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Water vapour and liquid permeability measurements in cementitious samples

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This paper shows how water permeability may be calculated from measurements of drying under a vacuum. The results obtained are for water vapour transport at low pressures and gas slippage theory is then used to compare them with liquid water permeability measurements on samples of the same mixes. The experimental work includes the drying procedure and also the initial surface absorption test (ISAT), a standard absorption test using a partially immersed sample and a 'through flow' high-pressure test for direct permeability measurements. The results from these tests are compared and additional gas and liquid permeability data from the literature is included for comparison. It is concluded that, with appropriate analysis, all of the procedures give comparable values for intrinsic permeability.

Notation

α	porosity
b, β_1 and β_2	constants for the Klinkenberg equation
Θ	cross-sectional area through which the transport is taking place (m^2)
e	viscosity in (Pa s)
F	flux (m^3/s)
h	head of water (m)
k	coefficient of permeability, also known as the hydraulic conductivity (m/s)
K	intrinsic permeability (m^2)
K_g	gas intrinsic permeability of concrete (m^2)
K_l	water intrinsic permeability of concrete (m^2)
K_v	intrinsic permeability for the vapour (m^2)
M	mass (kg)
P	pressure (Pa)
P_m	mean pressure at which gas is flowing (atmospheres)
r	typical pore radius (m)
ρ	density of liquid water (kg/m^3)
s	surface tension of water (N/m)
t	time (s)
V	Darcy velocity for the fluid (m/s)
W_F	ratio of the water vapour volume to the volume of the same mass of water as a liquid
x	distance (m)

Introduction

Permeability is one of several key properties which are indicators of durability. The mechanisms which cause permeability to affect durability are described by Claisse[1] and include the transport of chlorides dissolved in water causing corrosion of steel reinforcement. There are numerous methods available to measure it but some actually measure other properties which are assumed to correlate with permeability and are therefore indirect (such as the the 'rapid chloride permeability test',[2] which measures electromigration) and few of the others actually yield results for permeability itself. For example the initial surface absorption test (ISAT),[3] which measures both absorption and permeability is useful for comparing materials but the standard report from the test gives an ISAT value, not a permeability. Similarly the water penetration test in EN12390-8[4] records penetration depth, not permeability. The advantages of knowing the permeability in standard units are listed here.

(a) The results from one test can be compared with another.
(b) The results can be used in theoretical work to

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calculate durability of structures using, for example, finite element modelling of the transport processes.

(c) The permeability must be known in order to calculate the performance of structures in applications such as waste containment.[5] Indirect measurements of permeability are of little use for this.

The literature on the permeability of concrete is extensive. Previous work by the authors has been reported[6–9] to compare standard tests by calculating permeability values from them. The objective of this paper was to analyse an experiment which measures a permeability for water vapour and demonstrate that this may be related to gas and liquid permeabilities obtained using other test procedures. Water vapour transport is a key process in many durability-related processes such as carbonation. The permeability itself is a macroscopic property and will be controlled by other microscopic properties such as connectivity, tortuosity and pore size distribution and uniformity.

Research significance

This work is intended to give an improved understanding of the transport mechanisms that take place during laboratory testing. The particular emphasis of the work is to show how the fundamental property of permeability may be obtained and also to focus on vapour transport during drying as a means to measure it. The work will be of interest to researchers who are measuring or modelling durability or to practitioners who are designing containment structures for fluids or waste materials and need to know the permeability as part of the design. The analysis methods which are presented may also be used to confirm the reliability of any individual test.

Experimental methods

Four mortar mixes were used in this investigation and the mixture proportions and strengths are shown in Table 1. The test specimens (dimensions are detailed in each test procedure) were cast and kept under wet hessian for 1 day before demoulding. All samples were then cured in water at 20°C until testing at 28 days. All testing was carried out at 20 ± 2°C.

Drying test

Samples were cast in cylindrical moulds 30 mm diameter by 50 mm long. The moulds were laid on their sides for setting in order to keep their ends under identical conditions. After curing, polyvinyl chloride adhesive tape was wound round the curved surfaces of the cylinders to prevent moisture evaporation except from the top and bottom ends (Fig. 1). The cylinders were then placed in a glass desiccator connected to a vacuum pump with a 0–25 mbar (0–2·5 kPa) pressure gauge on it. The vacuum pump was run until the air in the desiccator became dry, as indicated by the colour change of silica gel in the desiccator (this took up to 30 h). The colour changes at a water content of 8% by mass, which occurs at a humidity of 15% at atmospheric pressure, i.e. a partial vapour pressure of 0·3 kPa.

The vacuum pressure was monitored during the drying process. At different times samples were taken out of the desiccator and weighed and then split down the axis and the depth of drying at each end was measured visually. No precautions were taken to prevent carbonation of the samples but since the experiments only lasted for a few days the weight gain from this process was not considered to be significant.

In addition to these tests some specimens were dried in an oven for 3 days at 105°C to calculate porosity.

High-pressure permeability test

The water permeability was measured in a modified Hoek cell manufactured by ELE International, USA.
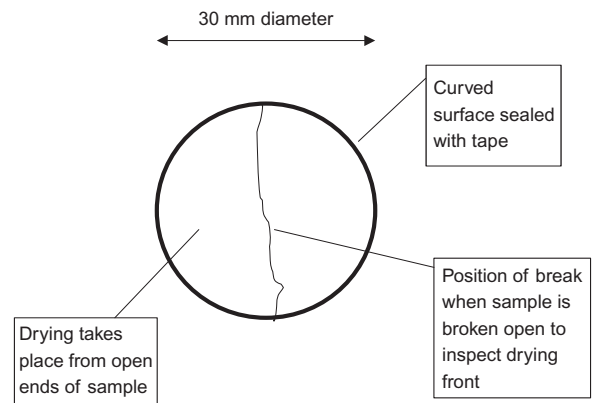

Fig. 1. End view of sample from drying test

Table 1. Mortar mix proportions

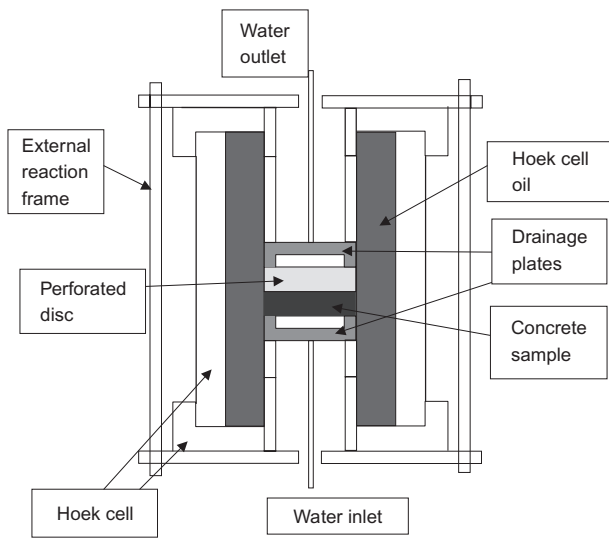| Mix	Design 28 day cube strength: MPa	OPC: kg/m³	Sand: kg/m³	Water/cement
A	20	449	1411	0·79
B	35	544	1342	0·65
C	50	679	1235	0·53
D	60	943	108	0·38

Fig. 2 Modified Hoek cell for permeability measurements

The cell and the modifications are shown in Fig. 2. All of the components identified in the figure except the cell and the oil are modifications for concrete permeability testing. Specimens were cast as 100 mm cubes and 55 mm diameter cores were cut through them and approximately 40 mm long samples were cut from the central part of the cores. The apparatus comprised a stainless-steel triaxial cell in which oil was used to apply pressure to the curved surface of the specimens through a rubber sleeve. The test method was based on feeding the water to the lower surface of the specimens at high pressure (6–8 MPa) while the oil pressure was maintained about 1 MPa higher to prevent flow around the specimens. Due to the high pressures used the flow through the samples became constant only approximately 1 h after the start of the test for most of the samples. The flow rate was determined by measuring the rate of water flowing from the upper surface using a graduated measuring cylinder.

Initial surface absorption test

Initial surface absorption test (ISAT) measurements were carried out using the method defined in BS1881[11] on 100 mm cubes which had been dried for 3 days at 105°C. A cap of known area (6360 mm^2) was clamped to the test surface. Two pipes led from the cap. One acted as a reservoir that can be isolated by a tap. The

other was connected to a calibrated capillary tube to measure the rate of absorption of water into the surface of specimen under the cap on closure of the tap. The flow was recorded at intervals up to 2 h.

Water absorption test

Some 100 mm cubes from mix C were oven dried at 105°C and then immersed in water to a depth of 20 mm and the mass gain was recorded at intervals up to 2 h.

Test programme

The programme of testing is shown in Table 2.

Methods of analysis of results

Transport processes

To analyse the results it was necessary to determine which processes were transporting the water during the tests. The main process that was considered in this paper was pressure driven flow measured by permeability.[1] The permeability may be defined in terms of a head of water Δh in the following manner:

$$V = \frac{k\Delta h}{x} \tag{1}$$

where V is the Darcy velocity for the fluid (m/s); k is the coefficient of permeability, also known as the hydraulic conductivity (m/s) and x is the distance over which the change in head occurs (m).

For the investigations performed in the present study this measure was not ideal because it only applied to liquid water and for the drying test vapour is considered and this has a substantially different viscosity. The intrinsic permeability K (m^2) is defined by

$$V = \frac{K}{e}\frac{\Delta P}{x} \tag{2}$$

where ΔP is the pressure drop and e is the viscosity in Pa s.

By including viscosity in the equation the coefficient should theoretically be the same for all fluids. This definition was used in the analysis in the present study.

It should also be noted that equations (1) and (2) can only be applied to a steady state in which the pressure is constant. For all of the work in the present study equation (2) was integrated with respect to time and the

Table 2. Testing programme

Test	Mixes tested	Number of replicates	Properties measured
Drying	A, B, C, D	3 (6 of mix C)	Dry depth, mass loss and pressure
High-pressure permeability	A, B, C, D	2	Flow rates in steady state
ISAT	C	2	Flow rates at 10, 20, 30, 60 and 120 min
Absorption	C	2	Mass at 0, 5, 15, 30, 60 and 120 min

condition of constant pressure through the time step was therefore met as the time step approaches zero.

Other potential transport processes include concentration-driven flow measured by the diffusion coefficient, electromigration driven by an electric field and thermal migration driven by a temperature gradient.[1] The flux from these processes may be increased by capillary suction or osmosis and they may be inhibited by absorption.

The drying test

The rate of loss of moisture from the specimens is governed by the movement of vapour from the drying front to the surface. There are two possible transport processes to consider for this test: pressure-driven flow and moisture diffusion. The differences between the processes is discussed by Neville.[11]

The pressure at any point in the system will be made up of contributions from several different gases and vapour. The effect of them will be additive and any pressure measurement will record the total. A change in one partial pressure will not affect the others. At the drying front the pressure of water vapour will depend on the equilibrium with the adjacent liquid and will be determined by the temperature and surface tension. The pressure outside the sample will be determined by vacuum pumping and will be substantially lower. Thus there is a pressure drop and the flow caused by it will be controlled by the permeability.

Diffusion is driven by a chemical concentration gradient and would typically be relevant to a liquid with a higher concentration of salt at one position than another. It is also used to measure the movement of one gas through another and could be the main mechanism to transport vapour through air from a drying surface. In the present experiment, however, there was virtually no air present with the pressure in the desiccator reduced to $0 \cdot 1$ kPa ($0 \cdot 001$ atmospheres) and the diffusion coefficient for vapour through air could not therefore be relevant. The transport was therefore controlled by permeability and described by the Darcy equation

$$V = \frac{K_V}{e} \frac{P}{x} \qquad (3)$$

where V is the Darcy velocity for the water vapour (m/s); K_v is the intrinsic permeability for the vapour (m^2); P is the vapour pressure difference (Pa); and x is the distance from the drying front to the surface of the sample (m).

The Darcy velocity may be related to the movement of the drying front by equating the water volumes as follows:

$$V = \alpha \frac{dx}{dt} W_F \qquad (4)$$

where α is the porosity; t is time (s); and W_F is the ratio of the water vapour volume to the volume of the same mass of water as a liquid.

Combining equations (3) and (4) and integrating gives

$$K_V = \frac{e W_F \alpha x^2}{2Pt} \qquad (5)$$

The drying depth is related to the mass loss by the relationship

$$x = \frac{M}{\alpha \Theta \rho} \qquad (6)$$

where M is the cumulative mass loss (kg); ρ is the density of liquid water (kg/m^3); and Θ is the cross-sectional area through which the transport is taking place (m^2).

The partial pressure of water vapour above a liquid surface at 20°C is 2 kPa. This pressure is correct for pure water but will have been affected by the presence of the dissolved ions in the water. This would be expected to lower the vapour pressure and lead to a slight reduction in the flow.

The measured pressure in the desiccator was initially $0 \cdot 6$ kPa but fell to $0 \cdot 1$ kPa during the test. Of the pressure in the desiccator the partial pressure due to vapour was initially approximately 1% of the total (which would be the case in a room at a humidity of 50%). Thus the vapour pressure in the desiccator was below 1% of $0 \cdot 6$ kPa and the drop in pressure from the drying front to the concrete surface was close to 2 kPa.

Not all of the pores will dry at exactly the same pressure. It has been shown[12] by measuring gas permeabilities at different humidities that the Kelvin equation gives a good indication of the pore sizes in concrete that will sustain a meniscus. This shows, however, that the smallest capillary pores ($0 \cdot 01$ μm) will not sustain a meniscus below 90% relative humidity. Thus all the pores will empty of liquid within 10% of the distance over which the pressure drops.

Using standard gas constants for a molecular mass of 18 the constant W_F was calculated to be $1 \cdot 25 \times 10^3$ by assuming the vapour was an ideal gas.

The viscosity of water vapour $e = 2 \times 10^{-5}$ Pa s.

The porosity was calculated from the weight loss on drying using the 'volume products of hydration' method[12] in which the cement and water were assumed to combine in fixed proportions and fixed values were assumed for the specific gravity of unhydrated and hydrated cement.

Equation (5) may be seen to have a similar form to the equation presented by Vuorinen[13] and Valenta (cited by Neville[11]) for water intruding into concrete under pressure:

$$k = \frac{\alpha x^2}{2ht} \qquad (7)$$

where k is, in this case, the coefficient of permeability or hydraulic conductivity (m/s) and h is the head of water. The analysis above extends Vuorinen and Valenta's work by applying it to water vapour.

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The absorption and ISAT

For the ISAT the transport process will be pressure-driven flow but in this case the pressure driving it will arise from the capillary suction at the wetting front. The analysis has been given by the present author[6] and the following relationship was derived

$$F = \left(\frac{Ks\alpha\Theta^2}{re} \right)^{\frac{1}{2}} t^{-\frac{1}{2}} \tag{8}$$

where: F is the flux (m^3/s); K and e are the liquid water permeability (m^2) and viscosity (Pa s); s is the surface tension of water (N/m); and r is the typical pore radius (m).

The results from this equation have been shown to agree with other measurements of permeability.[6] The terms for pore size and surface tension arise from the inclusion of capillary suction in the analysis.

For the analysis of the ISAT results it is only necessary to equate the Darcy velocity to the flux divided by the surface area and equation (8) may be used directly. The experiment gives a flow rate of water in a capillary tube and if this is reduced in proportion to the ratio of the area of the capillary to the wetted concrete surface the Darcy velocity is obtained.

For the analysis of the absorption results the flux is related to mass gain:

$$F = \frac{1}{\rho} \frac{dM}{dt} \tag{9}$$

and equations (8) and (9) are integrated to give

$$K = \frac{reM^2}{4s\alpha t\Theta^2 \rho^2} \tag{10}$$

where M is the cumulative mass gain.

The high-pressure test

The high-pressure (HP) test clearly measures the permeability and this was calculated by direct application of Darcy's law.

Results and discussion

When considering results for permeability testing Neville[11] states 'reporting the order of magnitude ... is adequate. Smaller differences in the value of the coefficient of permeability are not significant and can be misleading'. Oven drying of the samples causes microcracking and will also have contributed to the spread of data. The results are shown in Figs 3–8 and the spread of data predicted by Neville may be seen in addition to the trends from which the conclusions are drawn.

Figure 3 shows the average values for water permeability. It may be seen that the methods of analysis give consistent results from the different experiments
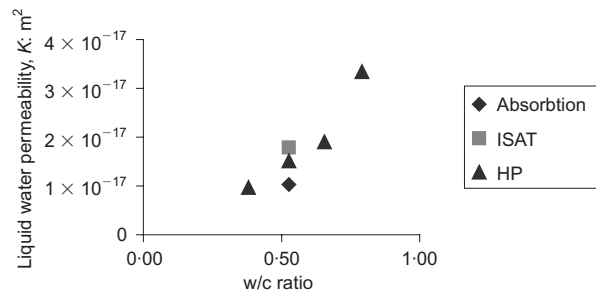

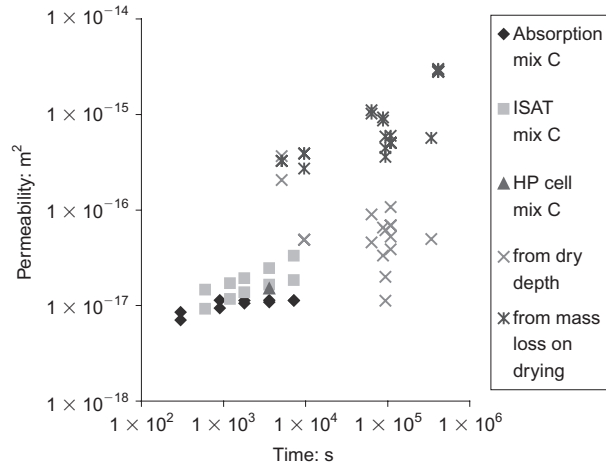Fig. 3. Results for liquid water permeability

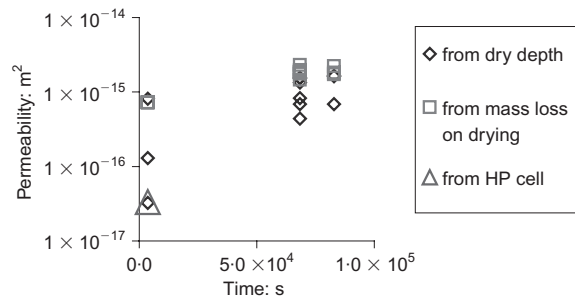Fig. 4 All permeability results plotted against time for mix C

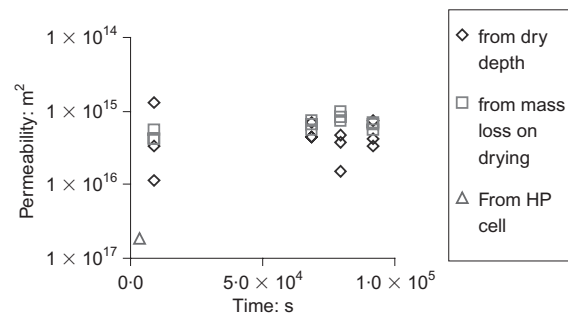Fig. 5. Permeability plotted against time for mix A

Fig. 6. Permeability plotted against time for mix B

and also the expected increase of permeability with water/cement (w/c) ratio.

Figure 4 shows all of the permeability results for mix C. The first three series (absorption, ISAT and high pressure) were very close. The vapour permeability
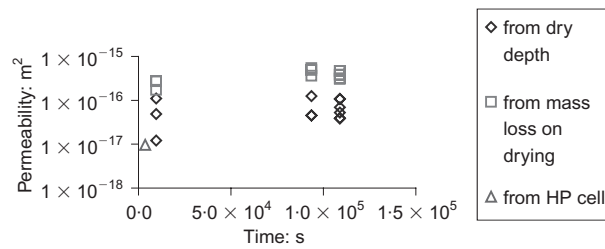
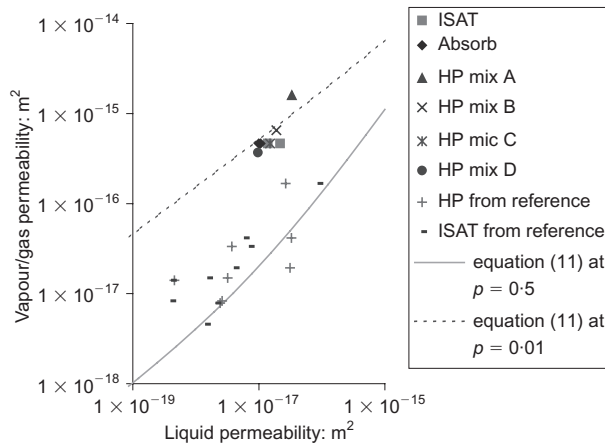*Fig. 7. Permeability plotted against time for mix D*

Fig. 8 Vapour permeability plotted against liquid permeability for all results

would be expected to be substantially higher but the results from the measured drying depths may be seen to fall over a very wide range. The results from mass loss were, however, grouped in an expected range and were therefore indicated to be far more reliable than the results from drying depth. The results from the drying depth measurements were generally lower than those for mass loss, indicating that significant drying may have occurred from regions which still appeared to be wet when the samples were inspected. Since the larger pores would dry first this implies that the visible moisture was in the smaller ones (possibly below 0·1 μm).

The reduction in permeability which is normally observed during testing with water may be caused by sedimentation causing blocking of pores and would thus not be expected to occur during vapour transport.

Figures 5–7 show the permeability plotted against time for mixes A, B and D for the high-pressure cell and drying experiment data. These support the observation that the mass loss data was far more consistent than the observations of drying depth. They all also show the drying depth data giving lower results. The drying front was readily visible on the tested samples but a further disadvantage of this method would be that on some samples (e.g. white cement) it could be very difficult to see. The mass loss was observed to be the more reliable test in these laboratory trials but this

conclusion might not be valid in other circumstances such as site testing.

The high-pressure cell data, which was for liquid rather than vapour was consistently lowest and the reasons for this are discussed below.

The new European standard test for permeability[4] relies on a visual observation of a wetting front but this is during wetting, rather than drying as in the experiments reported here. Thus the observed poorer quality of data recorded in this way would not be relevant to the EN test.

Relationship between liquid and vapour permeabilities

Klinkenberg derived an equation relating water and gas permeability, to the mean pressure as follows[13]

$$K_l = \frac{K_g}{\left(1 + \dfrac{b}{P_m}\right)} \qquad (11)$$

where K_l is the water intrinsic permeability of concrete (m^2); K_g is the gas intrinsic permeability of concrete (m^2); and P_m is the mean pressure at which gas is flowing (atmospheres).

The physical reason for the change of permeability with pressure in a gas is the 'slippage' effect which is caused by the finite velocity of molecules close to a wall and occurs when the mean free path of the molecules becomes comparable with the size of the pores through which it is flowing. Values of b were calculated by Bamforth[14] for concrete from the average values of water and gas permeability as follows

$$b = \beta_1 K_l^{\beta_2} \qquad (12)$$

in which β_1 and β_2 are constants and Bamforth proposed the values: $\beta_1 = 1·635 \times 10^{-8}$ and $\beta_2 = -0·5227$.

Substituting the value of b in equation (11), a relationship between water permeability and gas permeability is derived. Bamforth reported that the gas permeability values may be one or two orders of magnitude higher and the largest difference would occur when testing using a partial vacuum. It is therefore important to consider the effect of slippage when interpreting results obtained from gas measurement as a means of assessing concrete quality. The present author reported results[8] for gas and water permeability and found that Bamforth's constants gave a lower bound for the water permeability. The results from this work are shown in Fig. 8 and the line through the data was derived from equations (9) and (10) and using $\beta_1 = 3·5 \times 10^{-9}$ and $\beta_2 = -0·48$, which may be seen to fit the data. For the work from Claisse *et al.*[8] the average gas pressure was 0·5 atmospheres.

Figure 8 also shows a second line from the same equations using the same constants but with the average pressure at 0·01 atmospheres (1 kPa), which is applicable to the work reported here. The graph shows the results for the different tests on liquid water plotted

against the vapour permeabilities derived from mass loss and these may be seen to lie close to the line.

The investigation reported in this paper was carried out on mortar samples but the analysis would be equally applicable to concrete samples so there is no indication that the results could not be applied to other types of cementitious mixtures.

Conclusions

(a) A simple laboratory procedure in which mass loss is measured may be used to yield results for the permeability of concrete to water vapour which are consistent with the results from other tests.

(b) This work indicates that when measurements are made on samples dried under vacuum the depth of drying observed by breaking the samples open does not give the best results. The mass loss is far more reliable.

(c) Gas slippage theory must be used when calculating the intrinsic permeability from the transport of water vapour in concrete.

(d) These results therefore indicate that this method of analysis gives agreement between the permeability values calculated from observations derived from the different experiments and that they may be used to confirm the results from individual tests and identify testing methods that give inconsistent results.

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