Determination of the moisture storage characteristics of porous capillary active materials

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Abstract

With new improved calculation models a good agreement between computed and measured results of moisture transfer is obtainable on condition that correct material properties are used. Especially when capillary active materials are in hygric connection their moisture storage characteristics have a predominant influence on the moisture behaviour of the building component. In this contribution a pressure plate method to determine the moisture storage characteristics in the capillary moisture region is presented, which uses water as measuring medium and thus assures a more realistic description. Furthermore it is shown how to combine these results with the sorption isotherm, which describes the storage function in the hygroscopic moisture region, to get one storage function continuous over the whole interesting moisture region, from dry material up to capillary saturation.

Résumée

Des models récent pour le calcul de transfère d'humidité donnent des résultats qui correspondent bien avec l'expérience, si les propriétés de matériaux sont choisis correctement. Particulièrement les caractéristiques de la capacité de fixation d'eau des matériaux joue un grand rôle pour le comportement hydrique quand le flux liquide passe d'un matériau à l'autre. Dans ce travail une méthode pour déterminer les capacités de fixation d'eau pour les teneur en eau élevés est présentée. Cette méthode basée sur l'équilibre entre la pression capillaire de l'eau et la pression appliquée au matériau de l'extérieur utilise des plaques de succion. Pour obtenir la fonction de la capacité de fixation d'eau dans toute la domaine d'un matériau les résultats de cette méthode sont combiné avec des isotherme de sorption classiques.

1. Introduction

The assessment of the moisture behaviour of building materials is nowadays of increasing importance. Problems, like preventing the deterioration of historical buildings, the improvement of heating insulation as well as the retrofitting of the outer parts of existing buildings are strongly linked to questions concerning moisture behaviour. The demand of computation tools has increased in recent years due to the fact, that exprimental investigations in the moisture behaviour are expensive and lengthy. Furthermore with new improved calculation models a good agreement between computed and measured results is obtainable [16]. The application of these models requires correct material properties. With building components composed of different capillary active materials - aside from the transport coefficients for liquid transport and vapour diffusion - the moisture storage characteristics are of essential importance for the calculation of their moisture behaviour. Therefore investigations on this subject were performed at the Fraunhofer-Institut für Bauphysik, directed by Prof. Dr.-Ing. K.Gertis.

2. Fundamentals of moisture storage

Three regions may be distinguished in porous mineral building materials: the region of sorption moisture, the capillary water region and the region of supersaturation. Following is a more detailed discussion of these three regions.

The sorption moisture region

The sorption moisture region is characterized by the accumulation of water referred to as adsorption from the surrounding moist air until a state of equilibrium is reached. Under isothermal conditions, the relationship between the volume of the accumulated water and relative humidity is characterized by the sorption isotherm (see Fig. 1). For hygroscopic porous building materials sorption isotherms have a typical S-shaped profile ([5] [8] [10]) and often show a hysteresis effect between moisture absorption and release (absorption and desorption). This hysteresis effect is widely viewed as the result of differing wetting characteristics for adsorption and desorption or other phenomena created by pore space geometry [18]. However, measurements by Künzel [15] show that for most building materials the hysteresis effect is so slight that the absorption isotherm is adequate to characterize the sorption moisture region. In the case of somewhat more pronounced hysteresis, investigations by Rode [19], who compared results computed with and without taking the hysteresis into account, showed that a sufficiently accurate calculation of the moisture behaviour of such materials is possible by choosing a mean curve between absorption and desorption isotherms. The influence of temperature is indicated schematically in Fig. 1, bottom. The typical characteristics of a sorption isotherm for hygroscopic materials are shown in Fig. 1 (top). The lower area up to about 15% relative humidity is marked by a monomolecular layer on the solid surface, followed by transition into an area of multimolecular layer which rises in linear fashion, ending at about 50 % relative humidity. The subsequent progressively rising water content is attributed to capillary condensation. This involves the

Fig. 1 Schematic representation of a typical sorption isotherm for hygroscopic porous building materials [9].

Above: Areas of moisture absorption with indication of approximate pore radii (using the cylindrical pore model).
Below: Temperature influence and hysteresis effect between moisture adsorption and desorption.

occurance of condensation phenomena in the micropore area with radii between roughly 2 x 10^{-9} and 10^{-7} m. According to Kelvin's law these are responsable for a lowering of saturation vapour pressure over concave menisci ([5] [10]). The assumption of a spherical menisci in a cylindrical capillary yields, as derived in [8], the often-used Kelvin equation. It shows the relationship between relative humidity and that capillary radius up to which the cylinder pores are filled by capillary condensation:

$$\varphi = \exp\left[-\frac{2\sigma\cos\theta}{r\rho_w R_D T}\right] \tag{1}$$

φ [-]	relative humidity
σ [N/m]	surface tension of water
θ [dgr]	wetting angle
r [m]	capillary radius
R_D [J/kgK]	gas constant for water vapour
T [K]	absolute temperature
ρ_w [kg/m^3]	density of water

Capillary moisture region

At relative humidities above 95% the sorption isotherm rises very sharply. However it is for this very region that there is no known method of measurement to determine relative humidity with high a accuracy. It is therefore not possible to definitively apportion moisture content to relative humidity. It is here that the capillary moisture region, frequently also called the superhygroscopic region, begins. This region is marked by the ability of capillary-porous hygroscopic materials to take up water until capillary saturation is reached. Capillary saturation is defined as the material moisture content which can be attained through natural water absorption under normal pressure without the influence of exterior forces. For capillary-porous building materials, capillary saturation is always below the water content possible in the open pore space and is an important coefficient of the material. The reason that not all pore space is filled is the presence of entrapped air. The water absorption results from the surface tension of liquids and the wettability of solid bodies in contact with the liquid. In [5] [11] [20] there is a full description of the capillary phenomena which can most clearly be depicted by the simple model of a cylindrical capillary (Fig. 2).

If partially filled capillaries of differing radii are connected with one another, the smaller ones with higher suction pressure will draw moisture out of the larger capillaries until they themselves are filled or until the larger capillaries are emptied (see Fig. 2, middle, above). In a capillary-porous body having continous pore-size distribution, all of the smaller pores will therefore be filled with water up to a certain pore size as a function of the moisture content. Thus there exists a correlation between the moisture content of a capillary-porous material and its capillary pressure defined by the largest pores still filled. If a moist capillary-porous building material is placed in capillary contact with another dry building material, the latter will continue to extract water until a state of equilibrium exists. This equilibrium is marked by the existence of equal capillary pressures in the two materials. When the two materials do not have the same pore size distribution, different moisture contents will be established. The capillary pressure curve (capillary pressure

Fig. 2 Compilation of the principal capillary phenomena for simple cylindrical capillaries [8].

as a function of moisture content) hence yields the storage function for the capillary moisture region. Molenda, Crausse and Lemarchand [17] show via a calculation of the moisture balance that the measured hysteresis of the capillary pressure curve of a sand fill has a noticeable effect on its moisture balance. In contrast hysteresis has only a slight effect on aerated concrete. The reason for this lies in the fact that in aerated concrete, as in most mineral building materials, there is a significantly broader pore size distribution than in sand fill and consequently a much flatter shape of the suction pressure curve. Investigations by this author on specimens of natural sandstone show that roughly the same superhygroscopic equilibrium moisture contents are established regardless of whether they were reached by humidification or dehumidification of the reference specimens [16]. The effect of temperature on the suction pressure curve follows, as tests by Crausse [2] and Vetterlein [21] show, from the slight influence of temperature on surface tension.

Supersaturated region

Capillary saturation can only be exceeded by application of external pressure, application of a vacuum to remove attrapped air or forced condensation by pushing below the condensation point. Another possibility lies in delayed soaking in water since trapped air dissolves in the water with time. There is no unique relation between capillary pressure and water content in the region of supersaturation since replacing the air bubbles in a specimen does alter its water content but not its suction pressure. Consequently, the suction pressure curve of a vacuum-saturated specimen will even in the capillary water region differ from that of the suction pressure curve for a capillary saturated specimen. As tests have shown [13], no moisture equilibrium is reached through capillary transport between supersaturated and capillary-saturated regions even within 40 days. Since there is no clear connection between capillary pressure and moisture content the region of supersaturation is ignored.

3. Determination of storage characteristics

In determining storage characteristics it is necessary to distinguish between the sorption moisture region and the capillary water region. Different measuring methods have to be employed for the two regions. Determination of the sorption isotherm is quite well known and the measuring method standardized in DIN 52 620 [3] and as a result no further description of the method is given here. A relatively comprehensive catalogue of sorptionisotherms may be found in [6]. For some materials albeit the variation is relatively large, necessitating own measurements in some instances. Table 1 contains a listing of absorption moisture content figures at various relative humidities for the building materials investigated.

Table 1 Tabulation of sorption moisture contents for the building materials investigated, determined at various relative humidities.

material	sorption moisture content in [Vol.-%] at					
	10 % RH	30% RH	50 %RH	65 % RH	80 % RH	90 % RH
Obernkirchner	0.06	0.13	-	0.26	0.34	0.43
Rüthener	0.18	0.45	-	0.8	1.24	1.69
lime silica brick	-	-	1.7	1.8	2.49	4.02
aerated concrete	-	-	0.73	1.25	1.7	3.8

The moisture storage function in the capillary moisture region is determined using a pressure-plate apparatus. Using selected examples of salt-impregnated and non salt-impregnated natural sandstones the capillary pressure determined with the pressure-plate apparatus is compared with the test data from mercury porosimetry to illustrate the benefits and drawbacks of this measuring method. From the outcome of the sorption measurements and the pressure plate measurements a moisture storage function is developed encompassing all significant moisture storage characteristics of a porous building material all the way up to capillary moisture saturation.

3.1 Pressure plate measurement

The following gives a description of the measuring principle, the equipment setup and the procedure involved in measuring capillary pressure.

3.1.1 Measuring principle

The determination of storage characteristics using the pressure plate apparatus attributable to Gardner [4] is likewise used in soil science ([7] [20]). In a pressure vessel water is forced out of water-saturated specimen by applying different levels of overpressure and the equilibrium moisture content appropriate to the pressure is determined. To calculate the relationship between moisture content and maximum pore-size still filled, the following model is used. The pore volume of the capillary-porous body consists of a bundle of unbranched capillary tubes of varying radii (cylindrical pore model). The elevation h of water in a capillary of radius r is given by:

$$h = \frac{2\sigma\cos\theta}{r\rho g} \qquad (2)$$

σ [N/m]	surface tension (for water: 72.75 · 10^{-3} N/m at 20 °C)
ρ [kg/m^3]	density (for water: 1000 kg/m^3)
g [m/s^2]	acceleration due to gravity
θ [°]	wetting angle (for fully wetting liquids: 0°)

Because the hydrostatic pressure at maximum attainable elevation must be equal to the pressure needed to empty this capillary, the pressure can be correlated with a radius up to which all of the larger pores are emptied. For water, this yields the following approximation equation:

$$r = 15 \cdot 10^{-6} / P_o \qquad (3)$$

r [m]	pore radius
P_o [bar]	applied overpressure

Since normally there are no cylindrical unbranched capillaries in a capillary-porous body, the cylindrical pore model inherently has certain problems. Sometimes there can be considerable variation in diameter along the length of the capillaries. The pressure which has to be applied to empty the volume behind a constriction (the neck of the pore) is determined by the cross-sectional dimensions of this constriction in the pore. Thus equation (3) gives a correlation between the volume of water forced out and the radius of the pore-neck through which it was emptied [20]. However, since the same model shows the same systematic error when used in mercury porosimetry, we have the required conditions for a comparison between measurement of suction pressure and mercury porosimetry. The conversion of capillary pressure to a pore radius only serves to illustrate the experimental results; the choice of pore model has no effect on the applicability of the suction pressure curve.

3.1.2 Equipment set-up

The experimental apparatus for measuring the suction pressure consists of three pressure vessels, diameter about 25 cm, for pressures up to 5 bar, 15 bar and 100 bar. A pressure is generated up to 15 bars by a compressor and with a gas bottle above this level. Pressure reducers are used to maintain constant pressure. The schematic layout of a pressure vessel and a photographic view are shown in Fig. 3.

Depending on its size and the number of specimens involved, each pressure vessel contains one to three ceramic plates. They are selected as to porosity so that after saturation their water permeability is as high as possible but not permitting the passage of air up to the maximum applied pressure. The plates are sealed with a rubber membrane on one side and connected to the outside by a hose. To achieve a good capillary contact between the bottom of the specimen and the plate, the bottom of the specimen is normally coated with porcelain clay (caolin) and the specimen pressed against the plate. However because this can spoil the specimen and cause errors in weighing, after

Fig. 3 Schematic layout of a pressure vessel in the suction pressure test unit (left) and a photographic view of the test equipment (right).

thick layer of porcellain clay has been applied, the plate is covered with a very fine cloth through which very little of the clay can pass. Once the plate is moistened this produces a soft bed into which the specimens can be pressed. This likewise assures good hygric contact while significantly reducing the clay adhering to the specimen.

3.1.3 Procedure

The specimens to be investigated are cut into pieces about 4 x 4 x 1 cm^3 in size. Greater thicknesses than 1 cm are to be avoided since this can significantly increase measuring time. Ten specimens of each type are used so that averaging will even out discrepancies in measurement. The dry weight of the specimen tablets is determined, subsequently they are saturated with water under standard pressure, weighed and pressed onto the cloth. The plate is installed in the pressure vessel and the first pressure level is applied. The water is now forced out of the specimens, through the plate and out through the hose. In two to four days a state of equilibrium is reached depending on the dimensions of the specimens and their porosity characteristics, marked by cessation of water flow out of the vessel. The specimens are then removed from the plate, cleaned of any clay sticking to them and weighed, yielding the water loss for the particular pressure level. The specimens are then returned to the vessel to set the next pressure level. This procedure is repeated from the lowest pressure level to the highest. Overall measurements are taken at up to 10 pressure levels between 0.015 bar and 100 bar, covering a pore-radius range between 10^{-4} m and 1.5 x 10^{-8} m. From the readings for weight loss, pore volume or - expressed in terms of overall porosity - the relative pore volume can be determined for each pressure stage and hence from equation (3) for the corresponding capillary radius range.

3.2 Comparison with mercury intrusion porosimetry

To illustrate the advantages of pressure plate measurement vis-a-vis Hg porosimetry, measurements are carried out on salt-impregnated and non salt-impregnated sandstone materials. It is anticipated that the salt content exerts a significant influence on the water storage behaviour. The test results from the two methods are compared and the method-related differences shown while taking into account the effects of the type of stone and the influences of salt in the pore spaces of the various stones. A full description of these investigations may be found in [12].

3.2.1 Description of mercury intrusion porosimetry

Mercury intrusion porosimetry is well-known, used frequently and operates on the principle that mercury intrusion depends on the pressure. The operative principle is also based on the capillary law; the cylindrical pore model is usually applied here, too. Mercury, being a non-wetting liquid, is forced into the porous specimen material and the volume taken up by the specimen registered for each pressure level. A mercury porosimeter operates over a range from 0.15 to 2000 bar, corresponding to a radius range of 5×10^{-5} m to 3.7×10^{-9} m. It takes from 2 to 4 hours to complete measurement of a specimen to acquire up to 630 test points over the full pressure range.

3.2.2 Specimen preparation and experimental procedure

The investigations were carried out on two different types of sandstone - the Obernkirchner and the Rüthener sandstone - markedly different in their porosity characteristics, each involving unimpregnated specimens as well as others saturated with Epsomite ($MgSO_4 \cdot 7H_2O$) or with Nitrokalite (KNO_3). Salt-impregnation of the Rüthener sandstone is done in a 3% Epsomite solution and a 3% Nitrokalite solution; that of the Obernkirchner sandstone in a 3% solution of Epsomite and a 25% solution of Nitrokalite. Using these concentrations it is possible to achieve roughly the same quantity of absorbed salt in each of the specimens. The specimens, initially rod-shaped and measuring 4 x 4 x 20 cm^3, are saturated with the respective salt solution, then dried in dry air at 40 °C to constant mass, weighed and then returned to the salt solution. This process is repeated until there is no longer any appreciable increase in mass. To measure capillary pressure the specimens are cut to size at about 4 x 4 x 1 cm^3. For mercury pressure porosimetry, 5 to 10 g of specimen material is sufficient each time.Table 2 shows the porosity

Table 2 Tabulation of the porosity coefficients and the amount of absorbed salt of the varieties of stone investigated for comparison of pressure plate measurement and mercury intrusion porosimetry.

stone variety	bulk density [kg/m³]	porosity [vol.-%]	capillary saturation [Vol.-%]	absorbed amount of salt [mass-%]	
				$MgSO_4 \cdot 7H_2O$	KNO_3
Obernkirchner sandstone	2150	14	11	0.16	0.39
Rüthener sandstone	1950	24	21	0.19	0.41

characteristics of the unimpregnated, recently quarried specimens of rock measured using a helium pycnometer as well as data on the average amounts of salt absorbed by the specimens under the preparation described.

3.2.3 Test results and evaluation

Fig. 4 shows the results of these investigations for the two test methods and types of rock in the form of a cumulative pore-size distribution.The results obtained from the freshly quarried stones using the two test methods are shown as solid lines. The distributions found using the suction pressure apparatus are somewhat more angular due to the small number of pressure

Fig. 4 Cumulative pore radius distribution of freshly quarried and salt impregnated Obernkirchner and Rüthener sandstone measured using mercury porosimetry and the suction pressure plate equipment.

levels involved but show the same characteristics as those from Hg porosimetry. In the Obernkirchner sandstone, both methods show a sharp drop in the pore-radius range between 3 x 10^{-6} m and 1.5 x 10^{-6} m; the drop lies between 3 x 10^{-7} m and 3 x 10^{-6} m for the Rüthener sandstone.

In addition to the pore size distributions of the unimpregnated specimens, Fig. 4 also shows those for the salt-impregnated specimens. The curves found for the Obernkirchner sandstone (Fig. 4, top left) using mercury-intrusion porosimetry are almost identical up to a filled pore radius of about 3 x 10^{-6} m and there are practically no noticeable salt effects. Beyond this point, the curves diverge only slightly toward a differing overall porosity. Here the curves for the salt-impregnated specimens are also close together, hence showing scarcely any differences according to type of salt. In the pressure plate method there is likewise scarcely any difference among the curves for the salt-impregnated specimens, though they do differ from that of the unimpregnated specimen, especially in the lower pore-radius range < 10^{-5} m (Fig. 4, top right).

The differing results from the two methods of measurement are especially evident in the Rüthener sandstone (Fig. 4, bottom). Using Hg-pressure porosimetry all curves for salt-impregnated and unimpregnated rock are close together. The shapes of the curves differ markedly, however, when measured using pressure plate apparatus. The salt-impregnated specimens are scarcely differing in the upper pore-radius range though differing distinctly from the unimpregnated specimen. In the lower radius range the specimen impregnated with KNO_3, in contrast to the one impregnated with $MgSO_4$, have capillary storage characteristics barely differing from those of the unimpregnated specimen.

For the interpretation of these results the reader is directed to the appropriate remarks on differential pore-radius distributions contained in [12]. A comparison of the test results from the two methods shows that despite its more basic manual measuring procedure and the consequent lower number of test points, the pressure plate method yields results which are certainly comparable to those of mercury-pressure porosimetry. The use of water as the operating medium assures a more realistic result since, as under natural conditions, it covers the true interaction between water and pore wall including substances possibly present in the pores, especially hydrophilic or hydrophobic ones. This is particularly evident with salt-impregnated specimens as the test results described indicate. Moreover the use of capillary saturated specimen material also only includes in the analysis the pore-space available to capillary transport. In Table 3 the main points of the two analytical methods are again compared in terms of their characteristic features.

Table 3 Comparison of parameters for pressure plate measurement and mercury intrusion porosimetry.

parameter	pressure plate measurement	mercury intrusion porosimetry
analytical medium	water	mercury
pore space encompassed	only water-filled pores	all pores
influence of salt	measurable	no measurable response
hydrophobic effects	measurable	no measurable response
moisture condition	wet	dry
rate of measurement	low (ca. 4 weeks)	high (ca. 3 hrs)
accuracy	medium	high
gradation	coarse	fine
pore radius range [µm]	0.015 to 30	0.004 to 50

3.3 Determination of the storage function from pressure plate measurement and sorption measurement

The sorption isotherm and the suction pressure curve both give the moisture content as a function of true driving potentials, relative humidity φ as the ratio of vapour pressure to saturation vapour pressure and the capillary radius r as a quantity characterizing the capillary pressure. The two parameters are connected to one another through Kelvin’s thermodynamic equilibrium condition mentioned previously (see equation (1)). This means that moisture content may be expressed in a standardized fashion in both the hygroscopic and the superhygroscopic moisture regions as a function of either φ or r.

Universally applicable and better amenable to an intuitive grasp in practical work is the choice of relative humidity as general moisture storage potential. In non capillary-active building materials, as in most insulating materials for example, vapour pressure or relative humidity, but not capillary pressure, is defined. Using the example of lime silica brick, Fig. 5 shows how the moisture storage function is composed of:

- the sorption isotherm (top left, indicating the value for capillary saturation)

and

- the suction pressure curve (top right, indicating relative humidity for the respective pore radii according the Kelvin equation).

Fig. 5 Development of a storage function for the hygroscopic and super-hygroscopic region from the sorption isotherm (top left) and the recorded suction pressure curve (top right) using lime silica brick as an example.

Because the suction pressure measurement deals only with a relative humidity range between 93 % and 100 % as a result of the non-linear relationship between the pore-size and the relative humidity, this means that this way of depiction results in a steep curve for the capillary moisture range. To better compare the moisture storage functions of various building materials, capillary radius is therefore chosen instead as moisture storage potential for Fig. 6 and the moisture storage function is developed similarly.

The moisture storage functions shown for aerated concrete, lime silica brick and two sorts of natural sandstone all show a continuous transition (without steps or breaks) from the hygroscopic moisture region, in which the storage function is determined through adsorption measurements, to the super-hygroscopic region with pressure plate measurement as the analytical method. Despite the use of two different analytical methods, this allows the determination of a uniform moisture storage function which contains all of the practically significant moisture storage characteristics for a porous building material up to capillary saturation and which can be expressed as a function of a genuine potential variable (either relative humidity or pore radius). In [14] and [16] some more examples of moisture storage functions are shown and their correctness is evaluated by calculations and comparison with measurements.

Fig. 6 Moisture storage function of aerated concrete (top left), lime silica brick (bottom left), Rüthener sandstone (top right) and Obernkirchner sandstone (bottom right) developed from the sorption isotherm and the recorded suction pressure curve.

4. Summary

The storage of moisture in building materials is characterized by three regions. The first region marks the so-called hygroscopic equilibrium moisture contents. Next to this for capillary-active materials comes the capillary water region with water contents up to capillary saturation. The storage function for the hygroscopic moisture region is characterized by the sorption isotherm. Gravimetric determination of the sorption isotherm, which is relatively simple but mostly time-consuming, is feasible to an upper limit of 95 % RH. Because the sorption isotherms of most building materials are extremely steep at higher relative humidities, clearly defined sorption measurements and the correlation of water content with relative humidity are no longer possible above this value. For the determination of the storage function for the capillary moisture region a unit for measuring suction pressure familiar in the field of soil science and modified for this purpose is used for the first time in building physics. In contrast to mercury pressure porosimetry, this method, which uses water as the measuring medium, assures a more realistic description.It allows for the interactions between water and pore walls in a way which is identical with natural conditions. It also makes it possible to document not total pore space but the pore space accessible to moisture transport by basing the analysis on capillary saturated specimens.

The sorption isotherm and the suction pressure curve express water content as a function of true potential parameters not dependent on the material: relative humidity and capillary pressure respectively. The two parameters are linked by Kelvin’s thermodynamic equilibrium condition. This makes it possible to combine the sorption isotherm and the suction pressure curve into a storage function continuous over the whole region and including all the practically significant moisture storage characteristics of a porous building material up to capillary water saturation.

5. References

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