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Method for simultaneous determination of sorption isotherms and diffusivity of cement-based materials

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Abstract

A method for simultaneous determination of the diffusion and sorption properties of cement-based materials is presented. It is a gravimetric method where one small specimen is exposed to stepwise changes in relative humidity while its mass is being measured. As sorption in cement-based materials is slow, the change in relative humidity to the next level is made before final equilibrium has been reached on the previous level. Approximate final (equilibrium) levels are found by extrapolation using an exponential equation, and a factor is applied to correct for the fact that the sorption step does not start at equilibrium conditions. A correction for external mass transfer resistance is also included. The method can be used in desorption as well as absorption mode. Measurements of two materials are presented and compared with the results obtained using conventional methods.

Keywords: Sorption isotherm; Diffusion; Humidity; Adsorption

1. Introduction

Moisture is an important issue in building science as it influences several important material properties. Moist buildings have also been related to negative health effects in several studies [1]. Moisture related processes that may lead to poor indoor air quality are, for example, microbiological growth [2,3] and alkaline degradation of the polymers in flooring adhesives and floor coverings [4]. Most cementitious materials contain excess water when mixed and this water must dry out to prevent future damage to the building and deterioration of the indoor air quality.

Knowing the moisture properties of materials is essential for calculating drying times and moisture loads as well as for predicting biological growth, chemical reactions and transport of hydroxyl and chloride ions, for example. The two most commonly measured moisture properties are sorption isotherms (equilibrium moisture content as a function of relative humidity) and transport (diffusion) coefficients. These are measured by different techniques: for example, sorption isotherms by equilibrium over saturated salt solutions [5] and diffusion coefficients by cup measurements [6]. These techniques are time consuming and require many parallel specimens as measurements are usually performed in several different RH-intervals.

There are also ways to determine diffusion and sorption properties in one measurement. In the time-lag method, often used for membranes [7], the partial pressure is kept stepwise constant on one side of a specimen and the sorption and diffusion properties are calculated from the measured change in vapour pressure on the other side. In gravimetric methods [8,9], a material with known geometry is exposed to stepwise or continuous changes in vapour pressure. The change in mass of the specimen is continuously measured, giving the sorption isotherm at equilibrium and the diffusion coefficient from the kinetics and the sample geometry. A gravimetric method for evaluation of sorption isotherms and diffusion coefficients in absorption in cement-based materials was presented by Tada and Watanabe [10,11]. In this method absorption steps are made from dry conditions to different relative humidities. Thus, several test specimens are required or a single test specimen has to be dried before each measurement step. Each sample consists of 10 to 15 mm sized particles made from crushed material; the present method uses smaller specimens with a more well-defined

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geometry. For cement-based materials it is also more relevant to do measurements on un-dried specimens in desorption – as in the present method – as most moisture data is used when the material is on the desorption limb of the sorption isotherm, for example for drying calculations. Initial conditioning to a dry state of cement-based specimens may also damage their internal structure.

It is also possible to measure moisture content profiles developed under given conditions and to determine from these the diffusion properties [12]. In such measurements one can also evaluate the sorption isotherm by, for example, using built-in relative humidity sensors or by taking out samples for separate measurements of the relative humidity.

A gravimetric method for small specimens that measures both the sorption isotherm and diffusivity as a function of relative humidity is presented here.

2. Materials

The products tested were commercial self-levelling flooring compounds (SLCs) based on calcium aluminate cement, calcium sulphate, Portland cement and a small amount of silica fume. A typical composition of the mineral part of such an SLC is shown in Table 1. Product A was a normal SLC with water to binder ratio of about 1.0. This product is generally used in non-industrial constructions. Product B was a rapid drying SLC with water to binder ratio of about 0.7. This product is mainly used in renovations as one can walk on it after 1–2 h and apply final coverings in one day.

The materials were cast in short pieces of stainless steel tubes (length 5 mm, inner diameter 5.5 mm, outer diameter 6 mm). In this way a well-defined geometry with one-dimensional moisture flow was obtained. The products were mixed according to the manufacturer's guidelines and cast into the steel tubes that were standing on a plane substrate, and a plane lid was placed on top. The specimens were left to hydrate at 100% relative humidity (RH) and 20 ± 1 °C over four weeks. SLCs are suitable for these kinds of measurements as the maximum aggregate size is small, normally around 1 mm, and the low viscosity makes it easy to cast them into small moulds. For other materials that cannot be cast in this way, it is possible to use a sealant to make the flow one-dimensional.

3. Method

3.1. Experimental set-up

The present method is based on mass determination under stepwise changing RH conditions. To generate the RH steps we placed our specimens in a sorption balance (DVS-1000, Surface Measurement Systems Ltd., UK). Fig. 1 describes the principle of the sorption balance used. A flow of dry nitrogen gas is divided into two gas streams of which one is saturated with water vapour (S in Fig. 1) and the other left dry. The flow rates are controlled by two mass flow regulators (Rw and Rd). By mixing different proportions of the two gas streams, an RH between 0 and 100% can be generated, although practical limits are 0–98% RH. The trueness of the RH is ±2% and the balance resolution is 0.1 µg. The gas flow comes from below and its velocity is 0.4 cm/s. The specimens were freely suspended in the gas flow line, attached to the balance by a stainless steel hook that holds the stainless steel tube with the specimen. The reference holder was built up in the same way as the sample holder, except that the specimen was replaced by a counter mass in the steel tube as the samples used have higher masses than the dynamic range of the balance. The absolute mass of the sample was determined by weighing the steel tube and the dry sample in the steel tube. The sorption isotherm is evaluated from the final equilibrium mass value in each measurement step and the diffusivity is calculated from the kinetics of the mass change. Measurements can be made in both absorption and desorption mode, using either fixed times for each measurement step or a

Table 1
Approximate composition of the mineral part (excluding admixtures) of an SLC

Portland cement	2%
Calcium aluminate cement	15%
Calcium sulphate	6%
Limestone filler (calcite)	30%
Sand (siliceous)	45%
Silica fume	2%

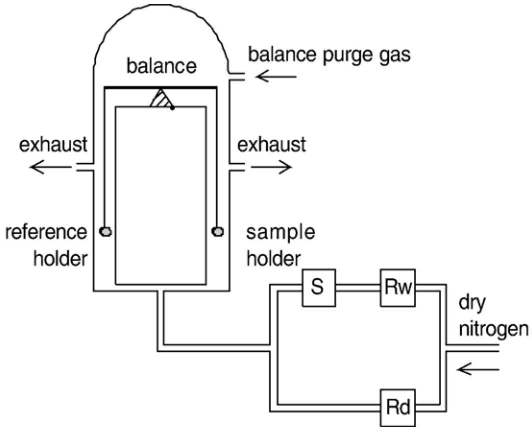

Fig. 1. Schematic representation of the DVS 1000 sorption balance.

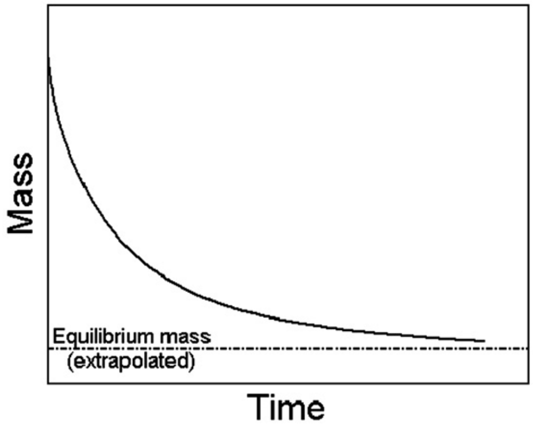

Fig. 2. A typical measurement step where the mass is plotted as a function of time (solid line) together with the extrapolated equilibrium mass (horizontal dashed line).

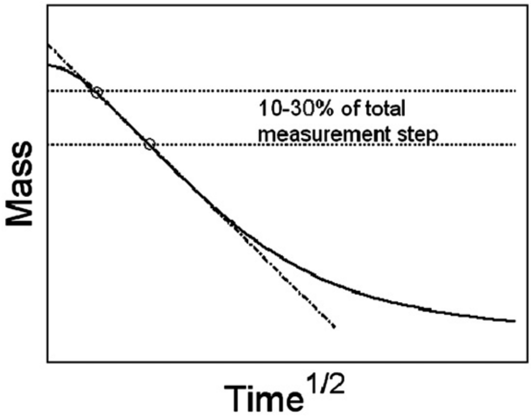

Fig. 3. A typical measurement step where the mass is plotted as a function of the square root of time. The slope of the part that is used for evaluating diffusivity (10–30% of total extrapolated step) is marked.

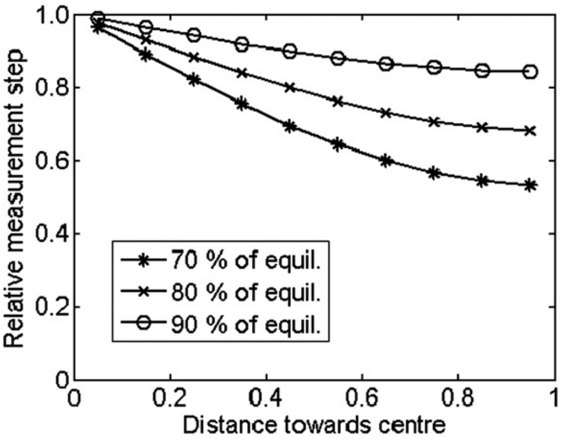

Fig. 4. Simulated moisture profiles when running each measurement step to 70, 80 and 90% of the final equilibrium value with constant diffusivities.

mass change rate criterion for when the measurement shall proceed to the next relative humidity.

The measurements were performed in seven desorption steps starting at 97% RH and ending at 10% RH. One full measurement lasted about 7 days.

3.2. Evaluation of the sorption isotherm

Fig. 2 shows a typical mass response to a step in RH. There is an initial relatively rapid change in mass, but there is also a long and slow attainment of sorption equilibrium. Each measurement step lasted until the specimen had reached a certain E_{crit}, for example 0.9 E, where E is defined as:

$$E = \frac{\Delta m}{\Delta m_{\infty}}. \tag{1}$$

Here, Δm (g) is the mass change at time t (s) and Δm_{∞} is the mass change at equilibrium. The final part of the curve was then curve-fitted and extrapolated using an exponential function to find an approximate equilibrium mass.

As chemically bound water will be lost, partly irreversibly, when drying the cement-based flooring compounds used in this study to 0% RH, the material was not dried further than 10% RH. We have noted a significant mass decrease when drying specimens of this type from 10 to 0% RH, but above 10% RH the sorption isotherms look as expected for a fine porous material. This agrees with what is known about ettringite, the main component of hydrated self-levelling flooring compounds. The ettringite molecule normally contains about 32 water molecules that are progressively lost at low water activities [13].

Results from the present measurements were compared to isotherms measured with a similar sorption balance method on materials cut out from larger specimens [14] to check that the casting in a small mould gave representative samples.

3.3. Evaluation of the diffusion coefficient

As is principally shown in Fig. 3, each step in RH gives an initial mass change that is essentially linear when drawn on a square root of time scale. This linear part can be used to calculate the diffusivity [15,16]:

$$D = \frac{L^2\pi}{16}\left(\frac{dE}{d\sqrt{t}}\right)^2 \tag{2}$$

Here, D (m^2/s) is the diffusivity (a mean diffusion coefficient with moisture content as potential) and L (m) is the thickness of the specimen. The diffusion coefficient with water vapour content as potential, D_v (m^2/s), can then be calculated by:

$$D_v = D\frac{dc}{dv} \tag{3}$$

where D (m^2/s) is the diffusivity from Eq. (2), and $\mathrm{d}c/\mathrm{d}v$ is the change in moisture concentration (g/m^3) in the specimen per change in water vapour content (g/m^3) in the ambient air, i.e. essentially the slope of the sorption isotherm. The final diffusivity was evaluated from the part of the measurement step that corresponded to 10 to 30% of the total change in mass using extrapolated values.

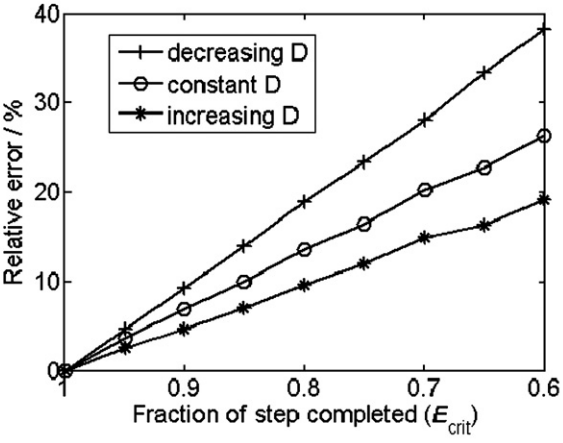

Fig. 5. Simulated errors in desorption mode when aborting measurement steps before final equilibrium is reached using increasing, constant and decreasing values of the diffusivity.

We used steady state cup measurements as a comparison. The specimens were cured in the same way as in the present method. Double specimens were used. A series of measurements with 55% RH outside the cups and different RH values inside the cups were made. The diffusion coefficient as a function of RH could then be calculated [17].

3.4. Error analysis

3.4.1. Delay

Eq. (2) is valid for a step change in RH. In the present measurements this step is not perfect, probably because of sorption on the internal surfaces of the sorption balance giving a slow attainment of the next RH. This gives a non-linear initial part of the curve. An equation given by Wadsö [18] gives a value for the error of −1% for the present type of measurements. This error will not be further discussed.

3.4.2. Non-complete steps

Using Eq. (2) the diffusivity is evaluated from the initial part of each measurement step, and it is strictly valid only when the previous measurement step ends at equilibrium. If not, the initial moisture content will not be constant throughout the specimen and, for example, may be too high for the desorption step resulting in a diffusion coefficient that is too high. In our measurements we deliberately continued with the next step when 80–95% of the previous step had been completed, and the diffusivity in the next measurement step evaluated using Eq. (2) will, therefore, be somewhat erroneous. Fig. 4 gives results of simulations showing the moisture profile after four consecutive step changes, where each step was started when the previous step had only reached 70, 80 or 90% of the final equilibrium value. To compensate for this we have made computer simulations of how the diffusivity evaluated using Eq. (2) changes when each sorption step is allowed to continue only to a certain E_{crit}, for example 0.90. Fig. 5 gives the result of simulations made for increasing, constant and decreasing diffusivities. The increasing and decreasing diffusivities changed linearly by a factor of 2 in each RH interval. The simulation was made for four different evaluation methods previously presented in [19], but the results are shown only for the present method (using the slope between 10 and 30% of the measurement step), which was found to be the best method. The simulations show that if the previous measurement steps were run to 90% of the total equilibrium values, the estimated error would be less than 10% using our method, assuming that the diffusivity does not decrease to less than half the original value in a measurement step.

3.4.3. Mass transfer limitations

The mass transfer coefficient between the material and the air in the sorption balance may influence the results, resulting in lower measured diffusivities than the true values. Crank [15] gives solutions to equations describing the mass increase during sorption steps both with and without an external mass transfer coefficient. Is the simpler solution without external mass transfer limitations, Eq. (2), a good approximation in the present case? In Crank's equations the concentration c (kg/m^3) in the sample is also used as potential for the external mass transfer coefficient k_{c} (m/s):

$$q_{\mathrm{m}} = k_{\mathrm{c}}\left(c_{\mathrm{surf}} - c_{\mathrm{gas}}\right) \tag{5}$$

Here, q_{m} (kg/(m^{2}s)) is the mass flow rate, c_{surf} is the surface moisture concentration and c_{gas} is the moisture concentration that the material would have in equilibrium with the gas humidity. However, when we measure mass transfer coefficients we work with vapour pressure p (Pa) in the gas phase as potential:

$$q_{\mathrm{m}} = k_{\mathrm{p}}(p_{\mathrm{surf}} - p_{\mathrm{air}}) \tag{6}$$

Here, k_{p} has units of kg/(m^{2}sPa). The two mass transfer coefficients carry the same information and have the following relation:

$$k_{\mathrm{p}} = k_{\mathrm{c}}\frac{dc}{dp} = k_{\mathrm{c}}\frac{\rho \cdot dm}{m_{\mathrm{dry}} \cdot p_{\mathrm{sat}} \cdot d\varphi} \tag{7}$$

Here, $(\mathrm{d}m/m_{\mathrm{dry}})/\mathrm{d}\varphi$ is the slope of the sorption isotherm, ρ (kg/m) is the material density, and p_{sat} is the saturation vapour pressure. A problem with this transformation is that the slope of the sorption isotherm is a function of the moisture state, so although k_{p} may be constant in an application, k_{c} will change value as the relative humidity changes.

Crank defines the dimensionless number L as the ratio of internal to external diffusion resistances:

$$L = \frac{hk_{\mathrm{c}}}{D_{\mathrm{c}}}. \tag{8}$$

Here, h (m) is half the thickness of the sample and D_{c} (m^2/s) is the diffusivity. To evaluate L, k_{c} must be calculated from k_{p} using Eq. (7). We can then solve Eq. (5) with a limited external mass transfer and check whether we need to take this into account. We have found that when $L = 10$ the simplified equation can be used with an approximate error of −10% in the evaluated diffusivity for the present materials with the present method. A higher L value gives a lower error and, when $L = 100$, the error is −0.5%.

To evaluate the external mass transfer coefficient k_{p}, we fitted a sponge, saturated with water, into a similar steel tube as is used in our measurements. The steel tube was inserted into the sorption balance and, by measuring the rate of change in mass under a given potential in vapour pressure, k_{p} can be determined

Table 2
Summary of error analysis for two RH ranges giving the highest errors in the present measurements

Case	RH	Non-complete step		Mass transfer limitation		Total error/%
		E_{crit}	Error/%	L	Error/%	
Product A	97–95	0.82	+13	7	−16	−3
	70–50	0.96	+3	90	−0.4	+3
Product B	97–95	0.93	+5	9	−11	−6
	70–50	0.89	+8	150	−0.1	+8

Table 3
Sorption isotherms presented as moisture content per mass at 10% RH

RH %	Material A		Material B	
	Present	Reference	Present	Reference
95	3.2	2.9	5.2	4.6
90	2.4	2.3	4.2	3.7
80	1.8	1.8	2.9	2.7
70	1.3	1.3	2.0	2.0
50	0.41	0.40	0.60	0.65
30	0.18	0.17	0.20	0.24
10	0	0	0	0

The reference material was taken from a larger sample and measured with a similar method.

from Eq. (6). The value was $1.2 \cdot 10^{-7}$ m/s, which corresponds to a 1.5 mm layer of air.

We have calculated the influence of the mass transfer coefficient using data from our measurements. The value of L was about 10 in the measurement range of 97–95% RH, increasing with decreasing RH to over 100 below 50% RH. The error is thus about 10% at 97–95% RH and decreases to about 0.5% at 50% RH.

3.4.4. Summary of error analysis

Table 2 gives examples of values from the error analysis of the measurements we performed. The errors from non-complete steps are dependent on E_{crit}, while the errors caused by mass transfer limitations are dependent on the diffusion coefficient. Measurements performed in intervals with higher diffusion coefficients will thus have larger errors.

To reduce the errors due to non-complete steps, a longer time for each measurement step would be needed. Using a higher gas flow rate or thicker specimens can reduce the mass transfer limitations. If thicker specimens were used, each measurement step would have to equilibrate longer to avoid increasing the error caused by non-complete steps.

4. Results

Table 3 gives the results of the sorption isotherm measurements using a reference mass determined at 10% RH. The results are compared with reference measurements that were performed over four weeks in the same instrument as the present measurements, but on specimens cut out from the centre of a larger specimen [14]. Comparing the sorption isotherm with the reference measurements gives an indication of whether a representative test specimen has been cast into the cylinder. Tables 4 and 5 give the evaluated diffusion coefficients for products A and B. Corrections for the mass transfer coefficient and for not reaching equilibrium in each measurement step have been made. The results are compared with measurements on the same material, cured in the same way, performed with the steady state cup method.

Table 4
Diffusion coefficients for material A measured with the cup method and the present method

Present method		Cup method	
RH %	Diff. coeff., $\cdot 10^{-6}$ m^2/s	RH %	Diff. coeff., $\cdot 10^{-6}$ m^2/s
97–95	7.0	98–96	3.4
95–90	3.5	96–94	4.4
90–80	1.9	94–84	1.7
80–70	0.88	84–75	1.4
70–50	0.48	75–55	0.85
50–30	0.33		
30–10	0.20		

Note the different RH-intervals.

Table 5
Diffusion coefficients for material B measured with the cup method and the present method

Present method		Cup method	
RH %	Diff. coeff., $\cdot 10^{-6}$ m^2/s	RH %	Diff. coeff., $\cdot 10^{-6}$ m^2/s
97–95	5.1	99–97	3.1
95–90	3.1	97–94	5.0
90–80	1.6	94–85	1.4
80–70	0.67	85–75	0.85
70–50	0.27	75–55	0.44
50–30	0.16		
30–10	0.12		

Note the different RH-intervals.

5. Discussion

The sorption isotherms presented in Table 3 show good agreement, except for small deviations at high RH. The deviations may be partly due to the fact that each result is from one measurement only. Other measurements that we have made on specimens of other SLCs and at other ages show similar agreements as the results presented in Table 3. As can be seen in Tables 4 and 5, the diffusion results from the two methods are also in good agreement, except at the highest RH where the cup measured diffusion coefficient decreases when the RH increases. The differences seen may be due to uncertainties in the generation of RH in the two methods. As the increase in diffusion coefficient is steep at high moisture levels for cementitious materials [20], even small errors in the assumed RH will give large deviations. As the cup measurements show the highest values in the second highest RH interval (and lower values in the highest RH interval), this indicates that the assumed RH of one or more of the salt solutions may not be correct. Literature values for the relative humidity produced by saturated salt solutions differ for some salts. We have chosen the values given by Greenspan [21], where, for example, the value for KNO_3 at 20 °C is 94.6% RH while the same value according to Wexler [22] is 93.1% RH. However, the uncertainty in this RH does probably not fully explain the seen discrepancy.

Using narrow RH-intervals where each RH is generated by individual salt solutions has the disadvantage that even slight errors in RH may cause large errors as the salt solutions may deviate from the assumed target values differently. Larger RH-intervals would be needed to reduce such uncertainties, but using larger RH-intervals will, of course, also reduce the usefulness of the results. This problem would not occur to the same extent in a sorption balance where the same device is used to stepwise change the RH. If the actual RH deviates at a target RH of, for example, 97%, the deviation would probably be similar at the next RH level, 95%.

The reference cup measurements of diffusion coefficients took about 8 weeks while the measurements with the present method take about 1 week, including the sorption isotherm. The first step to a well-defined starting RH, e.g. 97%, takes rather a long time as the sorption capacity is high and the RH difference between the specimen and the gas stream is low. It should be noted that even saturated cementitious materials do not have an RH of 100% as the pore solutions contain dissolved salts. A measurement of both desorption and absorption will take about 10 days as absorption measurements generally are faster than desorption measurements (and the starting point for absorption is the end point in desorption).

The present method is very efficient and it also requires less laboratory work than the cup method. However, the equipment used for the present method is more expensive, and it is also only possible to run one test specimen at a time. The size of the specimen may also be a limiting factor in the present method. In the equipment used in this investigation, the maximum mass of each test specimen is 1.5 g (dynamic range 150 mg).

Two advantages with this method are that it is possible to decide the precise intervals you use to measure the diffusivity and sorption properties and that increasing the number of measurement intervals will not involve more test specimens. It is also possible to make measurements in both desorption and absorption, and this will give results on the influence of hysteresis on both sorption and diffusion properties. The short time needed for performing the measurements also makes it easier to study how the measured properties develop in time, which may be of importance for cementitious materials that change properties during hydration.

6. Conclusion

A method for simultaneous determination of sorption and diffusion properties of cementitious materials has been presented. The method only requires one test specimen and can be used in desorption as well as absorption. Extrapolation of the final part of each measurement step gives a rapid measurement procedure. It takes about 10 days to obtain a complete sorption isotherm and diffusion coefficient in both desorption and absorption. Errors due to non-complete steps and external mass transfer limitations are typically less than 10%. The results show good agreement with previously published results.

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