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Date: April 2000



REFERENCE REPRINT

No. 164 (2000)

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From: Building and Environment
Vol. 35 (2000), pp 239-249

Funding for the work reported here was provided by Lissheds Stiftelse.

ISSN: 0111-7459

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A transient technique for determining diffusion coefficients in hygroscopic materials

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Received 8 June 1998; received in revised form 20 October 1998; accepted 2 March 1999

Abstract

A transient technique is described to find the diffusion coefficients of moisture transfer in building materials as a function of moisture content. Measurements that take many months by steady-state methods can be done in days or even hours. The material is subjected to a staircase function of relative humidity steps, the transient weight changes noted, and a numerical algorithm used which extracts the Kirchhoff potential, one value for each relative humidity step and corresponding moisture content. Diffusion coefficients are then calculated from the Kirchhoff potential. Experimental results show fair agreement between Kirchhoff potentials found by steady-state methods and those found by this transient technique, but good agreement between the corresponding diffusion coefficients. An important limitation to the technique at this state of its development is the need to know the material sorption curve. © 2000 Elsevier Science Ltd. All rights reserved.

1. Background

The moisture performance of a hygroscopic material is completely characterised in the hygroscopic regime by specifying its equilibrium moisture curves (sorption curves) and its transport coefficients. Consequently measuring these parameters is an important function, and a very time consuming one, in moisture research.

The usual way to measure equilibrium moisture content is with a steady state approach—the material in question is brought to equilibrium in an atmosphere of a known relative humidity and the weight noted. This may be done through an absorption or desorption cycle or both. On the other hand, various techniques exist for finding transport coefficients. The most common and straightforward technique is to measure at steady-state the moisture flux diffusing through the material [1].

Experimental techniques for finding sorption curves and transport coefficients are very time consuming. Diffusion coefficients in general have a strong dependency on moisture content, requiring measurements to be made at several moisture contents to fully characterise the material. It will usually take several days to several weeks for a material to reach equilibrium at a given relative humidity and until then the equilibrium moisture content cannot be determined—neither can the transport coefficient at that moisture content be determined until this equilibrium has been achieved, if a steady state approach is being used. Furthermore, since measurements must be made at several moisture contents to characterise the material, it is not unusual to consume many months in quantifying a material's moisture properties.

For this reason any technique which offers a faster route to finding material moisture properties is worth closer investigation. Transient techniques are attractive from this point of view because a very wide range of driving conditions can be explored in a relatively short time. However, for most materials the transport coefficients are a strong function of moisture content, which

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Nomenclature

D diffusion coefficient ($\text{m}^2 \text{s}^{-1}$)
 g moisture flux ($\text{kg m}^2 \text{s}^{-1}$)
 t time (s)
 w moisture content (kg m^{-3})
 W absorbed water (kg)
 x distance (m)

δ accuracy of weighing
 ε accuracy of D
 φ relative humidity (%)
 τ dimensionless time step
 ψ Kirchhoff's flow potential ($\text{kg m}^{-1} \text{s}^{-1}$)

means the interpretation of transient results is not straightforward.

For a constant diffusion coefficient the extraction of the moisture content driven diffusion coefficient, D_w , from that slope of the moisture uptake curve against the square of time, \sqrt{t} , is well known. Crank [2] describes some techniques for evaluating variable diffusion coefficients transiently using graphical techniques. A transient method by Duda and Vrentas [3] is also cited in Crank requiring experimentally a sorption-time curve and its derivative.

This paper develops a numerical algorithm, which allows the evaluation of transport coefficients as a function of moisture content from transient data, specifically from the weight uptake of a specimen under a staircase external driving force.

Under most conditions, depending upon the accuracy required, very considerable measurement time gains over steady-state methods can be made using this technique, up to factors of 1000, e.g. measurements that may have taken a year in steady-state can be completed in 1 day by the use of this algorithm. The use of this technique is demonstrated experimentally.

However, it is important to state that at this stage of the development of this transient approach it is necessary to know the sorption curve of the material which, presumably, would have been found by a steady-state technique.

2. Theory

2.1. Kirchhoff potential

The algorithm developed in this work uses the Kirchhoff potential [4] devised to simplify the differential equation of diffusion in the case where the diffusion coefficient is a function of moisture content.

The Kirchhoff potential is developed as follows.

Let g ($\text{kg m}^{-2} \text{s}^{-1}$) denote moisture flux and w (kg m^{-3}) moisture content mass by volume. In the case of one-dimensional isothermal moisture transport we have

$$g = -D_w \frac{\partial w}{\partial x} \quad (1)$$

Kirchhoff introduced a potential defined as [4]

$$\psi = \int_{w_{\text{ref}}}^w D_w dw \quad (2)$$

The reference value w_{ref} can be chosen arbitrarily. At the reference value, ψ_{ref} is set to zero i.e.

$$\psi(w_{\text{ref}}) = \psi_{\text{ref}} = 0 \quad (3)$$

The diffusion coefficient D_w is from (2) the derivative of the Kirchhoff potential ψ with respect to w , i.e.

$$D_w = \frac{d\psi}{dw} \quad (4)$$

We now have, from Eqs. (1) and (4)

$$g = -D_w \frac{\partial w}{\partial x} = -\frac{d\psi}{dw} \frac{\partial w}{\partial x} = -\frac{\partial \psi}{\partial x} \quad (5)$$

i.e. with this transformation, moisture flux becomes simply the gradient of the Kirchhoff potential ψ .

The moisture balance equation has the following simple form:

$$\frac{\partial w}{\partial t} = -\frac{\partial}{\partial x}(g) = \frac{\partial^2 \psi}{\partial x^2} \quad (6)$$

A more complete presentation of the use of the Kirchhoff potential can be found in Arfvidsson and Claesson [5].

2.2. Steady-state measurement of the Kirchhoff potential

In the case of a material of finite thickness Δx Eq. (5) can be written as

$$g = -\frac{\Delta \psi}{\Delta x}$$

i.e.

$$\Delta \psi = -g \Delta x \quad (7)$$

ψ is set to zero at an arbitrary moisture content and corresponding relative humidity. Steady-state measure-

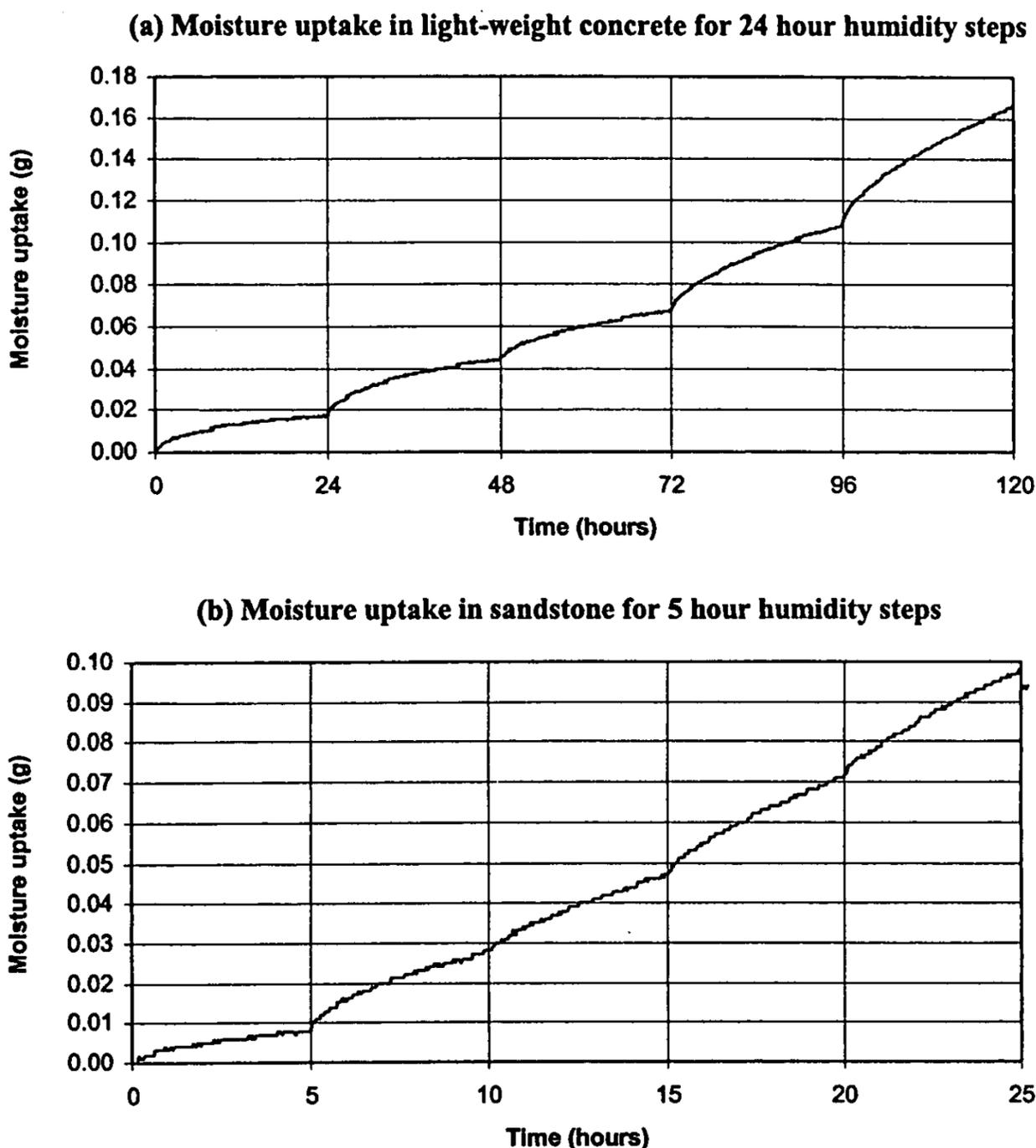


Fig. 1. Typical experimental runs showing moisture uptake in specimens subjected to steps of relative humidity. (a) Light-weight concrete with 24 h humidity steps. (b) Sandstone with 5 h relative humidity steps.

ments of the Kirchhoff potential are now done by establishing a steady-state moisture gradient across the specimen for successively higher mean moisture contents. Eq. (7) then states that at each new mean moisture content the new Kirchhoff potential is given by

$$\psi_n = \psi_{n-1} + |g|l \quad (8)$$

where ψ_i is the Kirchhoff potential at the i th step and l is the thickness of the specimen; in other words the increment of Kirchhoff potential is given by the thickness of the specimen multiplied by the flux of moisture through it.

2.3. The transient experimental method and numerical algorithm

Consider the case of one-dimensional moisture transport in a plane sheet of thickness l . The initial condition is taken as a uniform moisture content and

a staircase moisture content is imposed on one boundary. No flow is specified at all other boundaries. The transient moisture uptake is observed and, from this, the moisture transport coefficient is found as a function of moisture content (Fig. 1).

Mathematically, the problem to be solved is Given

$$\frac{\partial w}{\partial t} = \frac{\partial^2 \psi}{\partial x^2},$$

and given

$$W(t) = \int_0^l w \, dx$$

with the initial condition $w(x, 0) = w_0$ and boundary condition $w(0, t)$ solve for $\psi = \psi(w)$.

It is assumed that the sorption equilibrium moisture curve, $w = w(\varphi)$, is known.

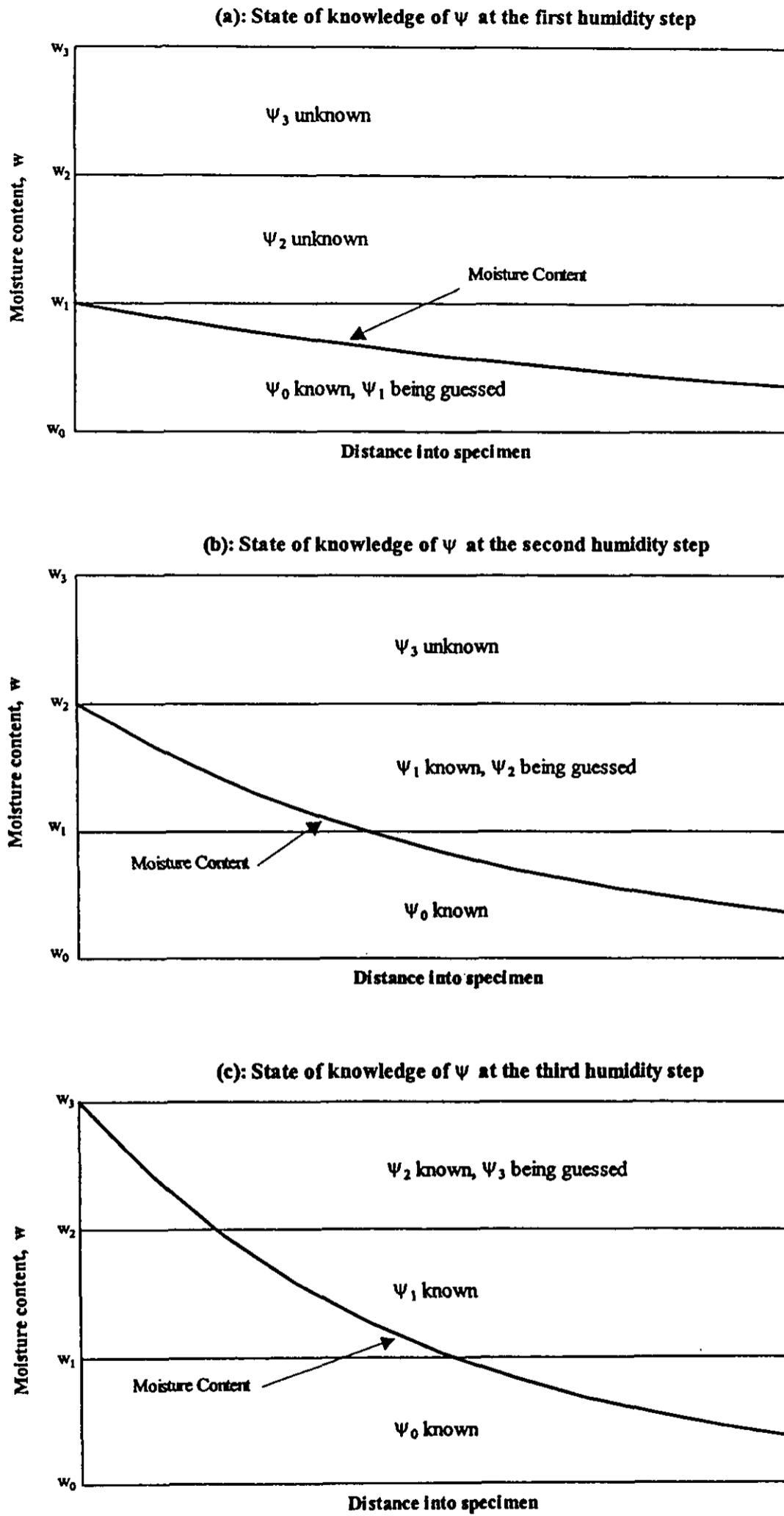


Fig. 2.. These diagrams indicate how, at each driving humidity step, previously found Kirchhoff potentials are used and the next potential ψ_i is guessed. Between w_i and w_{i+1} the Kirchhoff potential used is a linear interpolation between the potentials ψ_i and ψ_{i+1} .

Once the Kirchhoff potential is calculated, the diffusion coefficient D_w can be calculated from Eq. (4).

We start from a uniform moisture distribution w_0 which is known at the corresponding relative humidity, φ_0 . The relative humidity is then increased in a stepwise fashion, being held constant for a period of time at each step, and the weight changes of the specimen during each step is registered.

Between each step a linear relationship is assumed between the moisture content and the Kirchhoff potential, so between step $i - 1$ and i we have

$$\psi(w) = \psi_{i-1} + \frac{w - w_{i-1}}{w_i - w_{i-1}}(\psi_i - \psi_{i-1}). \quad (9)$$

For the first step ψ_0 is set to zero, and ψ_1 is found by trial and error. While doing this first step, the particular case of Eq. (9) will be

$$\psi = \frac{\psi_1(w - w_0)}{w_1 - w_0}. \quad (10)$$

The weight change is known (since measured) as the moisture content in the material rises in response to the external relative humidity step from φ_0 to φ_1 . The value of the Kirchhoff potential ψ_1 corresponding to the equilibrium moisture content w_1 at the driving humidity φ_1 is found by trial and error. In this process ψ_1 is guessed and a theoretical weight change is calculated using a finite difference numerical implementation of the diffusion Eq. (6). Known will be the boundary condition φ_1 and, from the sorption curve, the corresponding equilibrium moisture content w_1 , see Fig. 2(a). The guessed value of ψ_1 is adjusted until the resulting integrated moisture distribution gives a weight change equal to the measured weight change.

In finding ψ_1 the moisture distribution within the material after the first relative humidity step is also found. Knowing the moisture distribution makes it possible to go on to next step change of relative humidity, φ_2 , see Fig. 2(b). ψ_2 at equilibrium moisture content w_2 corresponding to the driving humidity φ_2 is found by trial and error in almost the same way as ψ_1 was found. The only difference is that in the part of the specimen where the relative humidity is still between the initial value φ_0 and the first value φ_1 the now known relation between w and ψ is used in the calculation. In the region where w is greater than w_1 Eq. (9) is used with the guessed value for ψ_2 . In this way it is possible to proceed stepwise, using newly found correspondences (w_i, ψ_i) and measured weight changes to get the next correspondence, (w_{i+1}, ψ_{i+1}) , see Fig. 2(c).

The diffusion coefficient is derived using

$$D_w = \frac{d\psi}{d\varphi} \frac{d\varphi}{dw} \quad (11)$$

where $d\psi/d\varphi$ is obtained directly from the experimental results of Kirchhoff potential ψ vs relative humidity, φ , and $d\varphi/dw$ is obtained as the inverse of the slope, $dw/d\varphi$, of the known material sorption curve.

If the diffusion coefficient for vapour pressure driving potential is required, then this can be found by the standard transformation

$$D_p = \frac{\partial m}{\partial p} D_m. \quad (12)$$

2.4. Sensitivity

Given that the method described here is a transient technique, it is important to know how long the transient should be allowed to last, i.e. what proportion of moisture uptake is required compared to the steady-state value, before a final weighing is made at each relative humidity step and the next relative humidity step begun.

The time to be allowed before each final weighing is determined by the required accuracy of the diffusion coefficient and the accuracy obtainable with the weight measurement. An idea of the time required can be gained by analysing the sensitivity at the first step when the initial moisture content distribution is uniform. For small time, specifically for $W/W_\infty < 0.5$ corresponding approximately to $t < 0.005 l^2/D_w$ we have for the solution to

$$\frac{\partial w}{\partial t} = \frac{\partial}{\partial x} D_w \frac{\partial w}{\partial x} \quad (13)$$

the following [2]

$$\frac{W}{W_\infty} \cong \frac{4}{\pi^{1/2}} \left(\frac{D_w t}{l^2} \right)^{1/2} \quad (14)$$

so

$$\frac{1}{W_\infty} \frac{\partial W}{\partial D_w} \cong \frac{1}{\pi^{1/2}} \left(\frac{4t}{l^2 D_w} \right)^{1/2} \quad (15)$$

i.e.

$$\frac{\Delta D_w}{D_w} \cong \left(\frac{\pi l^2}{4 D_w t} \right)^{1/2} \frac{\Delta W}{W_\infty} \quad (16)$$

i.e.

$$\frac{\Delta D_w}{D_w} \cong \left(\frac{\pi t_0}{4t} \right)^{1/2} \frac{\Delta W}{W_\infty} = \left(\frac{\pi}{4\tau} \right)^{1/2} \frac{\Delta W}{W_\infty} \quad (17)$$

where

$$t_0 = l^2/D_w \quad \text{and} \quad \tau = t/t_0. \quad (18)$$

This expression shows that the error in the calculated

Table 1

Uncertainty in diffusion coefficient given the uncertainty in weighing, as a function of the length of the time step (constant D and initial moisture distribution uniform)

Dimensionless time ($\tau = Dt/l^2$)	Actual time ($D = 10^{-10} \text{ m}^2 \text{ s}^{-1}$, $l = 40 \text{ mm}$)	Fraction of moisture uptake (W/W_∞)	Uncertainty multiplier ($(\Delta D/D)/(\Delta W/W_\infty)$)	Uncertainty in D if weighing is accurate to 0.1%
0.05	220 h	0.51	4.5	0.45%
0.01	44 h	0.23	10	1.0%
0.005	22 h	0.16	14	1.4%
0.001	4.4 h	0.071	32	3.2%

diffusion coefficient, not surprisingly, becomes large if a short time period is used at a given relative humidity level. Fortunately, this time dependence goes as the inverse square root which is not strong. Although this result has been derived only for the first relative humidity step where the initial distribution is uniform, numerical experimentation confirms that a similar relationship also holds for later relative humidity steps. Table 1 contains examples of the diffusion coefficient uncertainty for the initial relative humidity step, compared to the weighing accuracy, for a range of times.

In practice, if weighing is done to 1 part in 1000, a time step of a few hours might typically represent a τ value of around 0.001, and therefore give rise to an uncertainty in D_w of around 3%.

2.5. Time improvement

Once the degree of precision required in the diffusion coefficient, D_w/D_w , has been specified, then the minimum length of each (dimensionless) time step, $\tau_{\text{transient}}$, required can be estimated using formula (18), i.e.

$$\tau_{\text{transient}} = \frac{\pi}{4} \left(\frac{\Delta W}{W_\infty} / \frac{\Delta D_w}{D_w} \right)^2 = \frac{\pi}{4} \left(\frac{\delta}{\varepsilon} \right)^2 \quad (19)$$

where $\delta = \Delta W/W_\infty$ is the accuracy of the weighing and $\varepsilon = \Delta D_w/D_w$ is the required accuracy in the value of D_w .

This can be compared to the time, τ_{ss} , that would be

required to reach the same precision under a steady-state method.

In the steady-state method one measures the flux flowing through the system at equilibrium and infers the diffusion coefficient from the steady-state formula

$$g_\infty = D_w \frac{w_2 - w_1}{l} \quad (20)$$

where g_∞ is the steady-state flux ($\text{kg m}^{-2} \text{ s}^{-1}$).

Before steady-state, some of the flux is being used to fill or empty storage within the specimen. Crank gives a formula [2] for the approach to equilibrium of a plane specimen subjected to a step change in external moisture concentrations (starting from an initial uniform moisture content distribution). For large t Crank's formula yields

$$\frac{g_\infty - g}{g_\infty} \cong \frac{8}{\pi^2} \exp\left(-\frac{D_w \pi^2 t}{l^2}\right) \quad (21)$$

where g is the flux at time t ($\text{kg m}^{-2} \text{ s}^{-1}$).

If a steady-state value of D_w is to be found with a required accuracy of better than ε then the above expression must be less than this at time τ_{ss} , i.e.

$$\frac{8}{\pi^2} \exp\left(-\frac{D_w \pi^2 t}{l^2}\right) = \frac{8}{\pi^2} \exp(-\pi^2 \tau_{\text{ss}}) < \varepsilon. \quad (22)$$

Hence, at least

$$\tau_{\text{ss}} > \frac{\ln(8/(\pi^2 \varepsilon))}{\pi^2} \quad (23)$$

Table 2

Proportionate gain in measurement time of the transient technique over the steady-state technique

Accuracy required of D (ε)	Accuracy in weighing (δ)	Proportionate gain in measurement time	$D = 10^{-11} \text{ m}^2 \text{ s}^{-1}$, $l = 10 \text{ mm}$	
			Time for each steady-state measurement	Time for each transient measurement
0.01	0.001	47	52 days	0.91 days
0.01	0.01	0.47	52 days	91 days
0.05	0.001	760	33 days	0.87 h
0.05	0.01	7.6	33 days	3.6 days

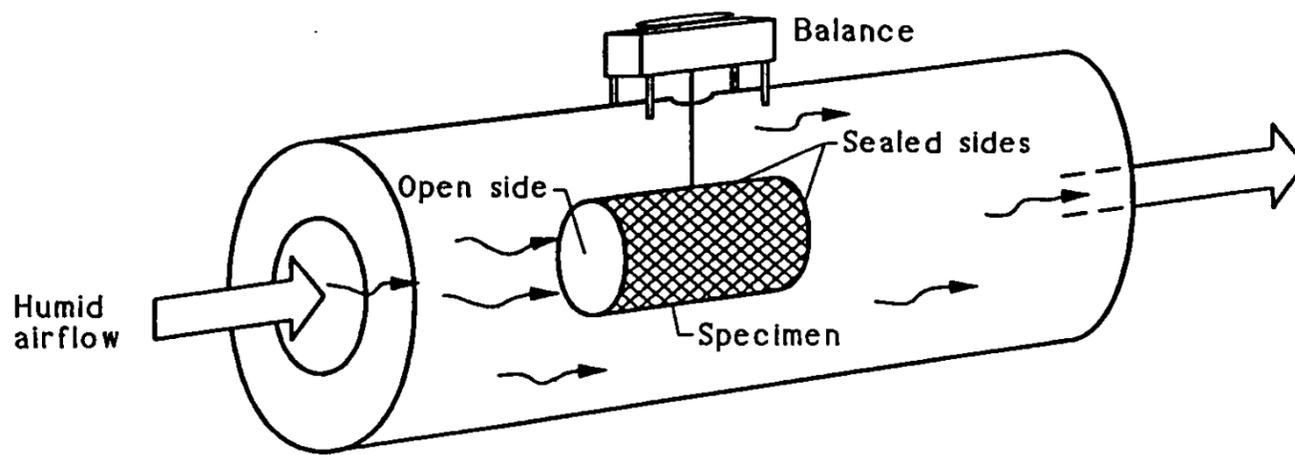


Fig. 3. The experimental set-up. The cylindrical specimen was placed in a tube connected to a 'Thunder 2500' humidity generator that provided an airflow of a specific humidity and temperature through the tube. The end and the curved sides of the specimen were sealed leaving only top of the specimen facing the airflow open to moisture transfer.

to achieve a steady-state measure to the required degree of accuracy.

Now the ratio of expression (23) to expression (19) gives the factor of gain in measurement time in using a transient technique over a steady-state technique, i.e.

$$\begin{aligned} \text{Proportionate gain in time} &= \frac{\tau_{ss}}{\tau_{transient}} \\ &= \frac{4}{\pi} \left(\frac{\epsilon}{\delta\pi} \right)^2 \ln(8/(\pi^2\epsilon)) \cong \frac{1}{\pi^2} \left(\frac{\epsilon}{\delta} \right)^2 \ln(1/\epsilon). \end{aligned} \quad (24)$$

For example, if weighing is done to 1 part in 1000 and the required accuracy in D_w is 5%, then the transient method offers a measurement time factor gain of 760. Table 2 shows the proportionate gain in measurement time for a range of parameters.

It is worth stressing that these minimum times required for acceptable accuracy for both the steady-state and transient measurements represent the fundamental limitation of these procedures, being set by sample geometry and uncertainty in weighing. Experimental uncertainties such as variability of the driving humidity and variability in material properties will add their own uncertainties to any results obtained.

3. Experimental

To show the method in use a number of experimental runs were undertaken. Cylindrical specimens with a diameter of 30 mm and length 40 mm were used. Three specimens were drilled out from a large deposit of sandstone from Uddvide on the Swedish island Gotland and another three specimens were drilled out from a block of lightweight concrete. A one-dimensional flow system was established by sealing all sides of the specimens except one flat face. The samples were conditioned at 35% relative humidity and 20°C for 1 month. Each specimen was then subjected to a five step relative humidity driving regime in absorption in a controlled climate chamber connected to a 'Thunder 2500' humidity generator, see Fig. 3. The temperature in the controlled climate chamber was held at 19.5°C ± 0.5°C. The relative humidities used were 50, 70, 80, 90 and 95% to within 1% relative humidity. Three different length in time steps were used on each material, 1, 5 and 24 h, making 6 runs in all. High surface air velocities were used to guarantee that surface mass transfer was not a controlling factor. The specimens were weighed continuously, and the weight data-logged at every two minutes for the 1 and

Table 3
Experimental runs undertaken

Run number	Material	Levels	Time step per level	Total time	Uncertainty in D_m (Eq. (14))
1	Sandstone	5	1 h	5 h	7%
2	Sandstone	5	5 h	25 h	3%
3	Sandstone	5	24 h	120 h	1%
4	L.W. Concrete ^a	5	1 h	5 h	7%
5	L.W. Concrete	5	5 h	25 h	3%
6	L.W. Concrete	5	24 h	120 h	1%

^a Light weight concrete.

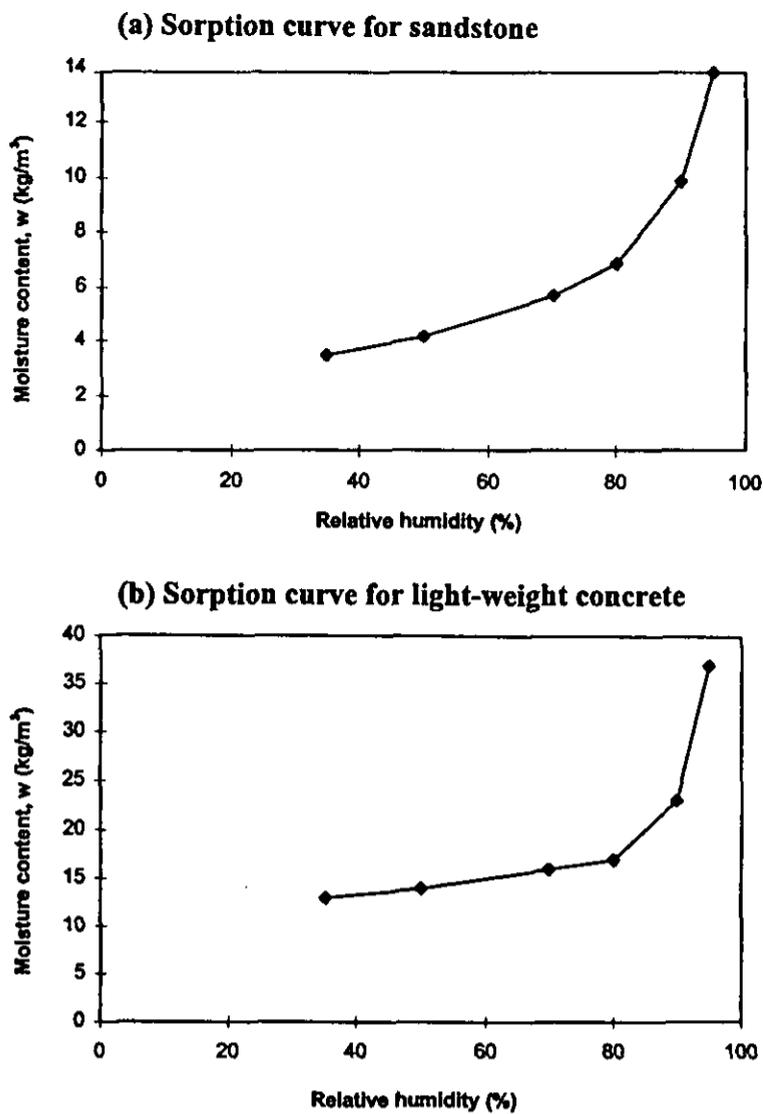


Fig. 4. Hedenblad's data [6] for the sorption curves for the sandstone and light-weight concrete studied in this work.

5 h steps, and every 5 min for the 24 h steps. Humidity was controlled to $\pm 1\%$ relative humidity and weighing was made to an accuracy of 1 g in 1000 g. The time constant of response of the controlled climate chamber was 3 min (Table 3).

Hedenblad's data [6] describing the sorption curve for these materials, see Fig. 4, was used for the calculations.

4. Results and discussion

The calculated Kirchhoff potentials and diffusion coefficients are shown in Figs. 5 and 6, together with steady-state measurements made by Hedenblad [6]. Hedenblad's steady-state measurements took about four months where as only 5 h were needed for the case of 1 h relative humidity steps.

It can be seen that in both the sandstone and the concrete the Kirchhoff potential begins to deviate quite significantly from the steady-state measurement at higher moisture contents—in the case of sandstone the steady-state derived Kirchhoff potential is lower than

the transient derived values, and the concrete case it is higher. However, when the Kirchhoff potentials are converted to diffusion coefficients the difference between the steady-state derived and transient derived values for these coefficients becomes much less. This is because the Kirchhoff potential is an integrated quantity of the diffusion coefficients, see Eq. (2), so that a consistently high or low value for the diffusion coefficient results in an accumulating error in the calculated Kirchhoff potential as the moisture content increases. Conversely, it is the slope of the Kirchhoff potential against moisture content that determines the value of the diffusion coefficients, and as can be seen in Figs. 5 and 6, the transient Kirchhoff potential slopes are not much different from the steady-state slope.

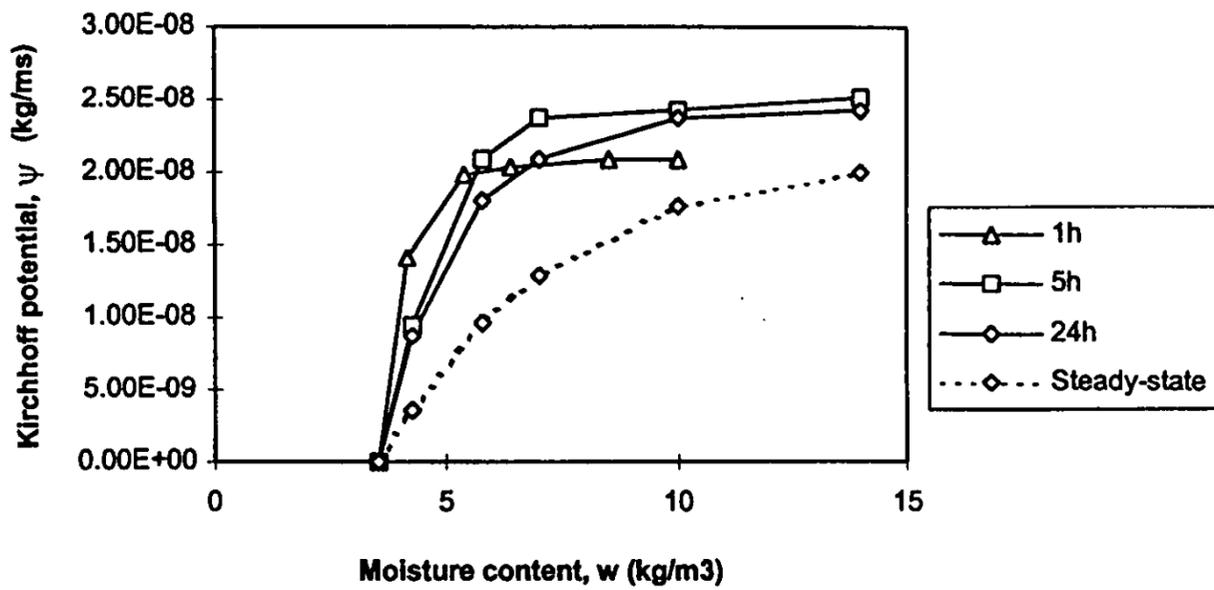
Countering this tendency for error accumulation in the calculation of the Kirchhoff potential as moisture content increases is the operation of a negative feedback mechanism in the algorithm. If at one humidity step the algorithm estimates a Kirchhoff potential that is too high (low), this high (low) estimate would cause the calculated weight increase at later humidity steps to be high (low), since the moisture will enter the material more (less) easily if the Kirchhoff potential were higher (lower). This forces the algorithm to compensate by estimating later Kirchhoff potentials that will be a little too low (high) in order to correctly match the new measured weight increases. In that way the calculated Kirchhoff potential versus moisture content curve will tend to be pulled back to the correct values.

It is encouraging that the Kirchhoff potentials calculated for transients with widely different step times are in close agreement. The fact that the difference between steady-state derived and transient derived Kirchhoff potential results for sandstone and for concrete are of opposite signs suggests that there are no systematic errors being introduced by the transient method. Neither, see above, is the increasing difference at higher moisture contents surprising—however, it is clear that the Kirchhoff potential values calculated at high moisture contents using the transient method are less reliable. In the cases studied here, this does not reflect into unreliable values for the diffusion coefficients at higher moisture contents.

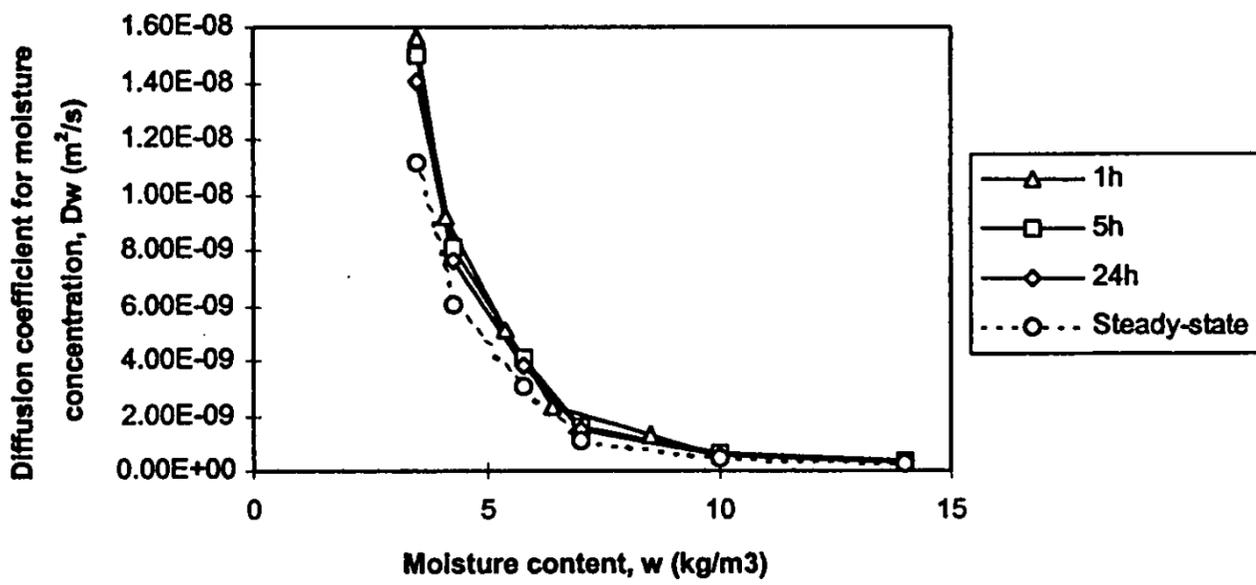
5. Conclusions

A transient technique for measuring variable diffusion coefficients has been described and demonstrated. The method utilises a staircase relative humidity driving function and calculates, from measured weight increases, the Kirchhoff potential and diffusion coefficients at the moisture level corresponding to each relative humidity step. This transient technique shortens

(a) Various measurements of sandstone Kirchhoff potential, ψ



(b) Various calculated values for D_w for sandstone



(c) Various calculated values of D_p for sandstone

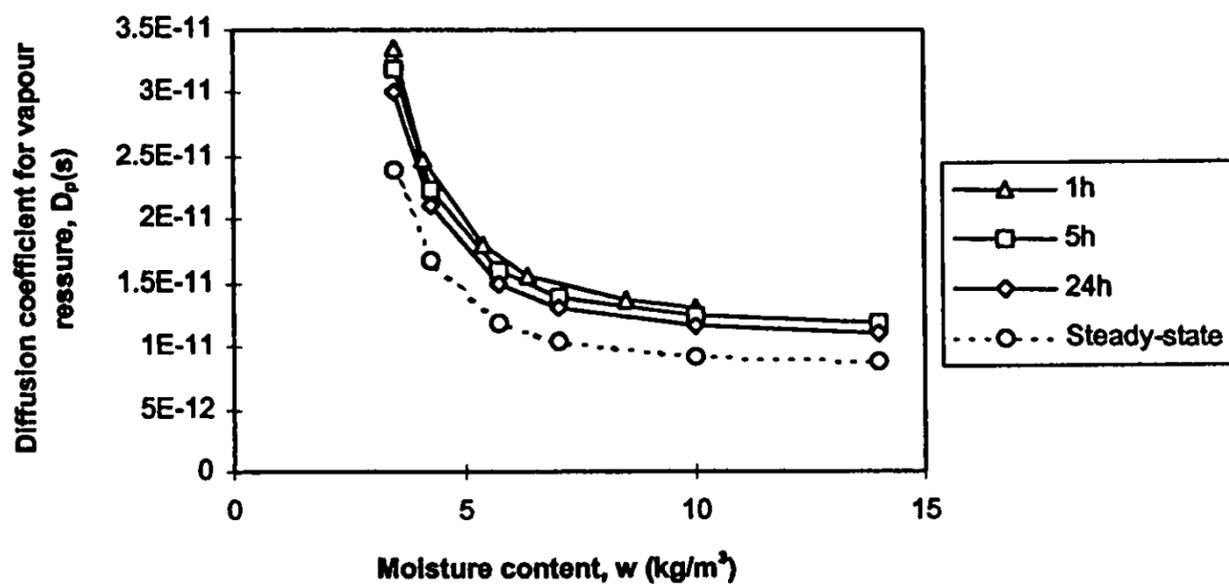
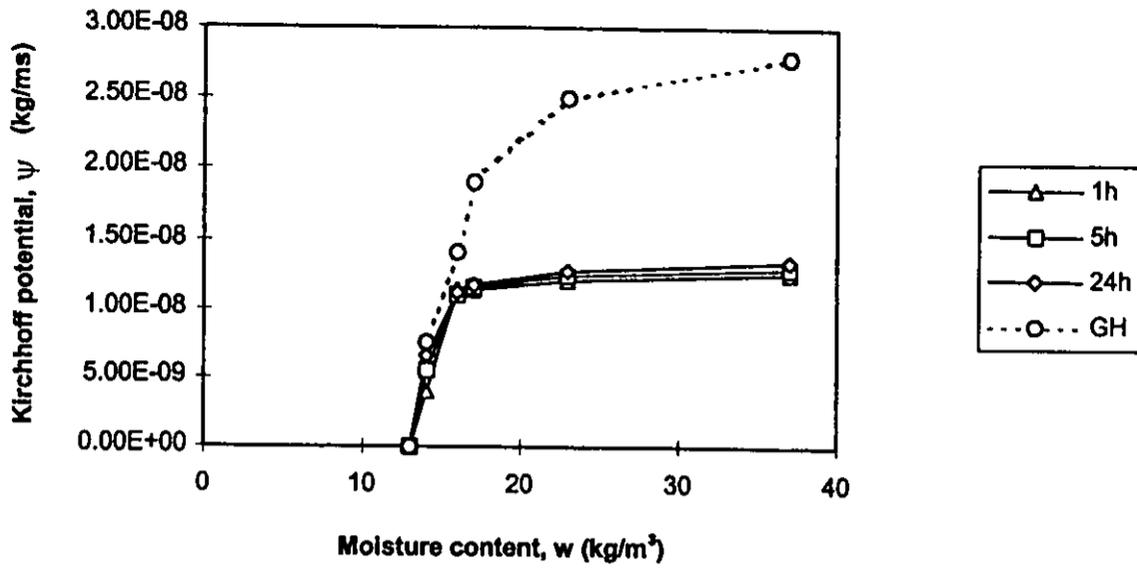
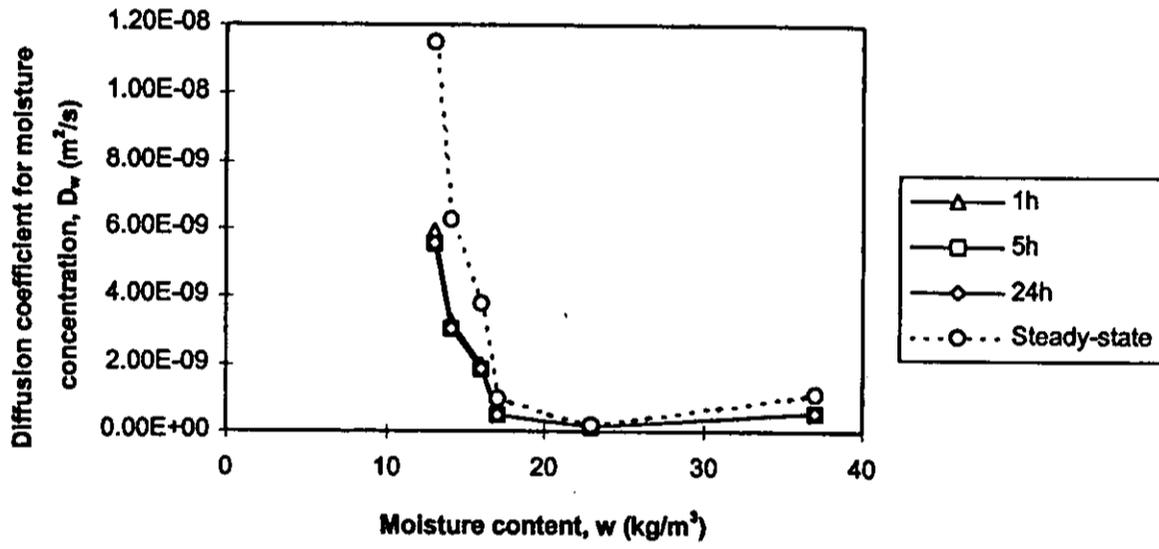


Fig. 5. (a) Sandstone experimentally determined Kirchhoff potentials for different transients, and for steady-state. (b) Corresponding calculated moisture concentration diffusion coefficients. (c) Corresponding calculated vapour pressure diffusion coefficients.

(a) Various measurements of light-weight concrete Kirchhoff potential ψ



(b) Various calculated values for D_w for light-weight concrete



(c) Various calculated values of D_p for light-weight concrete

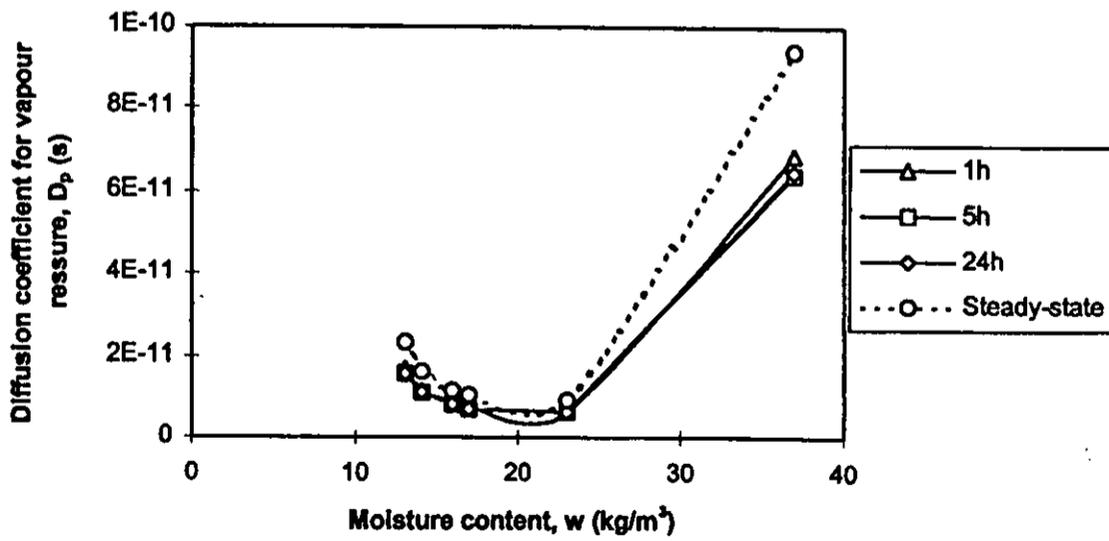


Fig. 6. (a) Light-weight concrete experimentally determined Kirchhoff potentials for different transients, and for steady-state. (b) Corresponding calculated moisture concentration diffusion coefficients. (c) Corresponding calculated vapour pressure diffusion coefficients.

the total time taken to find the diffusion coefficients as a function of moisture content, in some cases very substantially. This work showed examples where measurements that took four months using a steady-state technique were completed in 5 h using 1 h relative humidity steps.

The method here has been confirmed to the hygroscopic region. In principle it can be also used in the capillary region, because the technique is fundamentally a change in variable. There is, however, a significant limitation to the technique in that the sorption curve of the material is needed, which would have been found by steady-state methods. It is not possible from the data to estimate approximately the sorption isotherm values because we are very far from steady-state. It is intended to show in a later paper how to extend this technique to the simultaneous evaluation of the sorption curve of the material, but preliminary work in this more demanding case suggests that much smaller measurement time gains can be realised, perhaps up to a factor of 3.

Acknowledgements

Agneta Ohlsson is thanked for work to make the experimental set-up a functional one. Grants from Lissheds Stiftelse are acknowledged, making this collaboration between New Zealand and Sweden possible.

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