set-up is shown in Fig. 1. Three samples were tested to obtain a more reliable result.

3.2. Experimental results

The concentration of tritiated water in the solution of the downstream compartment was determined each time it was renewed. The measurement was made by determining the radioactivity of the solution by scintigraphy [20]. The time interval between two samples was chosen so that the increase in concentration (radioactivity) was sufficient to be measurable with good accuracy while not being too high to respect the boundary conditions. We chose an interval of 1 week (Fig. 4). In these conditions, for a diffusion coefficient of 10^{-12} m^2/s, the increase in the downstream concentration only represented 0.01% of the upstream concentration.

Table 1
Mix proportions of the concrete

	CEM I 52, 5 R cement	Water	Sand	Gravel 3/8 mm	Gravel 8/10 mm	W/C ratio
[kg/m^3]	450	180	666.3	631.3	456	0.40

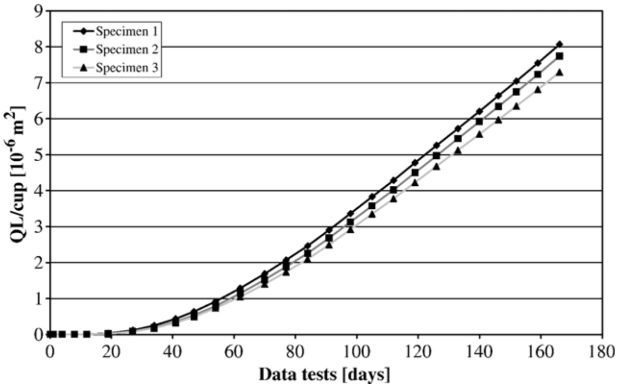

Fig. 4. QL/c_{up} variation as a function of time − experimental results of tritiated water diffusion test obtained by CEA DAMRI.

To evaluate the approach, the following method was adopted.

- The diffusion test set-up was run until the steady state was reached. The steady-state was considered to have been reached when the flux variation was less than 5% over the last five experimental data.
- The diffusion coefficient D_{Fick1} and α were calculated in the steady state. These results obtained from the conventional method were considered as reference values.
- The new method in the non-steady state was then developed and its results compared with the reference.The cumulative curves obtained for the three concrete samples are presented in Fig. 4. The thickness and the cross-sectional areas of the samples are indicated in Table 2.

4. Validation and application of the method

The effective diffusion coefficient D_{Fick1}, the α factor calculated from Fick's first law (see Eq. (2)) and the time-lag expression (see Eq. (11)) are given in Table 2.

The scatter of the diffusion coefficient (less than 7%) among the three samples was not very significant. It must be pointed out that variations of around 20% were obtained by Richet [20] on cement paste tests. The deviations of the α factor were limited to a relative variation of 2%.

4.1. Validation of the method for determining D_{Fick} in non-steady-state conditions

The method presented in the first part of this paper was used to evaluate the α factor and the diffusion coefficient. As mentioned in the theoretical description, the input data were a ratio $Q_a(L,t_a)/Q_b(L,t_b)$ and the corresponding times t_a and t_b. Theoretically, the outputs should be the same whatever t_a and t_b. Practically, a certain deviation was observed because the accuracy was relatively poor when smaller quantities passed and when t_a and t_b were very close. To evaluate the new method, all the data were processed. The numerical processing was performed in the Matlab® environment. n pairs of data $\{t_i, Q_i\}$ were keyboarded. An algorithm was

Table 2
Effective diffusion coefficients D_{Fick1}, time-lag t_i and α factor of concrete specimens tested

Sample reference	Thickness [10^{-2} m]	Cross section [10^{-4} m^2]	c_{up} [10^{10} Bq. m^{-3}]	Effective diffusion cefficients D_{Fick1} [m^2.s^{-1}]	Time-lag t_i [days]	Alpha factor
1	1.57	75.6	3.7	8.06E−13	50.6	0.086
2	1.58	75.6	3.7	7.94E−13	53.5	0.088
3	1.55	75.6	3.7	7.51E−13	54.0	0.088

developed in order to process all the experimental data (see Fig. 5).

Thus, when $t_a = t_1$, $n-1$ values were calculated for α and D_{Fick1}, when $t_a = t_2$, $n-2$ values were calculated, etc…

To make the method easier to understand, we propose to limit the discussion to the values of the diffusion coefficient. For each of the three samples, Fig. 6 shows the variation of the calculated diffusion coefficient D_{Fick1} versus t_{a} with $t_{\mathrm{b}} = 166$ days (curve 1) and $t_{\mathrm{b}} = t_{\mathrm{a}} + 7$ days (curve 2). On this figure, the value of the diffusion coefficient determined in the steady state is indicated, together with error bars corresponding to the experimental uncertainty. This uncertainty has been evaluated at 20% [20].

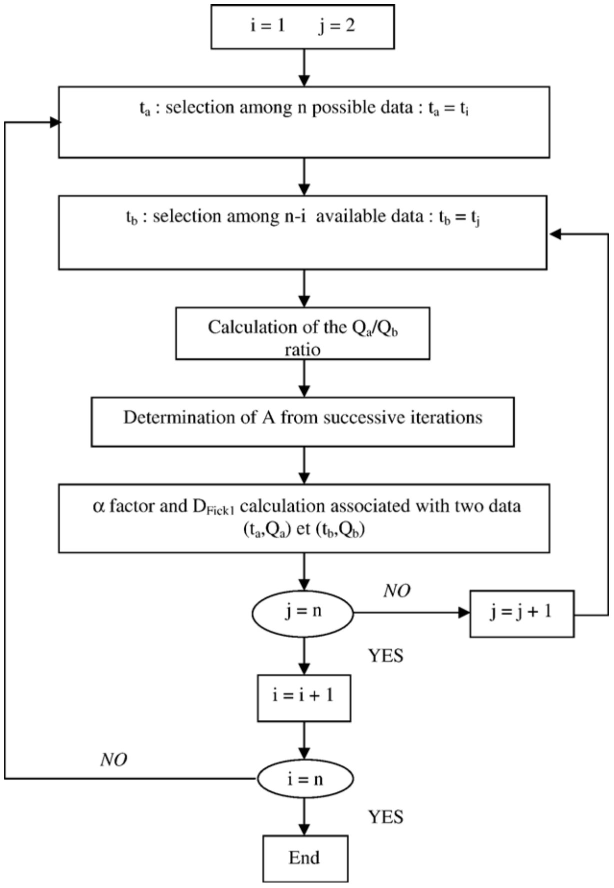

Fig. 5. Algorithm used to run the experimental data $\{t_i, Q_i\}$ obtained in the non-steady state.

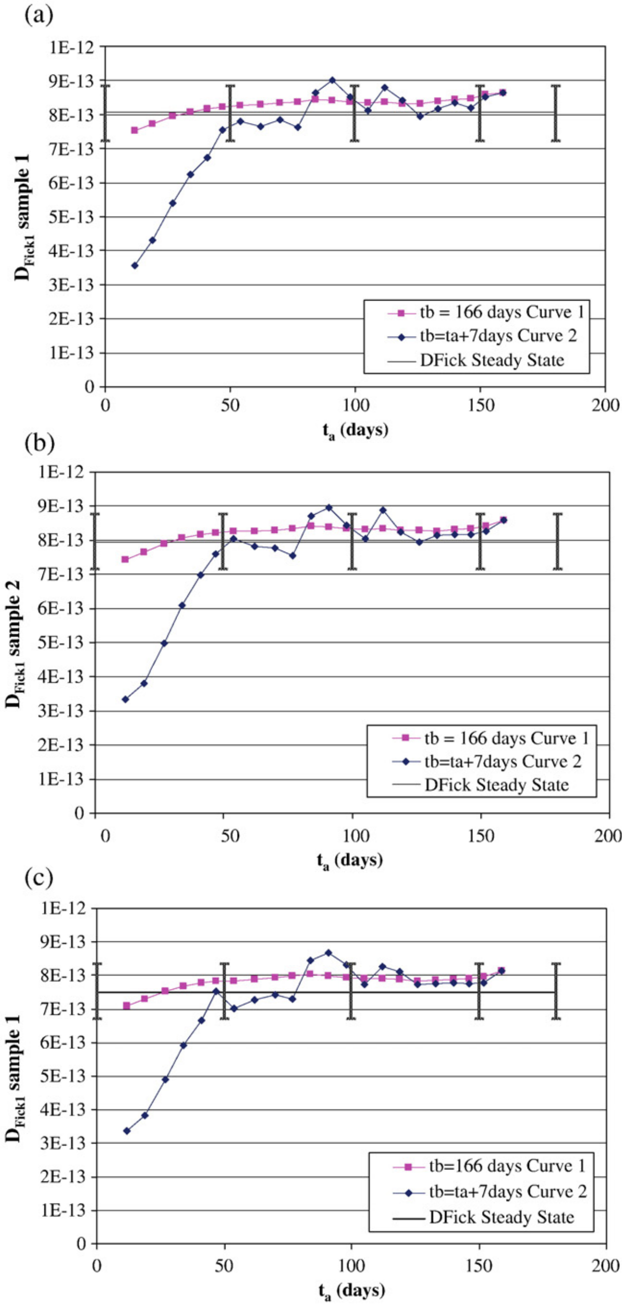

Fig. 6. (a) (b) (c): Variation of tritiated water diffusion coefficient D_{Fick1} (for samples 1, 2 and 3) versus t_{a} for different values of t_{b}.

For $t_{\mathrm{b}} = 166$ days (curve 1), the result obtained is practically always within the uncertainty limit of the steady state value. However, it is worth noting that the new method is of no interest in this case as it uses a measurement t_{b} made over a very long time.

For $t_{\mathrm{b}} = t_{\mathrm{a}} + 7$ days (curve 2), the result obtained comes within the uncertainty limit after about 50 days. The curve of Fig. 4 shows that, at this time, the test is not yet in the steady state.

Curve 2 nevertheless shows fluctuations of the value of the calculated D_{Fick1}. It can be shown (see Fig. 7) that the amplitude of these fluctuations decreases as the difference between t_{a} and

t_b increases. In all cases, the calculated diffusion coefficient is within the limits of uncertainty after 50 days.

4.2. Practical application of the new method

Let us now consider the practical case where the diffusion coefficient is unknown at the start of the test. The experimental data allow the curves of Fig. 6 to be plotted. We propose to choose an interval of 3 weeks between times t_a and t_b in order to limit the fluctuations as shown in Fig. 7. If Fick's law applies, the curves $D_{\mathrm{Fick1}} = f(t_a)$ tend towards a horizontal asymptote. In consequence, the variation between two successive values of D_{Fick1} tends towards zero, and the criterion for stopping the test can be chosen as a maximum value of the relative variation of D_{Fick1} between two successive values. The generally accepted accuracy for measurements of D_{Fick1} in the steady state is +/−10% [20]. We can take it that, for a given test, the value of D_{Fick1} calculated from the results in transient conditions is correct when the relative variation between two successive values of D_{Fick1} is less than 5%. Fig. 8 shows this variation versus time. On the basis of the above criterion, the test can be stopped after 48 days. The results obtained if the test is continued remain within the same uncertainty range (+/−5%). It can thus be concluded that, for the sample under test, the diffusion coefficient $D_{\mathrm{Fick1}} = 7.67\ 10^{-13}\ \mathrm{m^2/s}$ +/−5%.

5. Conclusion

A theoretical study of molecular diffusion has been developed considering the increase of tritiated content in the downstream compartment in the non-steady state. We have shown that it is theoretically possible to determine the diffusion coefficient D_{Fick1} and the α factor from only two experimental measurements.

This theoretical study is supported by experimental work conducted on three concrete specimens. Reference values for the diffusion coefficient were calculated from steady-state data. With this classical method the diffusion coefficients are calculated when the steady-state regime is reached i.e. after a period of 3 to 5 times the time lag. The diffusion coefficients determined with the new method in the non-steady state are very close to the reference values. The advantage of this new technique is obvious: good agreement is obtained between the two methods by using experimental data acquired after a test period as short as the time lag.

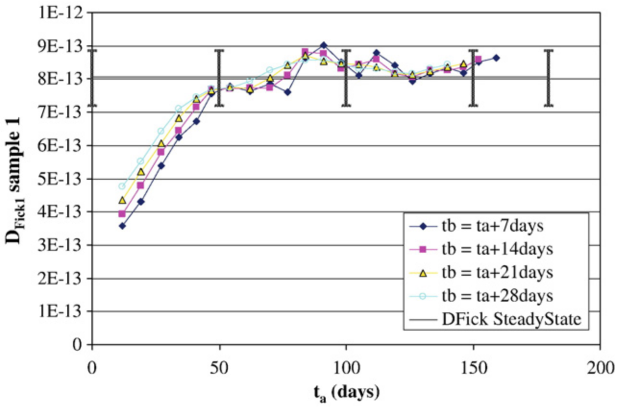

Fig. 7. Variation of diffusion coefficient with time t_a for different times t_b. Analysis of choice of delta t.

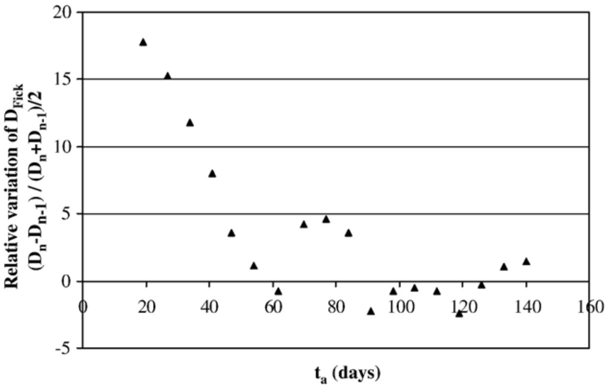

Fig. 8. Relative variation of D_{Fick1} versus time t_a.

Acknowledgments

The authors would like to express their thanks to the Commissariat à l'Energie Atomique for its technical support.

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