TECHNIQUE FOR MEASURING MOISTURE STORAGE CAPACITY AT HIGH MOISTURE LEVELS

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ABSTRACT: Knowledge of moisture storage capacity above the hygroscopic range is essential when calculating moisture, especially when different materials are to be connected. Generally, the storage capacity is represented by water retention curves, in which suction is plotted against the moisture content. In a number of laboratories in Europe and North America, the pressure plate and pressure membrane techniques are used to measure water retention curves. There are, however, various ways of carrying out such measurements. This paper compares two different methods of presaturating the specimens before testing, namely capillary and vacuum saturation. It also examines the effect of different beddings between the specimen and the ceramic plate. It is shown, as expected, that capillary and vacuum saturated specimens give differential results. Absorption tests clearly show that, for some materials, capillary saturation is a vague concept. For this reason, vacuum saturation is generally to be preferred. In the test with different beddings, no significant differences were found.

INTRODUCTION

The presence of free water or very high relative humidity inside the material is strongly associated with several important durability problems, including frost damage in materials; flaking of grout and paint caused by frost, mold, and rot on organic materials; and corrosion of steel reinforcement. Consequently, accurate prediction of the service life of structures requires good models of moisture and liquid (capillary) transport at high moisture levels, including knowledge of related properties such as moisture diffusivity and moisture storage capacity. The latter is of the utmost importance when calculating moisture transport in multilayer constructions.

Normally, moisture storage capacity is presented in sorption isotherms, with the relative humidity used as storage potential. However, at high moisture levels the relative humidity gives very low resolution, and consequently some other potential must be used to represent moisture storage capacity. In general, the alternative measure of storage capacity is a water retention curve that shows the relation between suction and water content. It is also possible to present the result as the relation between the pore radius and the water-filled porosity, i.e., a graph of the pore size distribution. Thus, this measurement can also be used to characterize the tested material.

Water retention curves are usually measured with the help of pressure vessels in which pressure plates or a pressure membrane have been mounted. In these vessels, moisture storage capacity can be measured with high resolution between 93 and 100% relative humidity. Several laboratories have carried out extensive work in this area [e.g., Brocken (1998), Hansen et al. (1999), and Krus (1999)]. However, different laboratories have used different experimental procedures, and in order to be able to compare their results or to use them in moisture calculations it is vital to know the effects of these different measurement procedures. Hence, a series of tests were conducted to investigate some of the differences.

WATER RETENTION CURVES

General

Water can be physically bound in pores through adsorption of water molecules on the surface of the pore or through surface tension effects causing capillary condensation of surrounding water vapor or capillary water uptake. Adsorption takes place at low moisture levels and capillary condensation and capillary water uptake at high moisture levels. Capillary-condensed water molecules are condensed on curved water menisci that are formed in small pores and other narrow spaces. The relationship between the relative humidity ϕ when condensation take place and the principal radii of the curvature of the meniscus in two orthogonal directions, r_1 and r_2 (m), is given by Kelvin's equation [e.g., Adamson (1990)]

$$\ln \phi = -\frac{\sigma M_w}{RT\rho_w}\left(\frac{1}{r_1} + \frac{1}{r_2}\right) \tag{1}$$

where σ = surface free energy of a surface liquid gas (J/m^2); M_w = molar weight of water (0.018 kg/mol); R = gas constant [8.314 J/(mol·K)]; T = temperature (K); and ρ_w = density of water (kg/m^3).

In the hygroscopic range (i.e., up to approximately 98% relative humidity), the relation between the moisture content of the ambient air and the moisture content of the material represents the moisture fixation. This relation is given by sorption isotherms. In the superhygroscopic range, the moisture fixation is represented by a relation between the suction s(Pa) and the moisture content of the material (suction is defined as the pressure difference between the pore water pressure and the ambient total pressure). This relation is shown in what are called water retention curves. In these curves the suction is plotted against the moisture content in the material. For many materials there is hysteresis between absorption and desorption, i.e., the relations are different, depending on whether equilibrium is reached by absorption or desorption. There is currently no full explanation for this hysteresis, but the classical ''ink-bottle'' theory is often used. This theory has, however, sometimes been questioned, especially for the superhygroscopic range [e.g., Fagerlund (1999)].

The Laplace equation gives a relationship between the suction s (Pa) and the radii of the curvature of the meniscus in two orthogonal directions, r_1 and r_2 [e.g., Adamson (1990)]

$$s = \sigma\left(\frac{1}{r_1} + \frac{1}{r_2}\right) \tag{2}$$

Combining Kelvin's equation [(1)] with the Laplace equation [(2)] gives a relationship between the relative humidity and the suction

$$\ln \phi = -\frac{sM_w}{RT\rho_w} \tag{3}$$

Thus, it is possible to represent the moisture fixation in both the hygroscopic and the superhygroscopic range with one sin-

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gle relationship with either relative humidity or suction as the moisture storage potential. Using relative humidity as the moisture storage potential will result in a very steep curve in the superhygroscopic range, i.e., the resolution in this region will be poor. At high moisture levels, it is therefore better to choose the suction as the moisture storage potential, i.e., water-retention curves.

If the pore system is modeled with a cylindrical pore model (i.e., the pore system consists of cylindrical tubes with varying radii r), it is possible to calculate the pore size distribution from the water retention curve using (2) (the curvature of the meniscus in two orthogonal directions is the same in a cylindrical pore, that is, $r_1 = r_2 = r/\cos\theta$)

$$r = \frac{2\sigma\cos\theta}{s} \tag{4}$$

where θ = contact angle; and r = pore radius corresponding to a certain relative humidity.

The contact angle is usually set to 0°. This is, however, probably not accurate. According to Letey et al. (1962), the angle is 50° between a certain type of sand and water. Nevertheless, the contact angle is probably similar for many inorganic building materials, and the error introduced when the contact angle is set to 0° is therefore about the same for these materials. Irrespective of the method used to measure the pore size distribution, the results should only be used to compare materials, and should not be regarded as true measurements. Moreover, no pore in an organic building material can be regarded as a cylinder; so that the error associated with using (4) is already rather sizable.

The pore radius is an additional way of presenting moisture storage potential. It has the same resolution and advantages as the use of suction. That is, the pore size distribution gives good resolution in the superhygroscopic range.

Methods of Measuring Water Retention Curves

There are several methods of measuring water retention curves. In this study, pressure plate extractors (PPE) and pressure membrane extractors (PME) were used. The method is described in detail below. There has been considerable study of this technique [e.g., Bomberg (1974), Krus (1995), Hansen (1997), Janz (1997), Brocken (1998), and Hansen et al. (1999)]. There is also a test standard for this technique, called the Nordtest Standard NT Build 481 (Nordtest 1997). The water retention curve is measured directly with PPE and PME since both the applied pressure (corresponding to the suction within the material) and the weight of the specimens (i.e., the water content) are measured.

It is also possible to calculate water retention curves from measured pore size distributions. To do this, it is first necessary to develop a geometrical model of the pore system. Generally, a cylindrical pore model is used, and then (4) is applied. There are several different methods of measuring pore size distributions. The most frequently used method is mercury intrusion porosimetry (MIP). The principal difference between PPE/PME and MIP is that mercury is almost nonwetting, which means that it has to be forced into the pores. With PPE/PME, water is used, which is wetting and therefore penetrates the pore system by capillary suction, and consequently must be forced out of the specimen.

In Krus and Kießel (1991, 1998), experimental results from using pressure plate and pressure membrane extractors on specimens of sandstone are compared to results using MIP. The overall porosity differs somewhat between the two methods, but the shapes of the obtained curves are very similar. The differences in porosity are not surprising given that the PPE/PME started with capillary saturated specimens, and hence did not measure the total porosity. However, their study did indicate that it is possible to predict water retention curves in sandstone reasonably well with MIP.

An advantage of using MIP is that measurements can be carried out more quickly than with PPE/PME (MIP takes hours rather than weeks). But since the pores are filled with mercury, MIP cannot measure the effect of salts in the pore system or hydrophobic effects (Krus and Kießel 1998). Moreover, in MIP great pressure is used on the mercury, which might cause fracturing of pore walls. This means that MIP might indicate a coarser pore structure than PPE/PME. Furthermore, mercury has negative environmental effects and thus its use should, if possible, be avoided.

There are also less widely used methods of measuring pore size distributions. Fagerlund (1973a) discussed a number of other possible methods, such as a centrifuge method, determination from the liquid penetration rate, and displacement methods. Fagerlund (1973b) and Sellevold and Bager (1980) also presented a method for determining pore size distribution from a calorimetric determination of freezable water.

DETERMINATION OF WATER RETENTION CURVE WITH PRESSURE PLATE AND PRESSURE MEMBRANE EXTRACTORS

The equipment and the techniques used for measuring retention curves originate from soil physics. Richards (1948) was the first to report porous plate techniques. The principle of the method is to force water out of saturated specimens using overpressure. To obtain this overpressure, three different pressure vessels mounted with pressure plates or pressure membranes are used. The pressure plates are ceramic and capable of withstanding pressures up to 15 bar. The pressure membrane is made of cellulose and is used in the range of 15−100 bar. The function of the two types of extractors is the same. The porous ceramic plate/cellulose membrane on which saturated specimens are placed is saturated with water. The saturation is obtained by letting the ceramic plate/cellulose membrane absorb water for 24 h or more. The side of the ceramic plate/cellulose membrane on which the specimens are placed is then exposed to an overpressure, while the other side is always at atmospheric pressure. This results in a pressure difference over the ceramic plate/cellulose membrane and the specimen (Fig. 1).

Capillary forces in the pore system of the ceramic plate/cellulose membrane are able to withstand the maximum-dimensioned pressure difference applied over the ceramic plate/cellulose membrane. Since the pores are saturated with water, they allow water but not air to pass through them. The maximum pressure that the ceramic plate/cellulose membrane can withstand before letting air pass through the pores is determined by the radius of its pores, r_0 (Fig. 2). In this study, three

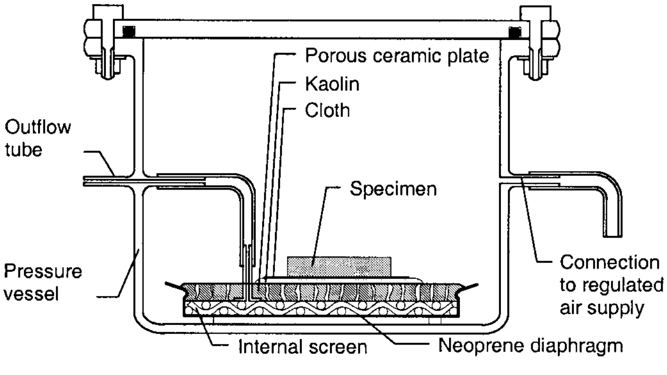

FIG. 1. Sketch of Ceramic Pressure Plate and Specimen Mounted in Pressure Vessel; Internal Screen Prevents Neoprene Diaphragm from Clogging Underside of Ceramic Plate When Pressure Is Applied

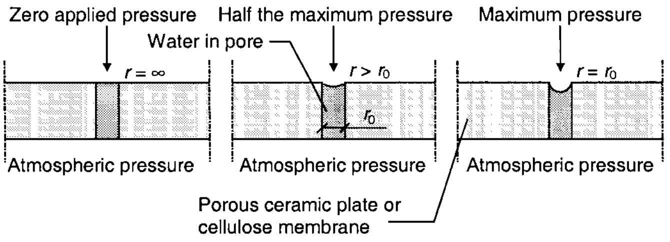

FIG. 2. Function of Ceramic Plate/Cellulose Membrane

different ceramic plates with different pore systems (pore diameters) were used. They were designed to withstand up to 1, 3, and 15 bar overpressure, respectively. The 1 bar ceramic plate had the largest pore diameter and the 15 bar ceramic plate the smallest. The advantage of using plates with the largest possible pores is that the test can be carried out faster.

Three different vessels were used to apply the overpressure to the specimens and the ceramic plate/cellulose membrane. The vessels could withstand pressures up to 5, 15, and 100 bar, respectively. Four ceramic plates could be mounted in the 5 bar vessel and three in the 15 bar vessel. In the 100 bar extractor a single layer of cellulose membrane could be mounted. The source of pressure was high-pressure nitrogen or air in tanks, and high-quality manifold pressure regulators regulated the overpressure applied. The lowest possible pressure set point with the equipment used was approximately 0.01 bar, which, according to (4) and (3), correspond to a pore radius of approximately 0.15 mm and a relative humidity of 99.9993%. The highest pressure set point (100 bar) corresponds to a pore radius of approximately 15 nm and a relative humidity of 93%.

Once the ceramic plate/cellulose membrane and the presaturated specimens had been mounted in the vessel, the first, and lowest pressure level was applied. Excess water in the specimen was forced out of the specimen by the overpressure, through the ceramic plate/cellulose membrane, and out of the vessel through the outflow tube (Fig. 1). The excess water was collected in a burette. Equilibrium is said to be achieved when the water outflow is <0.05 cm^3 in 48 h. At equilibrium, the mass of the specimens (i.e., the water content) was determined. Then, the specimens were remounted in the vessel and the next, slightly higher, pressure was applied. Since the test procedure started with saturated specimens, equilibrium was reached through desorption.

MATERIALS

The materials used in the tests were lime silica brick; autoclaved, aerated concrete; cement mortar; cement-lime mortar CL 50/50/650; and two types of bricks, brick 1 named ''Kanik Antik'' (red) and brick 2 named ''Kanik Gul'' (yellow and hard burned). All are well known and widely used building materials. Their porosity, density, and sorption coefficient were measured in order to characterize the materials studied. The porosity and density were determined according to Archimedes' principle (weighing in air and water). The sorption coefficient was determined on specimens dried in, and in equilibrium with, a normal room climate (approximately 21°C and 50% relative humidity). The results of this characterization are shown in Table 1.

TEST OF NECESSITY OF KAOLIN CLAY

In this study the effect of the bedding between the specimen and the ceramic plate was investigated. Apart from the beddings changing, the water retention curve measurements performed generally followed the instructions in Nordtest BUILD 481.

TABLE 1. Mean Values of Porosity, Density, and Sorption Coefficient for Air-Dried Specimens

Material	Porosity (%)	Density ($\mathrm{kg/m^2}$)	Sorption coefficient[a] [$\mathrm{kg/(m^2\ s^{1/2})}$]
Lime silica brick	28 (0.006)	1,894 (15)	0.08 (0.004)
Autoclaved aerated concrete	79 (0.004)	594 (12)	0.098 (0.008)
Cement mortar	22 (0.013)	2,053 (40)	0.015 (0.004)
Cement-lime mortar CL 50/50/650	30 (0.008)	1,853 (20)	0.091 (0.007)
Brick 1, Kanik Antik	26 (0.008)	1,978 (18)	0.35 (0.005)
Brick 2, Kanik Gul (hard burned)	33 (0.002)	1,863 (4.2)	0.13 (0.006)

Note: Standard deviation is in parentheses.
[a] Slope of early water absorption curve (Fig. 4).

The equipment used in measuring retention curves was initially designed for soil science. In adapting it for use with solid materials, saturated kaolin clay is generally used to ensure a perfect joint between the specimen and the ceramic plate and good hydraulic contact between the specimen and the ceramic plate. To prevent the kaolin from penetrating the specimen a fine cloth is placed between the kaolin and the specimen (Fig. 1). The use of kaolin clay and cloth is prescribed in the Nordtest standard NT BUILD 481. However, kaolin clay is never used on the cellulose membrane because the saturated cellulose membrane is soft and therefore the specimens have good hydraulic contact with it without any kaolin clay.

To test whether the kaolin clay is really required, tests were performed on the red brick in which the kaolin clay was used, omitted, or replaced with a microfiber cloth manufactured of 85% polyester and 15% polyamide. Approximately 10-mm high specimens were cut from the bulk of the brick with a diamond saw without any further processing. The water retention curves were measured on two separate occasions in an air-conditioned room at 20°C ± 1°C (23°C ± 1°C is prescribed in Nordtest standard NT BUILD 481). Before starting the test, the specimens were vacuum saturated using the procedure described by Fagerlund (1977).

In the first run, six specimens were used, all placed on a bedding of kaolin clay with a cloth separating the kaolin clay from the specimen. Seven pressure set points were used in the range of 0.13–100 bar.

In the second test, different beddings were tested simultaneously during the same run. Thus the water retention curves with the different beddings were measured under exactly the same conditions. In total, six specimens were placed on kaolin clay, nine on the microfiber cloth, and six directly on the ceramic plate. In this test, three pressure set points were used in the range of 0.1–10 bar.

Results

The measured water retention curve is represented with the suction as storage potential. The moisture content is represented by both the degree of vacuum saturation S_{vac} and the moisture content mass by mass u. To show the magnitude of the moisture content of the ambient air that various suctions correspond to, the relative humidity, calculated with (3), is also used in the water retention curve. Each dot in the water retention curves shown in Fig. 3 corresponds to a mean value of the moisture content of several specimens (the standard deviation of S_{vac} is maximum 0.007). It is clear that there is no significant difference between the result obtained on brick 1 when the kaolin clay was used, omitted, or replaced with a microfiber cloth.

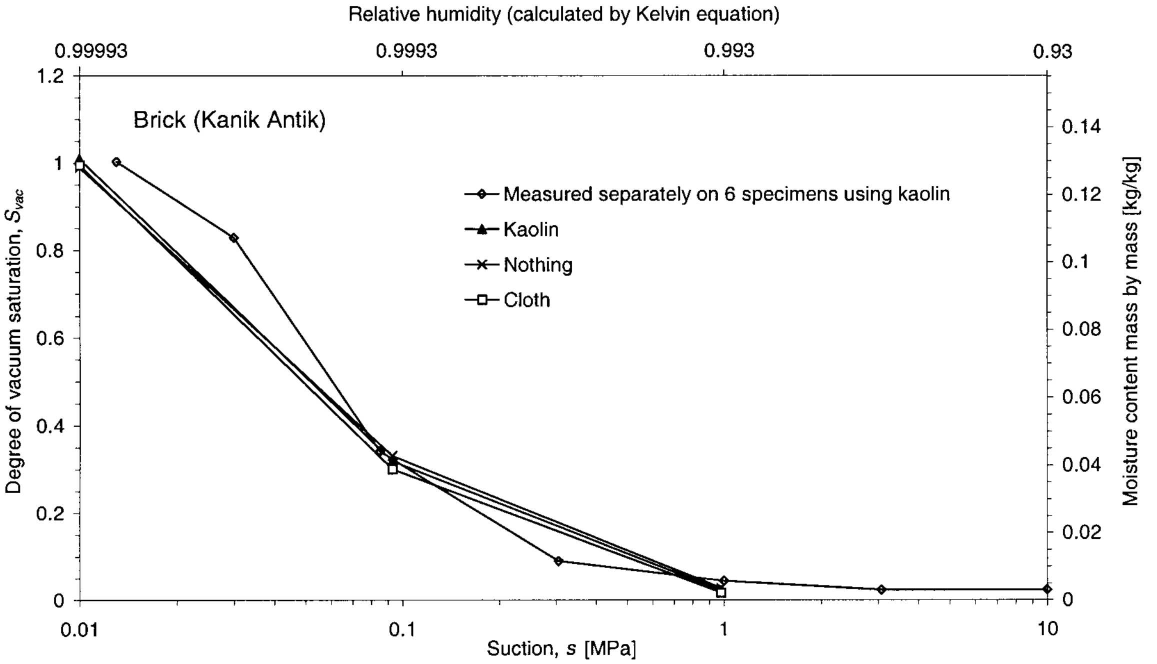

FIG. 3. Retention Curves for Brick 1, Red Brick "Kanik Antik"

COMPARISON BETWEEN METHODS OF PRESATURATING SPECIMENS

In this section of the study, the effect of the method of presaturating the specimens is studied. Apart from the changes in the method of presaturation, the water retention curve measurements performed followed the instructions in Nordtest BUILD 481.

Specimens must be saturated before they are tested, and there are two different ways to do this. Either the specimens can be capillary saturated or they can be vacuum saturated. Both methods have been advocated in the literature. For example, Krus (1995) and Janz (1997) used capillary saturation, while the standard Nordtest BUILD 481 prescribes vacuum saturation.

Provided that capillary and vacuum saturation differs on the material in question, the water retention curve will also differ. To investigate this effect, water retention curves were measured on both capillary and vacuum saturated specimens of lime silica brick. In total, 20 specimens with a height of approximately 5 mm cut from the bulk of the lime silica brick were used. Half of them were dried and thereafter submerged for 24 h, and in that way were capillary saturated. The remaining specimens were vacuum saturated using the procedure described by Fagerlund (1977). The water retention curves on the capillary and vacuum saturated specimens were measured simultaneously. Hence, all other circumstances were equivalent, and any differences between the curves are solely the product of the method of saturation used. Nine pressure set points were used in the range of 0.02–100 bar. The tests were carried out in an air-conditioned room at 20°C ± 1°C.

The correct definition of capillary saturation is the moisture content in the material when the first rapid capillary absorption comes to an end (Fig. 4). This definition is, however, impractical when it comes to the presaturation of specimens since the time until capillary saturation is reached (t_c in Fig. 4) must be known for every single specimen used. It is therefore more convenient to redefine capillary saturation as the moisture content obtained after a certain absorption time. To be on the safe side, the absorption time used must definitely exceed t_c.

This adopted definition was used in the test performed on the lime silica brick (submerged in water for 24 h). Gradual water uptake will, however, also occur after capillary saturation, because there is always a certain amount of enclosed air remaining in the pore system. This enclosed air will gradually dissolve in the water and diffuse to larger pores or out of the pore system to the surface of the material, which will lead to slow water absorption above capillary saturation. The rate of this dissolution is proportional to the solubility of air in water, which is in turn, proportional to the internal overpressure in the enclosed air. Thus the rate of gradual water absorption will be proportional to the dissolution rate of air in water and the diffusivity of air in the pore water. The moisture content obtained when the prescribed absorption time is reached will consequently exceed the correctly defined capillary saturation.

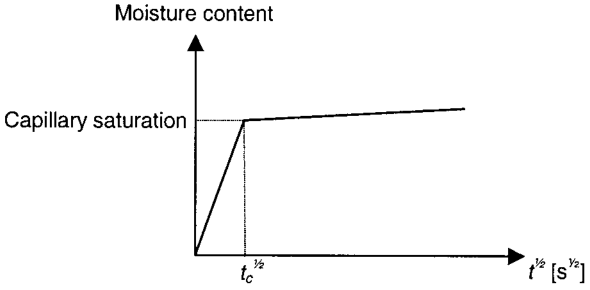

FIG. 4. Definition of Capillary Saturation

Several absorption tests were performed in order to investigate how the obtained "capillary saturation" is dependent on the absorption time prescribed and the size of the specimens used. The effect of the absorption method used—either submersion or exposure to water on one side—was also studied.

The influence of absorption time was tested on four specimens each of autoclaved aerated concrete, cement mortar, and cement-lime mortar CL 50/50/650 with heights of 21, 7, and 25 mm, respectively. Two specimens of brick (yellow and hard burned) were also tested. The heights of these were 11 and 25 mm. The tests were performed on specimens dried in a normal indoor climate and then exposed to water on one side during the absorption. The amount of absorbed water, the time until capillary saturation was reached t_c, the moisture content at capillary saturation u_{cap}, and the moisture content after 12, 18, and 24 h were registered.

A further absorption test was performed on brick 1 (the red brick), examining primarily the influence of specimen size. In this test as well, the specimens were dried in a normal indoor climate, but they were submerged in water during the absorption. During the absorption test the relation between moisture content and time was registered. The test was carried out on four specimens with a height of approximately 5 mm and two specimens with a height of approximately 25 mm. Both the absorption tests were performed in an air-conditioned room at 20°C ± 1°.

Results

The water retention curves measured on both capillary and vacuum saturated specimens of lime silica brick are presented in Fig. 5. Each dot in both water retention curves shown corresponds to the mean value of the moisture content of several specimens. As can be seen, no excess water was forced out of the specimen at pressures below 0.009 MPa. The water content of the capillary saturated specimen even increased slightly in the pressure range 0.002–0.009 MPa due to water absorption in air-filled pores. At a pressure of 1 MPa the two curves joined, and thereafter there are no differences between them.

The absorption tests noted the time until capillary saturation was reached (t_c), the moisture content at capillary saturation (u_{cap}), and the moisture content after 12, 18, and 24 h [$u(12)$, $u(18)$, and $u(24)$, respectively]. The moisture content at vacuum saturation u_{vac} was measured separately. The results appear in Table 2.

The result of the absorption test on submerged specimens of brick 1 with different heights is shown in Fig. 6. The absorption rate of the specimens with a height of 5 mm is considerably higher than that of the 25 mm specimens. This distinction between the two heights becomes even more obvious after capillary saturation is reached. The 5 mm specimens reach a moisture level corresponding to vacuum saturation after approximately 5 days, while the 25 mm specimens do not reach the vacuum saturation level during the test period of 1 month.

DISCUSSION AND CONCLUSIONS

Effect of Bedding

In the experiment testing whether kaolin clay is required for the hydraulic contact between the specimen and the ceramic plate, kaolin clay was used, omitted, or replaced with a microfiber cloth. The results indicate that kaolin clay is unnecessary since there is almost no difference between the results obtained with different beddings (Fig. 3). It was expected that the use of kaolin clay and microfiber cloth would yield similar results, but it came as a surprise that the same state of equilibrium was reached when no bedding at all was used. However, only a single test was performed and the time to reach equilibrium could not be measured for the different beddings since they were tested during the same run. It may be that the hydraulic contact was poorer for the specimens placed on a microfiber cloth or directly on the ceramic plate. The time to

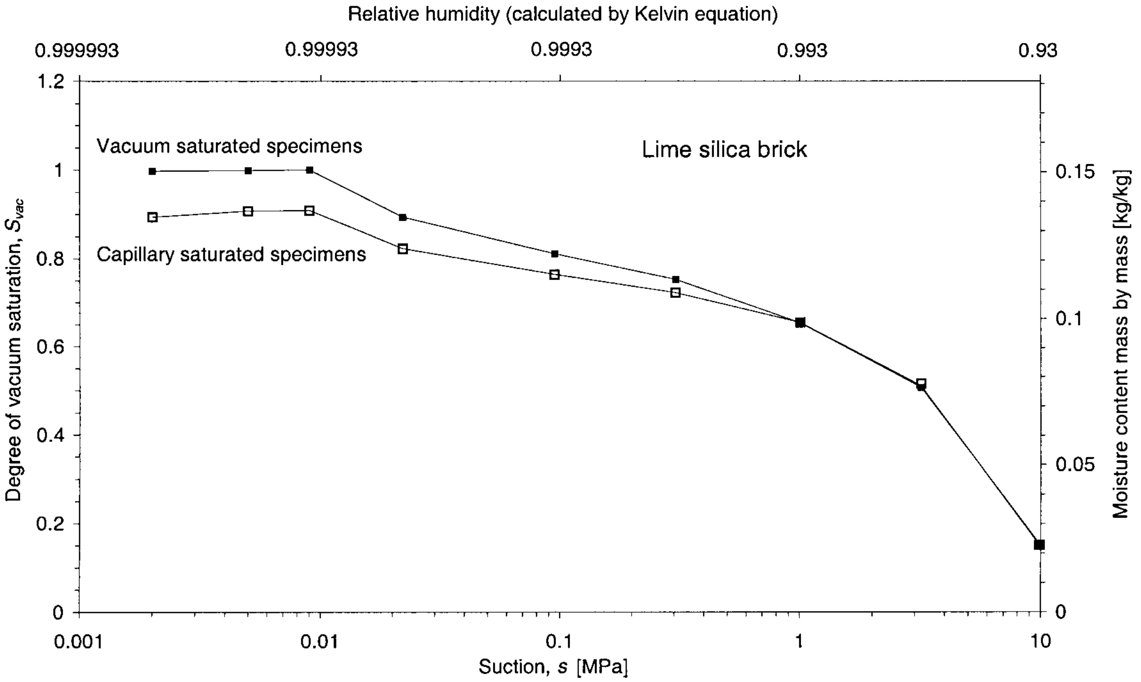

FIG. 5. Water Retention Curve of Lime Silica Brick Measured on Capillary and Vacuum Saturated Specimens

TABLE 2. Results of Absorption Tests

Material	Height (mm)	t_c (min)	u_{cap}	$u(12)/u_{\text{cap}}$	$u(18)/u_{\text{cap}}$	$u(24)/u_{\text{cap}}$	$u_{\text{vac}}/u_{\text{cap}}$
Autoclaved aerated concrete	21	100	0.8	1.012	1.014	1.018	1.875
Cement mortar	7	60	0.0888	1.020	1.023	1.028	1.182
CL 50/50/650	25	30	0.116	1.052	1.069	1.086	1.379
Brick 1[a,b]	5	0.1	0.0937	1.141	1.173	1.203	1.387
Brick 1[a]	25	1.3	0.0919	1.047	1.057	1.067	1.414
Brick 2	11	40	0.112	1.040	1.054	1.071	1.563
Brick 2	25	160	0.12	1.042	1.058	1.075	1.458

[a] Brick 1 was submerged in water; all other specimens were exposed to water on one side.

[b] Specimens had reached capillary saturation at first weighing occasion.

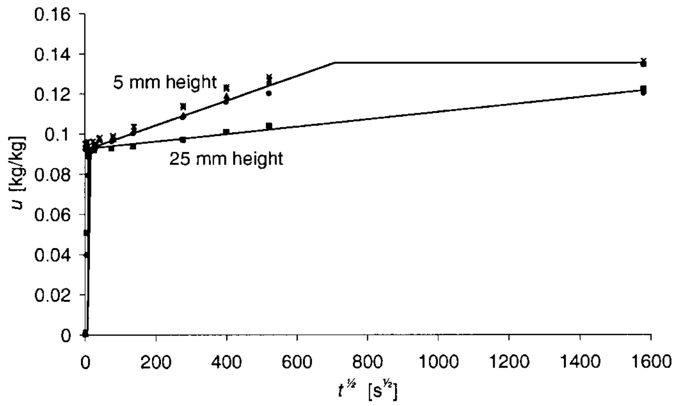

FIG. 6. Result of Absorption Test Performed on Specimens of Brick 1 with Height of 5 and 25 mm, Respectively

reach equilibrium might therefore differ between the methods, even if the state of equilibrium is the same. Further investigation is needed before a firm recommendation can be made to omit kaolin clay from the test procedure.

A side effect of this test was to show that the repeatability of water retention curve measurements is high. The results differ only slightly between the two independent measurements performed. In Hansen et al. (1999) it also appears that reproducibility is quite high, because water retention curves measured by the participating laboratories in the round robin test were similar. The test procedure therefore seems to have a high level of reliability.

Effect of Presaturation

As expected, the water retention curves of capillary saturated specimens differ from those of vacuum saturated specimens (Fig. 5). It is debatable which method of presaturating the specimens has the greatest validity. On the one hand, normally exposed building materials seldom have moisture contents that exceed capillary saturation, which points toward using capillary saturation. Materials exposed to water for a longer time might, on the other hand, reach a saturation level exceeding capillary saturation. Examples of materials with such exposure are parts of a structure exposed to groundwater or a wall behind a leaking drainpipe. It can also be argued that when the test results are to be used to characterize the material in regard to pore size distribution, vacuum saturation is preferable since not all pores will be measured on capillary saturated specimens.

Several practical factors concerning the difficulty of defining capillary saturation also point to vacuum saturation as the best way to presaturate specimens. The results from the absorption tests indicate problems that might occur when capillary saturation is used.

To a have manageable test procedure, capillary saturation is often defined as the absorption time needed. The prescribed absorption time must exceed true capillary saturation, whereafter slow water uptake will take place. Hence, the moisture content reached will differ from true capillary saturation. How much it differs is dependent, among other things, on the material in question. For materials with very slow absorption rates above capillary saturation, the error will be insignificant. For example, the autoclaved, aerated concrete reached a saturation level that exceeded capillary saturation by only 1.8% after 24 hours' absorption (Table 2). Other materials, however, have a considerable absorption rate even after capillary saturation. The 5-mm thick specimens of brick 1 reached a water content that exceeded capillary saturation by 14% after 12 h, 17% after 18 h, and 20% after 24 h (Table 2). Thus, the moisture content of the brick is in an indefinable area between capillary and vacuum saturation (which exceeds capillary saturation by 39%). A prescribed absorption time is consequently not suitable for some materials because of uncertainty about the moisture level reached.

Gradual water uptake may even occur during a test run when pressure is applied to the specimen. Such an occurrence is reported by Janz (1997) where a water retention curve was measured on Gotland sandstone that was presaturated through capillary saturation. A small amount of absorption also occurred on the capillary saturated lime silica brick tested here (Fig. 5). This results indicates that there is a unique relation between suction and moisture content even above capillary saturation. The state of equilibrium is reached when there are no air bubbles in the pore system with a smaller radius than would be predicted based on the existing suction in accordance with (4). In the case of the sandstone, where the applied pressure and consequently also the suction were 0.007 MPa, (4) gives a radius of 0.02 mm. That is, air bubbles with a radius <0.02 mm would dissolve in the water and diffuse to larger pores or out of the store. Consequently, all air bubbles would dissolve if zero pressure (atmospheric pressure), corresponding to an infinite radius, were applied. Accordingly, should a water retention curve be measured through absorption, an equilibrium above capillary saturation would be reached when no bubbles smaller than $2\sigma/s$ exist. It would, however, take considerable time for most materials to reach this equilibrium.

If the above reasoning is correct, water retention curves measured from capillary saturation are actually scanning curves from absorption to desorption. That is, the correct desorption curve starts at vacuum saturation. The water retention curve measured on the capillary saturated lime silica brick also behaves as a typical scanning curve that slowly approaches the desorption curve (Fig. 5). The scanning curve from capillary saturation is, however, more important for some applications than the correct desorption curve.

However, Krus and Künzel (1992) reported on a test that contradicts the existence of any unique relation between suction and moisture content above capillary saturation. Their test showed that no equilibrium was reached in materials saturated to levels above capillary saturation for 40 days. But 40 days is perhaps too short a measuring period since absorption above capillary saturation is a very slow process. According to Janz (1997), it took 230 days to reach vacuum saturation during an absorption test performed on Gotland sandstone, and the 25-mm thick brick submerged in water had not reached vacuum saturation after 30 days (Fig. 6). Further work is called for in the area of absorption above capillary saturation.

The absorption tests performed on brick 1 also clearly show that size has a considerable effect on the moisture level reached after a certain time. As mentioned above, the 5-mm specimens reached a moisture content that exceeds capillary saturation by 20% after 24 h, while the 25-mm specimens only reached a moisture level that exceeds capillary saturation by approximately 7% after the same period. Thus, if 24 h of submerged absorption is prescribed, the moisture content of the presaturated specimens will differ considerably depending on their size.

Furthermore, for some materials, capillary absorption will stop at different moisture levels dependent on the moisture content of the material before the absorption starts. Fig. 7 shows Schwarz' (1972) measurements of the capillary absorption of a brick. Depending on the initial moisture content, different capillary saturation levels are reached. The difference between the highest and the lowest level is approximately 10%. These results also indicate that vacuum saturation is probably preferable.

To sum up, vacuum saturation is the preferred option, especially if the results are also to be used to characterize the

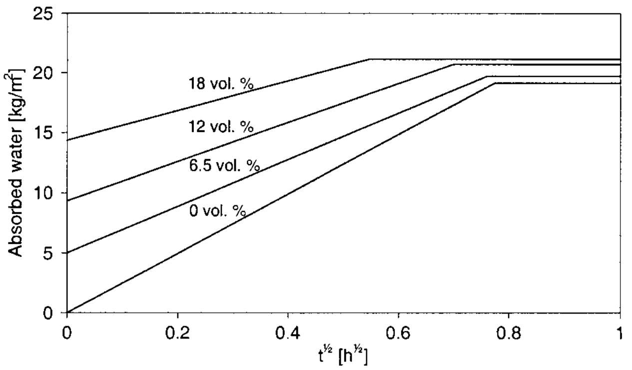

FIG. 7. Capillary Absorption of Brick with Density of 1,745 kg/m^3; Specimens Were Conditioned to Different Initial Moisture Contents before Absorption Test (0, 6.5, 12, and 18% by Volume) (Schwarz 1972)

material by pore size distribution. The results obtained on vacuum saturated specimens will be the same independent of their size and the moisture level before the saturation procedure. Vacuum saturated specimens also provide the correct desorption curve, while capillary saturated specimens provide scanning curves from absorption to desorption. Capillary saturation may also sometimes give indistinct saturation levels that depend on the prescribed saturation time and specimen size. If capillary saturation is to be used, a prescribed absorption time can only be used for materials with a very slow absorption rate above capillary saturation. Whether the absorption rate is slow or not must be decided case by case. The correct definition of capillary saturation (Fig. 4) must be used on materials with a high absorption rate above capillary saturation. A prescribed absorption time is inadvisable for such materials. However, determining an accurate definition of capillary saturation involves an arduous presaturation procedure.

Effect of Specimen Size

The absorption test also yielded additional interesting results. For some materials the gradual water uptake above capillary saturation will continue until the same water content as after vacuum saturation is reached. It appears in Fig. 6 that this occurred after approximately 5 days for the 5-mm specimens of brick 1. Janz (1997) reported similar results. Specimens of Gotland sandstone with a height of 25 mm exposed to water on one side reached vacuum saturation after approximately 230 days. The rate of the gradual water uptake above capillary saturation is obviously dependent on whether or not the specimens are submerged. But the test results also show with clarity that the time until vacuum saturation is reached and the absorption rate of this gradual water uptake above capillary saturation is strongly dependent on the size of the specimens. It has been suggested that the dissolution of air, and consequently water absorption, is taking place almost simultaneously throughout the entire specimen body of specimens that are below a certain thickness (Fagerlund 1979, 1993). These results in the present study indicate that this assumption may be wrong.

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