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Application of $\sqrt{t}$-type, logarithmic and half-time methods in desorptive measurements of diffusivity in narrow humidity ranges

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Abstract

This paper concerns the use of non-stationary desorptive measurement techniques for defining the mass diffusivity of cement based materials. Three different procedures are presented: $\sqrt{t}$-type calculation; logarithmic; and half-time procedures. Cement mortars of different water to cement ratios (w/c), equal to 0.50, 0.65 and 0.80 were selected as the model environment for testing the usability of the above-mentioned desorptive techniques. The study was carried out at the temperature (T) of 20 °C within narrow relative humidity (φ) ranges: from $\varphi_1 = 30\%$ to $\varphi_2 = 12\%$, and 50%→30%, 75%→50%, 85%→75%, 97%→85%. The results obtained are used to evaluate the conformity of these methods. The conformity is analyzed with regard to each mortar in all the above humidity ranges $\Delta\varphi$. The values of diffusivity D_m, defined by means of the $\sqrt{t}$-type calculation and the logarithmic procedure, demonstrated rather high conformity, all relative differences between $D_m(\sqrt{t})$ and $D_m(\ln)$ did not exceeded 20%. However, the half-time procedure can be applied for rough estimation of the diffusivity only. That is because deviations between $D_m(t_{1/2})$ and the values found by means of the two other methods were too large.

Keywords: Drying; Diffusion; Transport properties; Mortar

1. Introduction

Moisture processes within porous cement-based materials have been studied by a number of researchers, because moisture content affects many of the properties of such materials. Moisture can significantly change basic properties (strength, elasticity, etc.) and insulation (thermal conductivity), for instance. The life of building structures is also closely dependent on moisture issues. At early ages, water movement can have a damaging effect (for example, drying can disturb hydration, and moisture has a significant influence on the development of shrinkage stresses). At later ages, degradation of the building elements, e.g., to biological corrosion, can occur. An appropriate description of these processes may help to prevent, or at least to control, their effects.

The transport coefficient of water is necessary to determine the moisture processes and to identify the moisture gradients that are formed within structural elements concerned in result of the exchange of moisture with the environment. This paper deals with the use of a non-stationary measurement technique for defining the mass diffusivity of cement mortars. The non-stationary methods have widely been applied to define the mass diffusivity of coating materials, e.g., Refs. [1–4]. There have also been attempts to use the non-stationary desorptive methods for cement based materials as in absorption measurements, as shown in Ref. [5], for instance.

Immediately after production concrete elements have relatively high moisture content, but they begin to dry out and release the moisture to drier surroundings as soon as formwork is removed.

Non-stationary measurements are based on the general assumption that the observation of desorption rate of component A within a plane sheet of component B makes it possible to estimate the mass diffusivity of a medium. Examples of practical application of this method that are given in Refs. [1–6] indicate such possibility. The three calculation methods applied ($\sqrt{t}$-type method, logarithmic method and half-time method), although developed on the basis of different initial assumptions, led to satisfyingly convergent results.

In this study, the usability o f the methods to define the mass diffusivity of cement based materials was tested on cement

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mortars with $w/c = 0.50–0.80$. The selected material satisfies requirements of homogeneity and structural representativeness with respect to a small size of the samples. The paper presents the use of the three calculation procedures for desorptive measurements of the mass diffusivity at the temperature of $T = 20$ °C in five different humidity ranges, $\Delta\varphi$.

2. Theoretical aspects

2.1. Determination of D_m by $\sqrt{t}$-type method

According to [1,5–7], the initial change in mass of a disc sample with insulated side surfaces is:

$$\Delta m_t = \frac{4\Delta m_{\max}}{d}\sqrt{\frac{D_{\mathrm{m}}\cdot t}{\pi}} \tag{1}$$

where Δm_t (kg) denotes the change in mass of the sample during the desorption process, $\Delta m_{\max}$ is the total change in mass of the sample at the end of the process, i.e., after achieving the moisture equilibrium with surrounding, D_{m} denotes the mass diffusivity of water vapor (m^2/s) referred to moisture concentration ω (kg/m^3) in the material, t is the process duration (s) and d is the plane plate thickness (m).

Changes in mass in the initial phase of the desorption (as well as absorption) are proportional to the square root of time [1,5]. Rectilinear behaviour in some cases had been observed until the sample released 70% of the moisture content [5]. By rearranging Eq. (1), to give:

$$D_{\mathrm{m}} = a_{\sqrt{t}}^2\,\frac{\pi\cdot d^2}{16\cdot\Delta m_{\max}^2} \tag{2}$$

where

$$a_{\sqrt{t}} = \frac{\Delta m_t}{\sqrt{t}} \tag{3}$$

the mass diffusivity D_{m} can be determined from the slope $a_{\sqrt{t}}$ of the curve that describes the relation $\Delta m_t = f(\sqrt{t})$. Practical applications to determine diffusivity D_{m} with regard to non-stationary desorptive measurements are discussed in Section 4.1.

2.2. Determination of D_m by logarithmic method

According to [5–7], the change in mass during an advanced phase of the absorption or desorption in a plane plate of porous material is given by:

$$\frac{\Delta m_t}{\Delta m_{\max}} = 1-\frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp[-D_{\mathrm{m}}(2n+1)^2\pi^2 t/d^2]. \tag{4}$$

According to Ref. [5], in the expansion (4) the higher terms with $n>0$ can be neglected when the process occurs after absorbing (in the case of absorption) or releasing (in the case of desorption) 40% of the total exchanged mass. Having assumed this formula is to be used for the description of the more advanced phase of the phenomenon, just the first term of a series be taken into consideration. After logarithmic transformation, the following linear equation in relation to time is obtained: $\ln(1-\Delta m_t/\Delta m_{\max}) = f(t)$. The value of the slope of the function, $a_{\ln}$, can be used to determine the mass diffusivity according to the following formula:

$$D_{\mathrm{m}} = -a_{\ln}\frac{d^2}{\pi^2} \tag{5}$$

where:

$$a_{\ln} = \frac{\ln\left(1-\frac{\Delta m_t}{\Delta m_{\max}}\right)-\ln\frac{8}{\pi^2}}{t}. \tag{6}$$

The application of this method to determine diffusivity D_{m} with regard to the desorption experiment will be presented in Section 4.2.

2.3. Determination of D_m by half-time method

As shown in Refs. [1,5,6], the mass changes in the initial phase of the desorption are proportional to the square root of time. Introducing the so-called half-time $t_{1/2}$, [7], corresponding to "the half" change in mass, i.e., $\Delta m_t = 0.5\ \Delta m_{\max}$, formula (1) can be given as follows:

$$\frac{1}{2} = \frac{4}{\sqrt{\pi}}\sqrt{D_{\mathrm{m}}}\sqrt{\frac{t_{1/2}}{d^2}} \tag{7}$$

This implies:

$$D_{\mathrm{m}} = \frac{\pi}{64}\cdot\frac{d^2}{t_{1/2}}. \tag{8}$$

This simple formula can be used for determining mass diffusivity D_{m} of the process, provided that the above assumptions have been met. The identical equation is also obtained for the advanced phase of the (absorption/desorption) process.

A practical application of the half-time method to evaluate $D_{\mathrm{m}}(t_{1/2})$ of the mortars in some humidity ranges $\Delta\varphi$ will be discussed in Section 4.3.

3. Experimental aspects

The formulas (2), (5) and (8) may be used to determine the mass diffusivity of materials, once the experimental desorption process has been performed. Generally, the experimental arrangement consists of placing a plane plate made of the material to be studied with insulated side surface in an environment of constant temperature and fixed humidity φ_2. The plane plate should be characterized by uniform distribution of internal humidity φ_1 ($\varphi_1 > \varphi_2$). The exchange of moisture between the plane sheet of constant thickness d and surroundings is carried out through two faces, each of area A. The aim of the measurements is to observe mass decrements during desorption.

3.1. Preparation of samples

Experiments were carried out on three cement mortars of different w/c ratios. It is shown in Ref. [8] that, among various factors, the w/c ratio influences transportation properties of cement-based materials to the highest extent. The study program covered non-modified cement mortars of w/c within 0.5–0.8. The preliminary estimation showed that in the case of $w/c<0.5$ or $w/c>0.8$ the internal structure of samples would not be homogeneous. It is not easy to produce mortar of $w/c<0.5$ due to difficulties of compaction and the risk of defects (bubbles of air, etc.). In case of mortar of $w/c>0.8$ with the same amount of cement and aggregate, the mixture would be too liquid, causing segregation of the ingredients, and problems with cutting of the samples of low strength.

Mortars were made from Portland cement and quartz sand (0–2 mm). The cement/sand ratio was estimated in such a way that the same volumetric proportion of matrix to aggregate was maintained for all the mortars. The above criterion ensured the same proportion of a material volume, in which the transfer processes occur, to the one not affected by the same processes. The composition by weight of the mortars is shown in Table 1.

Cylinder samples $\varnothing=80$ mm, $L=160$ mm were made of three mortars. After one day, the samples were removed from their moulds and placed in a water bath. The samples were kept in water long enough to let the hydration processes make such progress that they would not cause significant changes in the structure of the samples during the long-lasting research on desorption. To assess the progress of hydration a MOM derivatograph of F. Paulik–J. Paulik–L. Erdey was used. The measurements were taken in intervals lasting a few months. The measurement operation was performed by differential thermal analysis (DTA), made simultaneously with thermogravimetric analysis (TG), at constant heating rate of the sample. The 500-g samples of material were heated from ambient temperature up to 1100 °C at a rate of 7.5 °C/min. Weight decreases for the temperature range from 120 to 1050 °C were used to calculate the degree of hydration α_H. The measurement and calculation technique for α_H was the same as in the work [9]. During the period between the 12th and 24th month of curing, the hydration degree increase for $w/c=0.50$ was 3.3%, and at $w/c=0.65$ reached 2.8%. In case of $w/c=0.8$, no measurable hydration progress was recorded. The 12-month period of water storage of the samples was recognized as sufficient for stabilizing the hydration process.

The target disc samples, $\varnothing=80$ mm, $d=10$ mm, were then made for the non-stationary study of the mass diffusivity. The middle part of the cylinder was sliced with a diamond saw and then ground to produce 10 mm thick discs with parallel faces. After cleaning, the side surfaces were coated with moisture-hardening silicone that provided high adhesion to the sample material. 45 disc samples, $\varnothing=80$ mm, $d=10$ mm, with insulated side surfaces were manufactured. For each of the five cycles, three samples of each mortar were prepared.

Table 1
Mix proportions by weight

Mortar	Portland cement	Quartz sand	Water
1	1	3.00	0.50
2	1	3.52	0.65
3	1	4.05	0.80

3.2. Conditioning of samples

Prior to the desorptive measurements, the samples were conditioned for a couple of months. The samples were dried at $T=105$–110 °C (to obtain constant mass) and were then stored at constant temperature and humidity conditions until the moisture content had stabilized and the mass transfer had ceased. The pre-conditioning process was carried out at the temperature of 20 °C in five separately defined ranges of the air relative humidity φ, according to the following scheme: $T=20$ °C, while $\varphi=0\%\rightarrow30\%$, $0\%\rightarrow50\%$, $0\%\rightarrow75\%$, $0\%\rightarrow85\%$ and $0\%\rightarrow97\%$.

The thermostatic chamber (±1 °C accurate) was used to maintain stable temperature throughout the several-month-long experiment. Inside the thermostatic chamber there were five desiccators with saturated water solutions of the following salts: $CaCl_2$, $Ca(NO_3)_2$, NaCl, KCl, K_2SO_4, which were used to obtain the assumed humidity φ, and were selected according to the available literature [10–13]. Such an arrangement made it possible to carry out the conditioning process simultaneously in five different humidity environments at 20 °C. The conditioning of the samples ended upon stabilizing the mass of the samples after about 4 months. It was assumed that after such a period of time the moisture in the samples exhibited uniform distribution.

3.3. Desorptive measurements

When the pre-conditioning process had been completed, the desorptive measurements of the mass diffusivity at $T=20$ °C were initiated. The samples, at equilibrium moisture corresponding to the five humidity ranges adopted previously, were transferred to five separate desiccators of lower stabilized humidity than before, i.e., $\varphi=12\%$, 30%, 50%, 75% and 85%. To keep the humidity at the constant values of $\varphi=30\%$, 50%, 75% and 85% accordingly, the same solutions were used as before, while the saturated water solution of LiCl stabilized the humidity of $\varphi=12\%$.

The desorption experiment consisted of five cycles (1d–5d) and was carried out with the same equipment as per the following scheme: $T=20$ °C, while $\varphi=30\%\rightarrow12\%$, $50\%\rightarrow30\%$, $75\%\rightarrow50\%$, $85\%\rightarrow75\%$ and $97\%\rightarrow85\%$.

There were 9 samples (3 of each mortar) in each desiccator. The samples were placed on racks that assured a fixed position during the whole period of the desorption measurements. Due to technical limitations there were no devices to force the air flow inside the desiccators.

At the beginning, the samples were weighed every 12 h, later every 24 h and finally every couple of days. The mass was taken by means of an electronic scales (accuracy ±0.001 g). In order to carry out the periodical weight check, the samples were taken out of the desiccator and weighed outside. The samples were put into a tight tailored container and the weight check of the sample on the electronic scales took a few seconds only. The short weighing time did not cause noticeable changes in the sample weight—compared to significant changes Δm_t recorded in the subsequent measurements at rather long time increments (minimum range Δt = 12 h). The short time interval, in which a desiccator was opened, might cause significant destabilization of humidity within. Resuming the stabilized humidity might take certain time depending on the differences between the inside environment parameters and laboratory ones. Prior to the desorption experiments, a study of the humidity stabilization time was performed after arranging the similar disturbance. The desiccator, which was placed inside the thermostatic chamber, was equipped with a humidity sensor connected to a reader located outside the chamber. The longest time required for humidity stabilization was about 2 h. The time increments used in the experiment (12-h, mainly 24-h and 48-h steps) ensured the environment of assumed humidity parameters for the samples for most of the time. During taking the measurement, the solid phase in the bottom of a desiccator was monitored to ensure that there was sufficient to avoid dilution of the solution.

Results of desorptive measurements will be presented in detail in Section 4. It should emphasized, however, that the process of exchanging mass between a sample and the environment was rather fast at the initial stage only. Then, as the time went on, the process dramatically slowed down and might last several months. For the 5 cycles, samples of each mortar showed on average the following final weight loss:

	Final weight loss, $\Delta m_{\max}$ (g)		
	w/c=0.50	w/c=0.65	w/c=0.80
Cycle 1d, 30%→12% (completed after 3.5 months)	0.479	0.363	0.289
Cycle 2d, 50%→30% (completed after 4.5 months)	0.957	0.775	0.617
Cycle 3d, 75%→50% (completed after 3 months)	0.720	0.654	0.638
Cycle 4d, 85%→75% (completed after 4.2 months)	0.492	0.471	0.505
Cycle 5d, 97%→85% (completed after 3.5 months)	2.137	2.406	3.006

The measurements during the cycles were stopped when the specimen weight loss Δm, calculated in week-long intervals was below 1% of the total weight change, as noted up to that moment of the desorption process.

The desorptive measurements of a single cycle ranged from 3.5 to 4.5 months. Such a duration might not be sufficient for achieving full moisture equilibrium. Even after a year, a small loss of mass of the mortar samples under study may be observed. It was understood that with such small weight changes (not exceeding 1%), the influence of other factors, i.e., the carbonization process or the temporary temperature and humidity instability, which was difficult to eliminate, would be more and more important. The above mentioned temporary temperature and humidity instability seems to be responsible for local peaks of the weight loss curves, which occurred during the measurements. Each uncontrolled temperature drop resulting from even a temporary power shortage to the thermostat may have its effect in an increase of the air relative humidity inside the desiccator, which, in turn, may cause an unwanted change to the humidity status of the samples kept in it. Changes of this sort can be observed in Fig. 3 that addresses the mortar w/c=0.80 being under study in cycle 5d, and manifest at t=804 h and t=1476 h. None of the three samples showed a decrease—on the contrary, they showed an increase in weight in relation to the measurement taken a week before. For instance, within the period from t=636 h to t=804 h, the averaged weight increase for three samples was 0.053 g. The conclusion was that the unintended influence of accidental factors might considerably exceed the influence of the systematic errors, resulting from the time limitations of the experiment.

Thus, the value of $\Delta m_{\max}$ from measurements, i.e., a difference between the non-stabilized final sample mass m_2 and the initial mass m_1, was slightly underestimated. It affected the accuracy of determined diffusivities $D_m(\sqrt{t})$, $D_m(\ln)$, $D_m(t_{1/2})$—and will thoroughly be discussed in Appendix A.

4. Practical applicability of three desorptive methods

4.1. $\sqrt{t}$-type method

The $\sqrt{t}$-type method is based on Eq. (2). Practical applications require thickness d to be taken for each sample

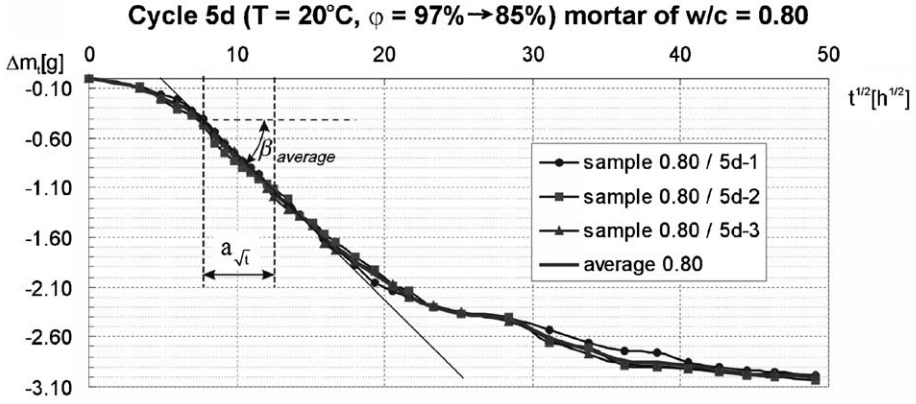

Fig. 1. Graph of function $\Delta m_t = f(\sqrt{t})$ for the mortar of w/c=0.80 at T=20 °C and φ=97%→85%.

Table 2
Mass diffusivities by √*t*-type method

T=20 °C, φ=30%→12%		T=20 °C, φ=50%→30%		T=20 °C, φ=75%→50%		T=20 °C, φ=85%→75%		T=20 °C, φ=97%→85%	
Sample	$D_m(\sqrt{t})$ (m^2/s)	Sample	$D_m(\sqrt{t})$ (m^2/s)	Sample	$D_m(\sqrt{t})$ (m^2/s)	Sample	$D_m(\sqrt{t})$ (m^2/s)	Sample	$D_m(\sqrt{t})$ (m^2/s)
0.50 / 1d-1	13.98 E−12	0.50 / 2d-1	6.48 E−12	0.50 / 3d-1	23.26 E−12	0.50 / 4d-1	21.53 E−12	0.50 / 5d-1	
0.50 / 1d-2	15.40 E−12	0.50 / 2d-2	6.43 E−12	0.50 / 3d-2	24.38 E−12	0.50 / 4d-2	24.40 E−12	0.50 / 5d-2	6.58 E−12
0.50 / 1d-3	14.68 E−12	0.50 / 2d-3	6.42 E−12	0.50 / 3d-3	25.49 E−12	0.50 / 4d-3	25.36 E−12	0.50 / 5d-3	8.26 E−12
0.65 / 1d-1	15.95 E−12	0.65 / 2d-1	6.79 E−12	0.65 / 3d-1	26.42 E−12	0.65 / 4d-1	27.40 E−12	0.65 / 5d-1	12.09 E−12
0.65 / 1d-2	15.62 E−12	0.65 / 2d-2	7.22 E−12	0.65 / 3d-2	27.52 E−12	0.65 / 4d-2	25.93 E−12	0.65 / 5d-2	9.16 E−12
0.65 / 1d-3	16.24 E−12	0.65 / 2d-3	7.45 E−12	0.65 / 3d-3	27.57 E−12	0.65 / 4d-3	24.23 E−12	0.65 / 5d-3	6.92 E−12
0.80 / 1d-1	18.13 E−12	0.80 / 2d-1	9.49 E−12	0.80 / 3d-1	26.56 E−12	0.80 / 4d-1	21.04 E−12	0.80 / 5d-1	12.15 E−12
0.80 / 1d-2	19.98 E−12	0.80 / 2d-2	9.66 E−12	0.80 / 3d-2	32.01 E−12	0.80 / 4d-2	23.40 E−12	0.80 / 5d-2	8.28 E−12
0.80 / 1d-3	17.58 E−12	0.80 / 2d-3	13.56 E−12	0.80 / 3d-3	30.49 E−12	0.80 / 4d-3	20.21 E−12	0.80 / 5d-3	11.17 E−12

under study and the maximum loss of mass $\Delta m_{\max}$, in a certain desorption cycle to be determined. To obtain an accurate value of the factor $a_{\sqrt{t}}$ it is necessary to record the mass changes on the regular basis in order to represent the relation $\Delta m_t = f(\sqrt{t})$.

In Fig. 1, example diagrams of function $\Delta m_t = f(\sqrt{t})$ made for mortar w/c=0.80 have been shown. For illustration purposes, the cycle 5d, which encompasses the range of the highest moisture and is the most difficult to carry out, has been chosen. On the abscissa, a range has been marked out, where the parameter $a_{\sqrt{t}}$ was defined as a tangent of the β inclination angle of the linear section of the function $\Delta m_t = f(\sqrt{t})$. After discarding the preliminary period, in all cases, a relatively long and easy to identify section $\Delta\sqrt{t}$, characterized by a steady inclination angle β, was present. For example, in Fig. 1, this section referred to time range from 60 h (average value $\Delta m_t/\Delta m_{\max}$ was 0.14) to 156 h (average value $\Delta m_t/\Delta m_{\max}$ reached 0.38).

The straight section of the curve was identified for each sample to define the value of $a_{\sqrt{\mathrm{t}}}$.

Diffusivity values calculated with the √*t*-type method for all samples of the tested mortars during the five desorption cycles have been shown in Table 2. The results scatter analysis have been made in Section 5.1 and the average diffusivity values $D_m(\sqrt{t})$ for the three mortars under study have been listed in Table 5.

4.2. Logarithmic method

The application of the logarithmic method as given by formula (5) required measuring the thickness d of each sample and determining factors $a_{\ln}$ as per Eq. (6) which, in turn, required a detailed representation of logarithmic functions for each case. The factor $a_{\ln}$ was obtained for the straight sections of the logarithmic curves after the sample had released approximately 40% of its moisture exchanged with the environment during the process discussed. It should be noted, however, that in some cycles a linear course of the logarithmic function started already from $\Delta m_t/\Delta m_{\max} \approx 0.3$.

The technique of calculating the factor $a_{\ln}$ has been illustrated with Fig. 2, showing courses of function $\ln(1-\Delta m_t/\Delta m_{\max}) = f(t)$, drawn for the three samples of w/c=0.80 in cycle 5d. On the drawing a section has been marked out, where the factor $a_{\ln}$ was calculated as a tangent of the inclination angle γ of the linear section in function $\ln(1-\Delta m_t/\Delta m_{\max}) = f(t)$. The beginning of the section was attributed to an ordinate from the range −0.4 to −0.5 ($\Delta m_t/\Delta m_{\max} \approx 0.3 \div 04$). The end of the section was identified individually for single function courses reproduced for each sample. For instance, in Fig. 2 this section encompasses the time span from t=120 h (average value $\Delta m_t/\Delta m_{\max}$ was abt. 0.3) to t=372 h (average value $\Delta m_t/\Delta m_{\max}$ amounted to abt. 0.7).

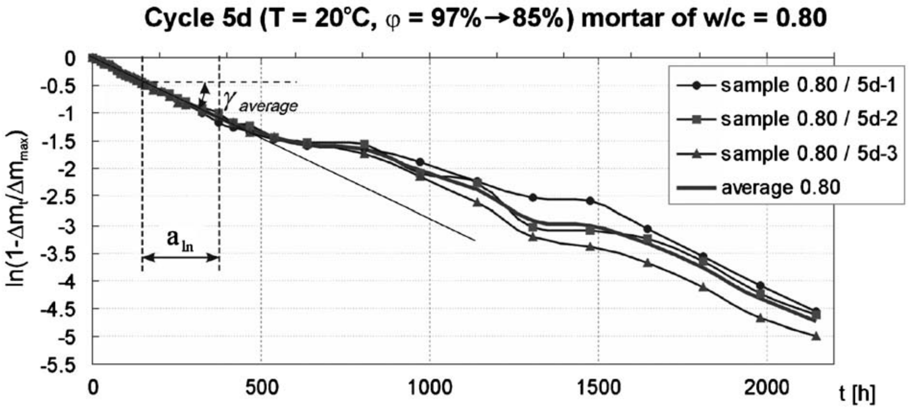

Fig. 2. Graph of function $\ln(1-\Delta m_t/\Delta m_{\max}) = f(t)$ for the mortar of w/c=0.80 at T=20 °C and φ=97%→85%.

Table 3
Mass diffusivities by logarithmic method

T=20 °C, φ=30%→12%		T=20 °C, φ=50%→30%		T=20 °C, φ=75%→50%		T=20 °C, φ=85%→75%		T=20 °C, φ=97%→85%	
Sample	D_m(ln) (m^2/s)	Sample	D_m(ln) (m^2/s)	Sample	D_m(ln) (m^2/s)	Sample	D_m(ln) (m^2/s)	Sample	D_m(ln) (m^2/s)
0.50 / 1d-1	11.57 E−12	0.50 / 2d-1	5.77 E−12	0.50 / 3d-1	20.82 E−12	0.50 / 4d-1	18.00 E−12	0.50 / 5d-1	
0.50 / 1d-2	13.12 E−12	0.50 / 2d-2	5.76 E−12	0.50 / 3d-2	22.13 E−12	0.50 / 4d-2	20.30 E−12	0.50 / 5d-2	5.26 E−12
0.50 / 1d-3	14.98 E−12	0.50 / 2d-3	5.79 E−12	0.50 / 3d-3	21.89 E−12	0.50 / 4d-3	21.76 E−12	0.50 / 5d-3	6.84 E−12
0.65 / 1d-1	14.35 E−12	0.65 / 2d-1	6.24 E−12	0.65 / 3d-1	23.75 E−12	0.65 / 4d-1	21.62 E−12	0.65 / 5d-1	8.87 E−12
0.65 / 1d-2	14.26 E−12	0.65 / 2d-2	6.39 E−12	0.65 / 3d-2	24.82 E−12	0.65 / 4d-2	26.47 E−12	0.65 / 5d-2	7.76 E−12
0.65 / 1d-3	14.83 E−12	0.65 / 2d-3	6.36 E−12	0.65 / 3d-3	23.64 E−12	0.65 / 4d-3	21.15 E−12	0.65 / 5d-3	7.18 E−12
0.80 / 1d-1	17.78 E−12	0.80 / 2d-1	8.01 E−12	0.80 / 3d-1	23.40 E−12	0.80 / 4d-1	17.17 E−12	0.80 / 5d-1	9.28 E−12
0.80 / 1d-2	18.81 E−12	0.80 / 2d-2	8.38 E−12	0.80 / 3d-2	27.57 E−12	0.80 / 4d-2	18.45 E−12	0.80 / 5d-2	7.16 E−12
0.80 / 1d-3	18.25 E−12	0.80 / 2d-3	11.45 E−12	0.80 / 3d-3	29.19 E−12	0.80 / 4d-3	18.42 E−12	0.80 / 5d-3	10.04 E−11

Table 3 contains diffusivity values obtained with the logarithmic method for all the samples used for the desorption experiment. Table 5 lists average diffusivity values D_m(ln), being an expectation value of three samples tested during individual cycles. A scatter analysis for individual results was made in Section 5.2.

4.3. Half-time method

The stage of processing the results of the half-time method is relatively less time consuming. Evaluation of $D_m(t_{1/2})$, according to Eq. (8), required measuring the samples thickness d and identification of time $t_{1/2}$. It is therefore necessary to spot two equilibrium states, the initial one of sample mass m_1 and the final one of sample mass m_2, in order to find $\Delta m_{max}=m_2-m_1$ and estimate time $t_{1/2}$ with a relatively high accuracy.

An example of $\Delta m_t/\Delta m_{max}=f(t)$, determined in cycle 5d for the mortar $w/c=0.80$, is shown in Fig. 3. For the sake of illustration, the half-time representing the average weight change $\Delta m_t/\Delta m_{max}=0.5$ has been marked on the abscissae. Working on the measurements results, single values of the half-time $t_{1/2}$ were determined separately for each sample, and on this basis a corresponding diffusivity value $D_m(t_{1/2})$ was calculated.

Diffusivity values determined with the half-time method for all samples concerned are shown in Table 4. The scatter is analyzed in Section 5.3 and the average values $D_m(t_{1/2})$ for all three samples during five cycles were drawn up in Table 5.

5. Analysis of results obtained by means of the three methods discussed

5.1. √t-type method

The recorded initial behaviour of function $\Delta m_t=f(\sqrt{t})$ always exhibited a divergence from the rectilinear course that occurred later. In certain cases it was found necessary to reject the very first measurement, in other cases the first few measurements had to be rejected and only the measurements, which were taken after several hours could be treated as the base for calculations. As mentioned in Section 3.3, there was no forced air flow within the desiccators. The consequences of that were most evident at the early stage of drying and their effect was the strongest on the behaviour of the graphs $\Delta m_t=f(\sqrt{t})$. Nevertheless, for each measurement (neglecting the non-representative initial period), it was possible to identify a long enough time interval characterized by a rectilinear function of $\Delta m_t=f(\sqrt{t})$.

The results of calculations by the √t-type method proved to be highly consistent (see Table 2). The scatter of single results compared with the average values $D_m(\sqrt{t})$ found on the basis thereof was of a few percent for most of the cycles. In the cycle that covered the highest humidity φ=97%→85%, i.e., the most difficult for stimulation measurement condition, the scatters for

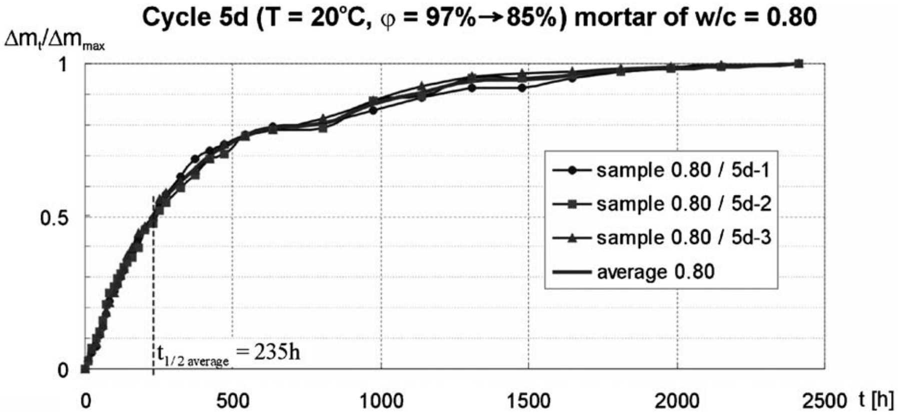

Fig. 3. Graph of function $\Delta m_t/\Delta m_{max}=f(t)$ for the mortar of $w/c=0.80$ at T=20 °C and φ=97%→85%.

Table 4
Mass diffusivities by half-time method

$T=20$ °C, $\varphi=30\%\rightarrow12\%$		$T=20$ °C, $\varphi=50\%\rightarrow30\%$		$T=20$ °C, $\varphi=75\%\rightarrow50\%$		$T=20$ °C, $\varphi=85\%\rightarrow75\%$		T=20 °C, $\varphi=97\%\rightarrow85\%$	
Sample	$D_m(t_{1/2})$ (m^2/s)	Sample	$D_m(t_{1/2})$ (m^2/s)	Sample	$D_m(t_{1/2})$ (m^2/s)	Sample	$D_m(t_{1/2})$ (m^2/s)	Sample	$D_m(t_{1/2})$ (m^2/s)
0.50 / 1d-1	7.92 E−12	0.50 / 2d-1	4.34 E−12	0.50 / 3d-1	14.97 E−12	0.50 / 4d-1	8.34 E−12	0.50 / 5d-1	
0.50 / 1d-2	10.24 E−12	0.50 / 2d-2	4.50 E−12	0.50 / 3d-2	15.85 E−12	0.50 / 4d-2	12.57 E−12	0.50 / 5d-2	4.41 E−12
0.50 / 1d-3	10.98 E−12	0.50 / 2d-3	4.56 E−12	0.50 / 3d-3	15.90 E−12	0.50 / 4d-3	12.50 E−12	0.50 / 5d-3	5.15 E−12
0.65 / 1d-1	13.11 E−12	0.65 / 2d-1	4.85 E−12	0.65 / 3d-1	17.10 E−12	0.65 / 4d-1	15.12 E−12	0.65 / 5d-1	5.74 E−12
0.65 / 1d-2	14.36 E−12	0.65 / 2d-2	5.03 E−12	0.65 / 3d-2	17.47 E−12	0.65 / 4d-2	19.12 E−12	0.65 / 5d-2	5.49 E−12
0.65 / 1d-3	13.41 E−12	0.65 / 2d-3	5.03 E−12	0.65 / 3d-3	16.60 E−12	0.65 / 4d-3	15.88 E−12	0.65 / 5d-3	4.70 E−12
0.80 / 1d-1	16.32 E−12	0.80 / 2d-1	6.03 E−12	0.80 / 3d-1	17.43 E−12	0.80 / 4d-1	11.78 E−12	0.80 / 5d-1	5.83 E−12
0.80 / 1d-2	17.04 E−12	0.80 / 2d-2	6.48 E−12	0.80 / 3d-2	20.22 E−12	0.80 / 4d-2	12.32 E−12	0.80 / 5d-2	5.63 E−12
0.80 / 1d-3	16.93 E−12	0.80 / 2d-3	8.24 E−12	0.80 / 3d-3	21.78 E−12	0.80 / 4d-3	17.45 E−12	0.80 / 5d-3	5.95 E−12

some samples exceeded 20%. It was also the cycle, in which the only rejected sample was noted (0.50/5d-1).

5.2. Logarithmic method

The satisfactory convergence of the main assumptions with the numerical results of the desorption study could be stated. Plotted curves of $\ln(1-\Delta m_t/\Delta m_{\max})=f(t)$ exhibit the rectilinear course for a clearly defined range of time Δt. The linear response of the function under study was observed just after the samples had released about 30%–40% of the total moisture content. The curves obtained made it possible to define relatively unambiguously the value of the slope factors $a_{\ln}$ for each sample. It should also be noted that the curves plotted for the samples in the case of each mortar were almost identical, confirming a good accuracy of the measurements taken.

The scatter analysis of the results obtained by the logarithmic method for three samples of a given mortar studied in five cycles makes it possible to confirm a good compliance of results (Table 3), the deviations from average $D_m(\ln)$ ranged from a few to more then ten percent. In one specific case the difference between a single measurement (sample 0.80/2d-3) and the average exceeded 20%. Since the sample 0.50/5d-1 was rejected in the $\sqrt{t}$-type procedure, this sample was also ignored in the logarithmic calculations.

5.3. '$t_{1/2}$' method

The calculation procedure of the half-time method seems quite simple when compared to the other two. It does not require the reproduction or detailed analysis of functional course of changes recorded. However, the measurement itself is as time consuming as in the two other methods. Thus, it is necessary to study desorption starting from the initial state corresponding to the initial moisture equilibrium in material ω_1 (sample of mass m_1) and continue until a new moisture equilibrium ω_2 is reached (sample of mass m_2). The point is to find $1/2\ \Delta m_{\max}$ ($\Delta m_{\max}=m_1-m_2$), and then time $t_{1/2}$, which corresponds to the change of the half of the mass. The detailed calculations show that, e.g., in the case of lowest humidity ranges the time $t_{1/2}$ was 143 h for mortar $w/c=0.50$ and only 81 h for mortar $w/c=0.80$. In the case of the highest humidity ranges $t_{1/2}$ reaches 278 h at $w/c=0.50$ and 235 h at $w/c=0.80$ accordingly. Thus, it is advisable to take frequent measurements of mass—even over the first two weeks of a process—to find the time with the sufficient accuracy in case of a material of compact structure and in specific humidity conditions.

The analysis of data given in Table 4 shows that the half-time method results in the acceptable scatter. Single results of individual samples differ from the average obtained on their basis generally within a few or more then ten percent. In case of

Table 5
Averages $D_m(\sqrt{t})$, $D_m(\ln)$, $D_m(t_{1/2})$ and their mutual quantitative relations

Desorption $T=20$ °C	Mortar	$D_m(\sqrt{t})$ (m^2/s)	$D_m(\ln)$ (m^2/s)	$D_m(\ln)/D_m(\sqrt{t})$ (−)	$D_m(t_{1/2})$ (m^2/s)	$D_m(t_{1/2})/D_m(\sqrt{t})$ (−)	$D_m(t_{1/2})/D_m(\ln)$ (−)
$\varphi=30\%\rightarrow12\%$	$w/c=0.50$	14.69 E−12	13.22 E−12	0.90	9.71 E−12	0.66	0.73
	$w/c=0.65$	15.94 E−12	14.48 E−12	0.91	13.63 E−12	0.85	0.94
	$w/c=0.80$	18.56 E−12	18.28 E−12	0.98	16.76 E−12	0.90	0.92
$\varphi=50\%\rightarrow30\%$	$w/c=0.50$	6.44 E−12	5.77 E−12	0.90	4.47 E−12	0.69	0.77
	$w/c=0.65$	7.15 E−12	6.33 E−12	0.89	4.97 E−12	0.70	0.79
	$w/c=0.80$	10.90 E−12	9.28 E−12	0.85	6.92 E−12	0.63	0.75
$\varphi=75\%\rightarrow50\%$	$w/c=0.50$	24.38 E−12	21.61 E−12	0.89	15.57 E−12	0.64	0.72
	$w/c=0.65$	27.17 E−12	24.07 E−12	0.89	17.06 E−12	0.63	0.71
	$w/c=0.80$	29.69 E−12	26.72 E−12	0.90	19.81 E−12	0.67	0.74
$\varphi=85\%\rightarrow75\%$	$w/c=0.50$	23.76 E−12	20.02 E−12	0.84	11.14 E−12	0.47	0.56
	$w/c=0.65$	25.85 E−12	23.08 E−12	0.89	16.71 E−12	0.65	0.72
	$w/c=0.80$	21.55 E−12	18.01 E−12	0.84	13.85 E−12	0.64	0.77
$\varphi=97\%\rightarrow85\%$	$w/c=0.50$	7.42 E−12	6.05 E−12	0.82	4.78 E−12	0.64	0.79
	$w/c=0.65$	9.39 E−12	7.94 E−12	0.85	5.31 E−12	0.57	0.67
	$w/c=0.80$	10.53 E−12	8.83 E−12	0.84	5.80 E−12	0.55	0.66

two samples (0.50/4d-1 and 0.80/4d-3) the deviation from the average exceeded 20%. Obviously, as in two other procedures, the sample 0.50/5d-1 was rejected.

6. Conclusions

Fig. 4 shows the average values of diffusivity D_m for the tested mortars, where the values were determined by means of $\sqrt{t}$-type method, logarithmic procedure and half-time method. Plotted points that represent the variation of D_m refer to the average moisture content of five ranges $\Delta\varphi$. The figure demonstrates the similar nature of variation of D_m courses determined with different methods.

Table 5 compares the three methods in the study and assess their conformity. The table lists average values of diffusivity factors $D_m(\sqrt{t})$, $D_m(\ln)$ and $D_m(t_{1/2})$. The ratios $D_m(\ln)/D_m(\sqrt{t})$, $D_m(t_{1/2})/D_m(\sqrt{t})$ and $D_m(t_{1/2})/D_m(\ln)$ were used to compare the calculation procedures.

The $\sqrt{t}$-type method and the logarithmic method were quite compatible, and the results therefrom differed by a few or more then ten percent. The logarithmic method showed lower diffusivity than those by the $\sqrt{t}$-type method in the case of each mortar and each humidity range $\Delta\varphi$.

The half-time method proved to be quite different from the two other methods. The significant differences were observed when comparing $D_m(t_{1/2})/D_m(\sqrt{t})$ — they ranged from 10% to about 50% and tended to rise along with the increasing humidity. The relation $D_m(t_{1/2})/D_m(\ln)$ seems to be more favourable but the results differ and in most cases exceed 20–30%.

The $\sqrt{t}$-type and logarithmic methods require more efforts in handling the results since it is necessary to reproduce the detailed behaviour of the considered functions in order to identify the time period which fulfills the assumptions. It is therefore possible to reject a range affected by possible disturbances of the process and identify representative slopes, $a_{\sqrt{t}}$ or $a_{\ln}$. Consequently, high accuracy of measurements could be achieved, cf. Ref. [14]. The diffusivity values found by the $\sqrt{t}$-type and logarithmic techniques differed from each other by less than 20% in the case of each cycle and each mortar, which turns out comparable with the scatter among particular samples. Thus, both methods can be regarded as equivalent calculation techniques.

Diffusivity $D_m(t_{1/2})$ calculated by the half-time method always showed lower values than those of $D_m(\sqrt{t})$ and $D_m(\ln)$. To some extent this might reflect too low rate of changes in the initial stage of desorption processes due to the lack of the air flow within desiccators. Consequently, the time $t_{1/2}$ increases, causing the diffusivity $D_m(t_{1/2})$ calculated on its basis to drop. The calculation technique, however, which in fact identifies the one particular value of $t_{1/2}$, does not provide possibilities to avoid related errors — the opposite of the other two techniques concerned. Thus in case of the experiments carried out in similar conditions the half-time method could only be regarded as a technique to allow for a rough estimation of the diffusivity of a tested material.

Improvement in the accuracy of each method concerned could be achieved by a climate chamber with controlled air flow inside, having continued weighing of the sample always placed inside the climate chamber.

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Appendix A. Uncertainties in $D_m(\sqrt{t})$, $D_m(\ln)$, $D_m(t_{1/2})$ due to incomplete mass stabilization

The shortest cycles, 1d and 5d, lasted about 3.5 months each. The changes in mass in cycle 1d ($\varphi=30\%\rightarrow12\%$) were generally rather small, and the values Δm reported in about 1-week intervals in the final phase amounted to some 0.001–0.003 g. The differences Δm did not exceed 1% of the measured final mass, and were close to the measuring uncertainties influenced by random factors. The amount of moisture released

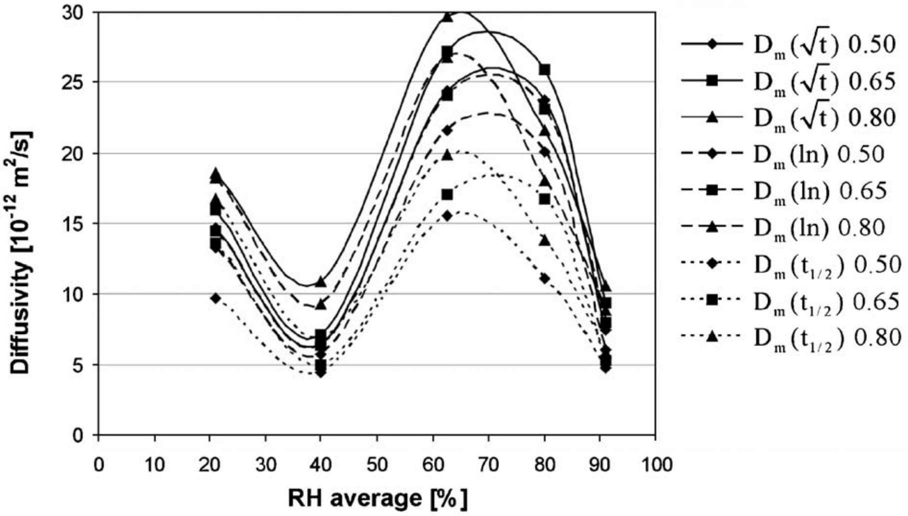

Fig. 4. Mass diffusivity $D_m(\sqrt{t})$, $D_m(\ln)$ and $D_m(t_{1/2})$ for each mortar (w/c=0.50; 0.65; 0.80) versus relative humidity φ.

in desorptive cycle 5d ($\varphi = 97\% \rightarrow 85\%$) was generally several times larger than in the cycle 1d. The cycle was stopped when mass differences in 1-week intervals did not exceed 1% when compared with the global mass change. The other cycles required a longer measuring period for the same acceptable results. The longest cycle 2d, for instance, was stopped after 4.5 months. But even in the latter case the final mass was not fully stabilized. Thus, each value $\Delta m_{\max}$ determined after the completion of measurements is underestimated to some extent. The presented below estimation of accuracy assumed that the uncertainty in measurements of final mass m_2 is about 0.01 g, with the electronic scales accuracy of 0.001 g. Obviously, the difference Δm_2 between the assumed and real equilibrium mass might be larger. The main idea was to estimate the range of uncertainties that affect the coefficients $D_{\mathrm{m}}(\sqrt{t})$, $D_{\mathrm{m}}(\ln)$, $D_{\mathrm{m}}(t_{1/2})$ found by all the three methods.

The assessment of the accuracy of tested methods has been based on the rule of propagation of uncertainties, cf. Ref. [15]. When a quantity z is a function of directly measured quantities $y_1, y_2, \ldots, y_n$:

$$z = f(y_1, y_2, \ldots, y_n) \tag{9}$$

it can be shown, using Taylor's series expansion that the first-order uncertainty in z is given by:

$$|\Delta z| = \sum_{i=1}^{n} \left| \frac{\partial f(y_1, y_2, \ldots, y_n)}{\partial y_i} \Delta y_i \right| \tag{10}$$

where Δy_1, $\Delta y_2, \ldots$, Δy_n are the increments of y_1, $y_2, \ldots$, y_{n}, respectively, in the power expansion, [16].

In the case of $D_{\mathrm{m}}(\sqrt{t})$ formula (2) was transformed to a form based on the measured simple data:

$$D_{\mathrm{m}} = \frac{\pi}{16} \cdot \left(\frac{m_t - m_1}{m_2 - m_1} \right)^2 \cdot \frac{d^2}{t}. \tag{11}$$

The absolute partial derivatives $\partial D_{\mathrm{m}} / \partial m_2$ were found for each particular measurement to establish a share of m_2 in the uncertainty of D_{m}:

$$\frac{\partial D_{\mathrm{m}}}{\partial m_2} = -\frac{\pi}{8} \cdot \frac{(m_t - m_1)^2}{(m_2 - m_1)^3} \cdot \frac{d^2}{t}. \tag{12}$$

The analysis of uncertainties shows that the results of cycle 1d are affected by the uncertainty in the final mass m_2 measurement most. Uncertainties for samples of certain mortar are within 2. 2–3. 3% at $w/c = 0.50$; 4.3–5.0% at $w/c = 0.65$ and 5.1–6.0% at $w/c = 0.80$, respectively. The measurement uncertainties in other cycles prove to be smaller, the lowest can be found in cycle 5d (0.3–0.5% at $w/c = 0.50$; 0.3–0.4% at $w/c = 0.65$ and 0.4–0.5% at $w/c = 0.80$). Therefore, if, in the cycle 5d (the one of the biggest weight variations), the underestimation had been even 10 times bigger than expected, i.e., if it had reached the level of 0.1 g, the resulting inaccuracies would have settled on the level between 3–5%.

The diffusivity $D_{\mathrm{m}}(\ln)$ was found according to formula (5). After entering physical values from direct measurements, the formula could be given as:

$$D_{\mathrm{m}} = -\frac{\ln\left(1 - \frac{m_t - m_1}{m_2 - m_1}\right) - \ln\frac{8}{\pi^2}}{t} \cdot \frac{d^2}{\pi^2} \tag{13}$$

and the partial derivatives $\partial D_{\mathrm{m}} / \partial m_2$ for each particular measurement were found:

$$\frac{\partial D_{\mathrm{m}}}{\partial m_2} = \frac{m_t - m_1}{(m_2 - m_t)(m_2 - m_1)t} \cdot \frac{d^2}{\pi^2}. \tag{14}$$

The obtained set of uncertainties shows some tendencies identical to the ones of the $\sqrt{t}$-type method. The numerical values are as follows: cycle 1d-uncertainty of 3.0–3.6% at $w/c = 0.50$; 4.9–5.6% at $w/c = 0.65$; 6.1–6.6% at $w/c = 0.80$ and cycle 5d-uncertainty of 0.6% at $w/c = 0.50$; 0.5–0.6% at $w/c = 0.65$ and 0.4–0.6% at $w/c = 0.80$, respectively.

The diffusivity $D_{\mathrm{m}}(t_{1/2})$, determined according to formula (8), includes two spatial and time parameters — the sample thickness d and half time $t_{1/2}$. The accuracy of $D_{\mathrm{m}}(t_{1/2})$ depends on the accuracy of d and $t_{1/2}$, with the partial derivative $\partial D_{\mathrm{m}} / \partial t_{1/2}$ given by:

$$\frac{\partial D_{\mathrm{m}}}{\partial t_{1/2}} = -\frac{\pi}{64} \cdot \frac{d^2}{t_{1/2}^2}. \tag{15}$$

The accuracy of determining $t_{1/2}$, however, depends not only on the time measurement regime, but also on the accuracy of estimating final mass m_2, so $t_{1/2} = f(m_2)$. The following formula was applied to estimate how the uncertainty in estimating final m_2 affects the uncertainty in $D_{\mathrm{m}}(t_{1/2})$:

$$\frac{\partial D_{\mathrm{m}}}{\partial m_2} = \frac{\partial D_{\mathrm{m}}}{\partial t_{1/2}} \cdot \frac{dt_{1/2}}{dm_2} = -\frac{\pi}{64} \frac{d^2}{t_{1/2}^2} \cdot \frac{dt_{1/2}}{dm_2}. \tag{16}$$

The application of formula (16) required the relationship between time and mass for each of samples and then calculations of derivatives evaluated at the point corresponding to a half mass change.

Analysis of results collected in the above-mentioned way makes it possible to assume similar trends to the ones obtained by other methods. The final mass m_2 that was not entirely stabilized causes the largest uncertainty in $D_{\mathrm{m}}(t_{1/2})$ in the cycle 1d, and affected results of $D_{\mathrm{m}}(t_{1/2})$ in cycle 5d to a smaller extent. Uncertainties for the samples of certain mortar are as follows: the cycle 1d-uncertainty of 3.4–4.4% at $w/c = 0.50$; 5.8–7.4% at $w/c = 0.65$ and 5.5–7.5% at $w/c = 0.80$ and the cycle 5d-uncertainty of 0.3–0.6% at $w/c = 0.50$; 0.3–0.7% at $w/c = 0.65$ and 0.3–0.4% at $w/c = 0.80$, respectively.

The measurement uncertainty related to mass m_2 in cycle 5d (the highest humidity φ considered) is well below 1% in all the three methods, so incomparably small with regard to the scatter of component results. On the other hand the uncertainty related to mass m_2 in cycle 1d (the lowest humidity φ considered) is comparable to the scatter of results. The largest uncertainty values connected with the underestimation of final mass m_2 do not exceed 8% of determined D_m, which makes it possible to approve the accuracy of measurements in cycle 1d—the one affected by the uncertainty of that kind to the largest extent.

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