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Measurement of the mass diffusivity in cement mortar: use of initial rates of water absorption

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Abstract

This paper presents a method of defining mass diffusivity D_m of a porous material, based on non-stationary processes of moisture absorption. The results of testing absorption in 15 cycles, Is–XVs, are presented. The experiment covered series of cement mortars of different water–cement ratio, $(w/c = 0.50, 0.65, 0.80)$, and was carried out at three values of temperatures (T = 20, 35, 50 °C), at which the testing referred to five selected ranges of relative humidity of air, $\Delta \varphi$. The testing technique itself and the influence of certain factors on D_m defined by means of \sqrt{t} -type calculations were evaluated. © 2002 Elsevier Science Ltd. All rights reserved.

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1. Introduction

The specification of any porous building material with respect to moisture characteristics requires, among other things, the defining of its isotherms of absorption and desorption as well as a factor which represents diffusive transfer of water and is given by the stationary method called cup method (subject to standards applied in many countries, e.g. [1,2]).

This paper presents results of the experiment aimed at evaluating the suitability of the non-stationary method to define mass diffusivity of a porous material. The assumptions to start the study were as follows. Since it is necessary anyway to define the isotherms of absorption and desorption for a given material, thus it might be beneficial to define another parameter, i.e. diffusivity at the same time. The attempts so far, e.g. [3–6], have proved the chance to do so.

The study as shown below covered the same mortars, the same temperatures and similar humidity values as in the stationary testing of the mass diffusivity that previously followed the cup method technique [7]. Generally the experiment was carried out in two phases of identical scopes [8]. The end of phase one that covered absorption processes was regarded as the commencement of phase two, i.e. moisture desorption processes. This paper presents results of absorptive measurements of the mass diffusivity determined by measurements of the mas
means of the type \sqrt{t} rule.

2. Theoretical

The subject of this analysis is the one-dimensional isothermal moisture absorption in a porous homogeneous medium. Such a process, which occurs with the negligence of gravity forces, was described in [9–11] by the following formula:

$$
\frac{\partial c_{\mathbf{m}}}{\partial t} = \frac{\partial}{\partial x} \left(D_{\mathbf{m}} \frac{\partial c_{\mathbf{m}}}{\partial x} \right),\tag{1}
$$

where D_m represents the diffusivity of the medium. The above formula was defined on the assumption that coefficient D_m of a certain material at the given temperature is only dependent on the moisture contents designated here by c_m .

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Nomenclature

The below considerations, which form theoretical assumptions of the experiment described in Section 3, refer to non-stationary moisture fields developed during the absorption in a plane sheet $0 \le x \le d$ without any constraints along y , z . It was assumed that upon the commencement of the process there was no physically combined water within the sample, i.e. the sample characterized by the constant initial concentration $c_{m1} = 0$. It was also assumed that all points of the surfaces $x = 0$ and $x = d$ are subject to the same moisture conditions $(\varphi_2 = \text{const.}, c_2 = \text{const.})$ that are not variable in time which stabilizes the concentration of moisture on outer surfaces and sets it to the constant level of c_{m2} . It was also assumed that the exchange of moisture is effected through diffusion only, and coefficient D_m at the certain temperature might be given a constant value which is the average for the range in study, $c_{m1}-c_{m2}$. On these assumptions the moisture field concerned would be a 1D field described by means of function $c_m(x, t)$ which satisfies the differential equation

$$
\frac{\partial c_{\mathbf{m}}}{\partial t} = D_{\mathbf{m}} \frac{\partial^2 c_{\mathbf{m}}}{\partial x^2}
$$
 (2)

and appropriate initial and boundary conditions. The following initial and boundary conditions were formulated after taking into consideration the initial phase of the process concerned, namely:

$$
c_{\rm m}(t=0, 0 < x < \infty) = c_{\rm m1} = 0,\tag{3}
$$

$$
c_{\rm m}(t > 0, \ x = 0) = c_{\rm m2}.\tag{4}
$$

The solution to a diffusion equation on the above assumptions is as follows [12,13]:

$$
c_{\rm m} = c_{\rm m2} \bigg(1 - \text{erf} \frac{x}{2\sqrt{D_{\rm m}t}} \bigg). \tag{5}
$$

The rate of the penetration of sample face unit area $(x = 0)$ by water vapour is described by [12]:

$$
\left(-D_{\mathbf{m}}\frac{\partial c_{\mathbf{m}}}{\partial x}\right)_{x=0} = D_{\mathbf{m}}c_{\mathbf{m}2}\frac{1}{\sqrt{\pi D_{\mathbf{m}}t}}.\tag{6}
$$

After integrating in respect of time t the total amount of substance which has migrated through surface $x = 0$ inside the sample in time t is obtained. Initial changes in mass of a dry sample Δm_t , which absorbs moisture from the environment of constant concentration c_2 through two faces of area A each are described then by the following formula:

$$
M_t = \frac{\Delta m_t}{2A} = 2c_{\text{m2}}\sqrt{\frac{D_{\text{m}}t}{\pi}}.\tag{7}
$$

Graphical representation of sample mass increments registered during the absorption in respect to square root of time results in a line for the initial phase of the process and after crossing the certain time the line would turn into a curve tending to reach the maximum asymptotically. The inclination of the line designated by $a_{\sqrt{t}}$, makes it possible to calculate mass diffusivity D_{m} according to the following formula:

$$
D_{\rm m} = a_{\sqrt{t}}^2 \frac{\pi d^2}{16\Delta m_\infty^2}.
$$
\n(8)

3. Experimental

Cured cement mortars of different porosity structure given by factor w/c were used as a model medium for the estimation of non-stationary measuring techniques. It was confirmed according to the literature [14] that w/c is a key factor as far as the mortar structure is concerned. The study was reduced to typical mortars with $w/c = 0.50, 0.65, 0.80$. The non-stationary testing of isothermal absorption processes was to be carried out at the temperature of $T = 20$, 35 and 50 °C. The temperature of mass diffusivity measurements set at 20, 35 and 50 \degree C (formerly stationary measurements [7], and non-stationary measurements at present) was based on results of pilot research described in [15]. Five ranges of humidity were studied at each temperature within the frames of the hygroscopic interval. Thus the absorption experiment covered 15 cycles designated as Is–XVs (see Table 1).

Disc-shaped mortar samples, 80 mm dia and 10 mm thick, made after slicing cylinders, 80 mm dia, 160 mm long, were used in the study of the mass diffusivity by means of non-stationary methods. Each cycle covered nine disc samples, \oslash 80 mm/10 mm, three samples of each mortar thus the total number of samples used in 15 cycles amounted to 135. After the preliminary measurements (e.g. d , A , V), the insulation of sample side surfaces commenced. The samples were put into a dryer (105–110 \degree C) after the silicone had hardened. Measurements of the absorption started immediately after the completion of drying. The initial mass (m_1) of the samples was found and then the samples were placed at one of three defined temperatures; three samples of each mortar at five selected air relative humidity. The simulation of humidity was effected by properly selected hygrostatic solutions. The measurements were carried out for several months until the moisture equilibrium $(m₂ = const.)$ in all the samples tested at the certain temperature was reached.

4. Results

Calculation sheets used to estimate mass diffusivity Calculation sheets used to estimate mass diffusivity D_m on the basis of \sqrt{t} -type rule, as per formula (8), were produced for each single cycle (Is– XVs) for each mortar in the study. They formed grounds for the graphic rep-

Table 1 Average D_m for mortars tested in cycles Is–XVs

resentation of relation $\Delta m_t = f(\sqrt{t})$ for subsequent cycles and samples of the mortars in study in certain atmosphere. Fig. 1 shows curves for samples of the $w/c = 0.50$ mortar in the middle cycles at each temperature, i.e. cycles IIIs, VIIIs, XIIIs. Values of mass diffusivity D_m were calculated on the grounds of $a_{\sqrt{t}}$, Δm_{∞} , *d*, separately for all 135 samples. Table 1 lists average D_m , found for each mortar, and equal to the arithmetic mean of three samples tested in each of Is– XVs cycles.

The assessment of the accuracy of a test method has been based on the formula used for calculating the measurement error of any indirectly measured quantity $z = f(y_1, y_2, \ldots, y_n)$ [16]:

$$
|\Delta z| = \sum_{i=1}^{n} \left| \frac{\partial f(y_1, y_2, \dots, y_n)}{\partial y_i} \Delta y_i \right|,
$$
 (9)

where Δy_i is the systematic error of certain simple quantities y_1, y_2, \ldots, y_n .

The presented test method deals with indirect measurements where the diffusivity D_m is not found directly but other quantities that are closely related thereto are found and the said relation is as follows:

$$
D_{\rm m} = \left(\frac{m_t - m_1}{\sqrt{t}}\right)^2 \frac{\pi d^2}{16(m_2 - m_1)^2}
$$

= $\frac{\pi}{16} \left(\frac{m_t - m_1}{m_2 - m_1}\right)^2 \frac{d^2}{t}.$ (10)

Systematic error of certain primary values in the study was as follows: $\Delta m_1 = \Delta m_2 = \Delta m_t = 0.001$ g, $\Delta t =$ 0.25 h, $\Delta d = 0.0001$ m. Detailed calculations of the share of certain primary values in the error of final results of all measurements could be found in [8].

Fig. 1. Changes in mass related to square root of time registered for mortar samples of $w/c = 0.50$ in some selected cycles: (a) IIIs; (b) VIIIs; (c) XIIIs.

5. Conclusions

Pursuant to the study the following general conclusions may be drawn:

- 1. All mortars tested in the absorption processes exhibited the absolutely certain tendency of growth of coefficient D_m assigned to a given range of humidity, the said growth followed the temperature rise. The intensity of influence of this factor was dependent on the type of the material in study. The influence of temperature in the majority of $\Delta\varphi$ ranges tested was the strongest in case of the 0.50 mortar, slightly weaker in case of the 0.65 and generally the weakest in case of the 0.80 although in the latter case the increase in temperature from 20 to 35 °C resulted in the 2–3-fold increase in D_m , and in case of increase to $50 °C$ – even 5-fold in the extreme case. Temperature is then regarded as the significant factor of the strong influence on the diffusion flow within a porous material that has its thermodynamic reasons.
- 2. The absorption study exhibited a general regularity: the increase in factor w/c resulted in the increase in coefficient $D_m - w/c = 0.65$ mortar -2 times maximum, $w/c = 0.80$ mortar -2.8 times maximum as compared to the $w/c = 0.50$ mortar. The diversification of structure within a group of materials of the same type is not predominant, but cannot be neglected.
- 3. The influence of humidity on mass diffusivity D_m determined on the grounds of absorption measurements exhibited the same tendencies (as previously

described in, e.g. [17]) in all mortars and at all temperatures. The D_m increased temporarily along with the growth of the RH (in the range of medium RH values) and then dramatically dropped (being even some times lower) although the fall was gradual during the study at higher and higher humidity. The regularities found might be explained as follows: the D_m , estimated on the assumptions adopted in Section 2, describes only the gaseous phase transfer in porous medium. The progressive moistness of the material produces less favorable conditions for the process to carry on. The intensity of the process slows down since finer pores are blocked by particles of the condensing water whereas the diameter of larger ones is reduced by subsequent layers of molecularly bound water.

by subsequent layers of molecularly bound water.
4. The type \sqrt{t} procedure applied to estimate coefficient D_m shall be regarded as a sufficiently accurate technique – provided the measuring regime at least comparable to the one used throughout this paper is observed. Detailed results listed in [8] prove that even in the least favorable case – the combination of influence of all measured primary values – the final result would carry the systematic error of 3–6%.

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