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TECHNICAL NOTE

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Improvements to Water Vapor Transmission and Capillary Absorption Measurements in Porous Materials

Reference

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ABSTRACT

The vapor permeability (or equivalently the vapor diffusion resistance factor) and the capillary absorption coefficient are frequently used as inputs in hygrothermal or heat, air, and moisture (HAM) models. However, it has been well documented that the methods used to determine these properties are sensitive to the operator, and wide variations in the properties have been reported in round-robin testing. This paper presented an investigation into how these errors can be minimized for porous materials by different edge sealing techniques and also looked at whether automating these techniques can reduce operator artifacts. To automate the measurements, specimens were attached to a balance or load cell and then required no further interaction, which allows massive amounts of data to be collected. The extra data is advantageous for the beginning of the capillary absorption test where the moisture uptake is rapid. Most of the potential for errors in the vapor diffusion tests resulted from uncertainties in how the sample was sealed between the chambers and determining when the steady state region was reached, neither of which can be improved by automation.

Keywords

vapor diffusion, capillary absorption, wood-moisture relations, hygrothermal modeling

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Introduction

Heat, air, and moisture (HAM) and hygrothermal (heat and moisture) modeling are widely used in building science in the analysis of building enclosure design and investigation of building failure [1–3]. Accurately predicting the hygrothermal performance of buildings and assemblies through

HAM models requires accurate material property data on the storage and transport of moisture and heat. Moisture storage is characterized by the sorption isotherm in the hygroscopic region and by either the pressure plate technique or mercury intrusion porosimetry in the overhygroscopic region. Similarly, moisture transport is described through two tests. Water vapor transmission is characterized through a "cup" test where the material is sealed to the top of a cup containing desiccant, water, or a saturated salt solution causing a difference in water vapor partial pressure across the material. Standard methods for measuring water vapor diffusion include ASTM E96/E96M-15 [4] and ISO 12572 [5]. Liquid water transfer can be described in hygrothermal models using a diffusivity approach which requires data from a capillary absorption test [6–8]. In this test, a material is brought into contact with liquid water. If the material obeys Fick's law, there should be a linear region when the water uptake (mass per unit area) is plotted as a function of the square root of time and the resulting slope, called the capillary absorption coefficient, or "Acap" can then be used to calculate the liquid water diffusivity. This test is standardized in both ISO (EN ISO 15148 [9] and ASTM C1794-15 [10].

It is generally recognized that measurements of moisture storage are easier and more repeatable than moisture transport measurements [11,12]. For vapor diffusion, numerous interlaboratory round-robin tests have been conducted and differences as high as 400 % have been reported between laboratories for identical materials [13–17]. Measurements are affected by changes in buoyancy of the sample caused by changes in atmospheric pressure, the vapor resistance of the air layer inside and outside of the cup, transport through "masked edges" on the sample surface, as well as operator error such as improper sealing and allowing liquid water to splash on the specimen surface when moving the cup [12,18,19]. Galbraith [16] noted that individual laboratories were consistently on one side or the other of the median which suggested that there were systematic errors in how the tests were conducted. Less work has been done examining the uncertainties in the capillary absorption test [12,20]. Roels et al. [12] noted problems with the ability to collect data rapidly enough near the origin; sparse, unevenly spaced points made it hard to reliably select and extrapolate a linear region [12]. Bomberg et al. [20] reviewed several unpublished studies that examined uncertainties in capillary absorption measurements. The greatest errors were caused by differences in sealing the edges perpendicular to the surface of the water [20]. Other factors, such as how the surface was blotted before weighing, time between measurements, and the initial moisture content also caused statistically significant differences.

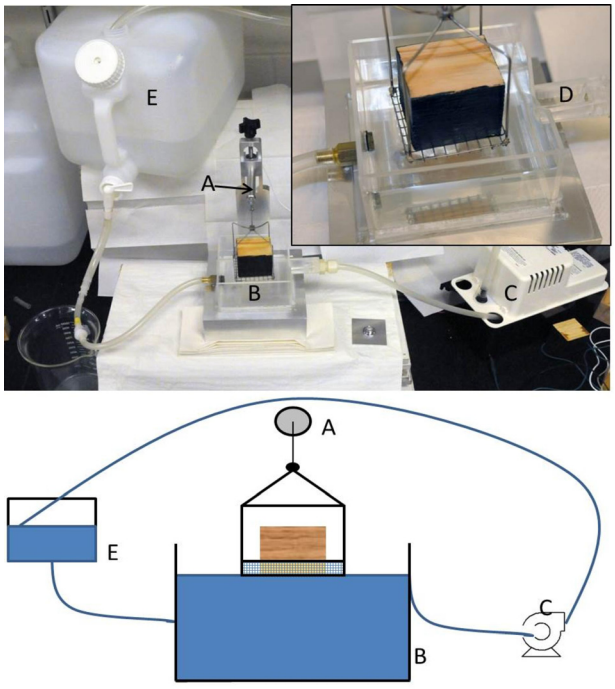
This paper examines potential ways to improve the quality and repeatability of vapor diffusion and capillary absorption tests. From the literature, it is clear that improper sealing can have a large effect on the measured values for both the capillary sorption and vapor diffusion measurements, although a wide variety of sealing materials have been used. In this work, we compare several sealants for both the vapor diffusion and capillary absorption tests. Additionally, we explore automation as a potential way to reduce operator artifacts and to see if the quality of the measurements could be improved by obtaining more data than is possible in traditional, manual, measurements. Relatively little prior work has examined automated moisture transport testing. Vapor diffusion measurements have been automated by placing the material at the interface of two chambers and measuring the moisture flux from one chamber to the other [21–24]. Even less work has been done in examining potential automated methods for capillary absorption testing [25]. The discussion of this paper focuses on potential best practices to ensure high quality data in these measurements.

Materials and Methods

CAPILLARY ABSORPTION

The apparatus for measuring capillary absorption is shown in **Fig. 1**. It is capable of collecting data at a rate of 25 kHz and detecting changes in mass of 0.02 g, although for file-size optimization, we collected data at no more than 0.5 Hz and reduced to 1 sample per minute after the first 800 data points. The specimen sits on the bottom of a basket made from galvanized wire that is brought into contact with a reservoir of

FIG. 1 Apparatus to collect capillary absorption data, with schematic image below. The specimen is suspended from a load cell (A) and placed in contact with a water reservoir (B), whose level is controlled by a weir (D), a pump (C), and a secondary reservoir (E). The insert is a closer view of the specimen, reservoir, and weir (D).



water (B). The basket is attached to a hook so that it can rotate and does not place eccentric loads on the load-cell (A). The longest tests lasted for 2 days which results in a potential error of 0.2 g. Water level was maintained by a pump (C) and weir (D) to control for water level changes caused by evaporation and absorption.

As others [12,20] have noted, the choice of sealants for the sides of the specimen can have a large effect on the results of the test. On preliminary measurements of southern pine, the sides were sealed with hot paraffin wax and we measured a value of 0.033 kg m^{-2} $s^{-0.5}$ in the transverse direction, about a factor of 10 higher than published values for pine, which range from 0.0014–0.0040 kg m^{-2} $s^{-0.5}$ [26,27]. Because of this large discrepancy, we tested several different sealing materials: paraffin, neoprene paint, shoe polish, and 2 different commercially available polyurethane sealants on blocks cut from the same parent board. We found that the neoprene paint gave the lowest measured value for Acap (0.0025 kg m^{-2} $s^{-0.5}$), nearly half that of the next lowest sealant, the polyurethanes, which ranged from 0.0040 to 0.0048 kg m^{-2} $s^{-0.5}$. In addition to providing superior edge sealing properties, the neoprene paint had several other advantages: it provided adequate coverage in one coat and, because it was black and opaque, it was easy to confirm that the sides were entirely covered.

Because the sample was suspended, the data were sensitive to small changes in the water level that could not be observed with the naked eye. We managed this by controlling the water level by flowing water through an in-line flow restrictor at 131 mL/min into the reservoir; the water was emptied on the other side of the reservoir over a weir with a V-notch to maintain the water level constant. Although the change in water level was too small to be observed, it resulted in a very small saw-tooth pattern (amplitude 0.2 g) superimposed over the data.

We tested several designs to connect the specimen to a weighing device during the uptake experiment before arriving at the final design. It was originally envisioned that the specimen could sit on a platform connected to an underwater load cell. However, in our original testing we found that the level of drift in the underwater load cells was unacceptable. We measured drift by placing a 200 g weight on the load cell and measured load as a function of time. We observed 3.5 g of drift over 48 h (0.04 % drift per hour).

The final design used a load cell located above the water, which held a basket that contained the specimen. This allowed us to use a load cell with much lower creep (listed at 0.1 % of full scale). Using our setup with a 100 g weight, we observed a 0.03 g drift over 48 h (<0.001 % drift per hour). The load cell was used in a temperature controlled environment, and allowed to warm up before use, to reduce thermal drift. Electrical noise in the system was reduced by averaging 400 readings taken over a span of near 0.25 s for each recorded data point. The resolution of 0.02 g was determined by averaging the variation in readings over repeated measurements of a stable weight. The system accuracy was primarily determined by a software calibration factor used to match the system voltage output to true mass, resulting in accuracy better than 0.1 g.

While the overhead load cell was more stable, it presented the additional challenge of aligning the specimen so that it did not produce an eccentric load and keeping the face of the specimen parallel to the surface of the water. To prevent an eccentric loading of the load cell, we created a coupling so that the basket could swivel freely from the end of the load cell. We kept the specimen parallel to the surface of the water by placing a small metal plate on the upper surface of the specimen that had a bulls-eye level in the middle of it. The level was necessary to align the specimen on the basket. The plate served two purposes: minimizing evaporation from the top surface and assuring that buoyant forces did not lift the specimen during the uptake experiments on wood. The plate stayed on top of the sample for the entire test.

VAPOR DIFFUSION

One potentially large source of experimental error in diffusion cup measurements, as mentioned previously, is an imperfect seal. Our cup was designed so that the samples could be interchanged easily between different dishes or conditions without resealing the sample. It consisted of a stainless steel canister and a stainless steel lid with a window where the sample could be mounted (**Fig. 2**). The lid was sealed to the canister with a fluoroelastomer ring flange gasket that was compressed by applying force to the lid using wing nuts on threaded rods secured to a base which held the container. With this apparatus, the specimen could be mounted once and moved between all vapor

FIG. 2 Cup used in the vapor diffusion test. The lid was sealed with a gasket held down with threaded rods. The edges of the sample were sealed with butyl tape (inset).

diffusion conditions. By only sealing the specimen one time, it eliminated a source of error within measurements of a single specimen.

Several different methods were used to permanently mount the specimen to the window: sealing gum, paraffin wax, foil tape, hot melt adhesive, and butyl tape. Their ability to seal the cup was measured by sealing an impermeable sheet of stainless steel over the window. We found that the leakage of the various sealing materials decreased in the following order: hot melt adhesive > sealing gum > foil tape > butyl tape ≈ wax. Svennberg and Segerholm [28] tested the water vapor transmission of the sealing materials by coating the surface of a cardboard specimen with the sealing material and mounting the modified cardboard to a cup. While differences in the method preclude an absolute comparison, both foil and butyl tape performed well in both sets of experiments. Butyl tape and paraffin wax both gave leakage rates of 3–7 mg per day in our experiments. We attribute this leakage to the gasket on which the lid sat and not butyl tape or wax seal, since in other experiments where stainless steel lids were sealed directly to dishes with butyl tape no measureable leakage could be detected after several weeks. The leakage rate through the gasket with our sample area of 8570 mm^2 requires a correction to the specimen permeance of approximately 15 ng Pa^{-1} s^{-1} m^{-2}.

Other than the difficulties in the cup test itself, automation of the test was straightforward. The cup sat on a balance (0.01 g resolution) located inside of an environmental chamber capable of maintaining ±0.1 % RH and ±0.1°C. The balance was connected to a personal computer which recorded data every 3 min. The largest difficulty was that the environmental chamber produced vibrations, which in turn vibrated the scale and caused the data to appear noisy (shown later). We were able to reduce the noise to approximately 0.2 g oscillations by placing the balance on a heavy plate inside of the chamber and isolating it with a viscoelastic urethane polymer made specifically to reduce vibrations. As will be discussed below, the effect of this noise on measurement error was negligible.

The permeability was calculated from the rate of change in mass with respect to time (slope) with measurements taken every 3 min across a period of multiple days or weeks, depending on the type of material, the sample thickness, and the boundary conditions. The slope changes as moisture transfer approaches steady state and the sample establishes equilibrium with the relative humidity of the boundary conditions. Slopes are fit to successive sample periods until the change in slope is small, typically less than 5 % change between periods; this condition often takes 3 weeks or more to achieve for materials such as wood. Statistically, the mass oscillations mentioned previously have little effect; the standard error in the slope was typically less than 0.3 % of the slope.

Results and Discussion

CAPILLARY ABSORPTION

Fig. 3a shows the results of capillary absorption tests of several different materials collected with the apparatus at 22°C. Tests on the gypsum, fire brick, and house brick were terminated the next working day; however, the wood was clearly still gaining moisture, so it was transferred to another container and the test was continued manually for over a month. **Fig. 3b** shows the first hour of data. It is clear that the gypsum and fire brick have reached capillary saturation in this period.

Bomberg et al. [16] plotted the derivative of water uptake with respect to the square root of time to determine when capillary saturation has been reached; the slope has a constant, but non-zero, value in the "Acap" region, which then trails to zero once saturation has been reached [20]. We show the calculated slopes in **Fig. 4**. All the materials appear to exhibit a region of constant slope in the first 4 min of the test. The constant slope region is short for gypsum, it extends from 8.5 to 13.9 $s^{0.5}$, for fire brick 3.9 to 7.7 $s^{0.5}$, for the house brick 7.7–14 $s^{0.5}$. Interestingly, wood also appears to have a constant slope region from 10–13 $s^{0.5}$, similar to the location and duration of the linear

FIG. 3

Water uptake as a function of square root of time for the entire test (a) and the first hour of the test (b).

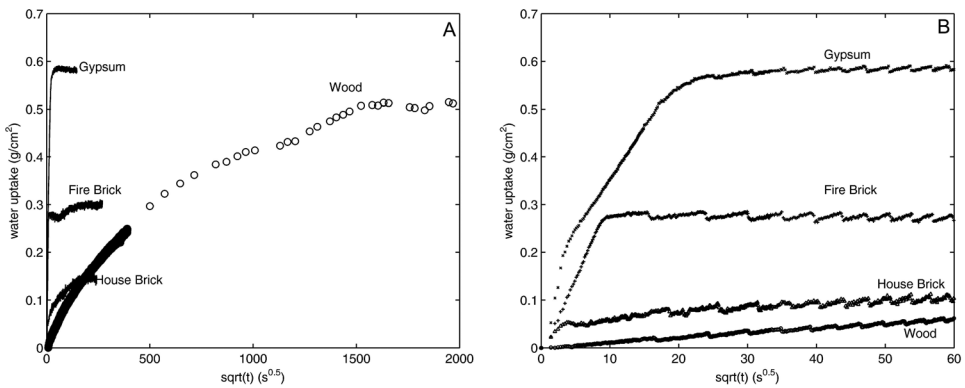
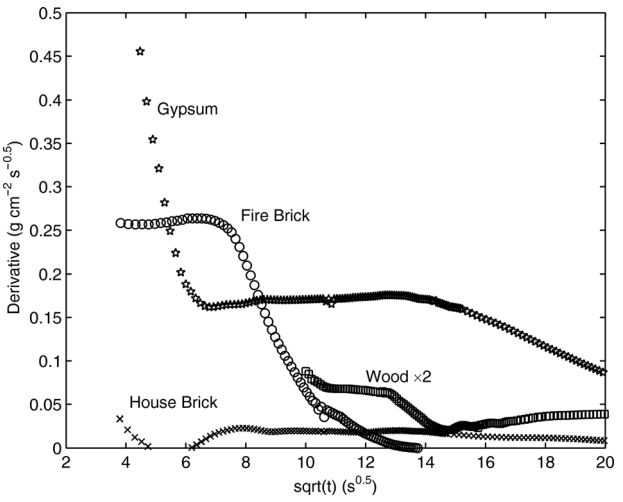


FIG. 4 Derivative of the capillary water uptake with respect to the square root of time. The data for wood was multiplied by a factor of 2 for clarity.



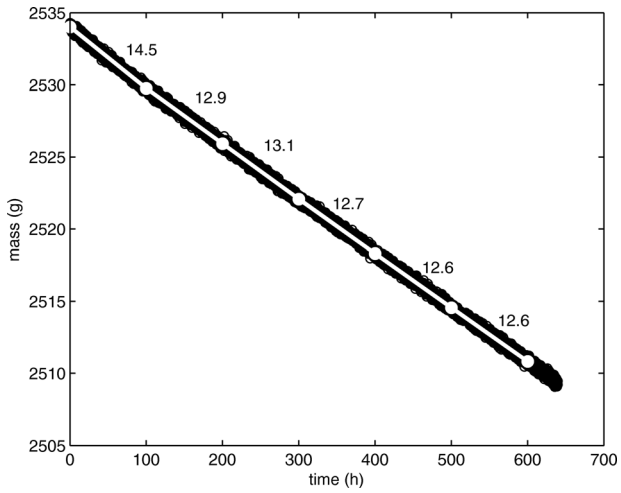
regions in the other materials. However, in wood, this constant slope region may not be representative of Acap since the data do not appear to have a linear region when the whole curve is plotted (**Fig. 3**).

It is interesting that the derivative of the uptake exhibits a sharp decrease at the very beginning of the test for several of the materials lasting only a few seconds. We were able to capture this data because of the automation; in a traditional test this data would not be accessible. From these limited measurements, it is not clear whether the pre-constant slope data have physical meaning, or are instead an artifact of the test. Physically, it is possible that this data is non-Fickian because the decrease of interfacial energy causes a rapid "jump" when the water contacts the material. It is also possible that this initial data is an artifact of how the sample is brought into contact with the water.

VAPOR DIFFUSION

Fig. 5 shows typical vapor diffusion data collected with the system. The data are from wood in the radial direction with boundary conditions of 94 % RH on the inside and 50 % RH on the outside of the cup at 23°C. The white line/markers represent linear regressions of 100 h increments of the data. The slope changes considerably during the first 400 h (approximately 2 weeks) as moisture is desorbing from the sample (the test prior to this one was run at a higher RH condition: 94 % inside and 80 % outside). After this time, the slope remains roughly constant for the remainder of the test. For the data shown in **Fig. 5** for the next 200 h, the final slope was approximately constant from 300 to 600 h and had a value of 12.6 ± 0.1. If taken as the true permeability, the slope in the first 100 h represents a 15 % overestimation of the permeability.

FIG. 5 Example result of a vapor diffusion test on wood (radial direction) with boundary conditions of 94 and 50 % RH. The white line shows the average slope over 100 h segments and the number next to the line is the water vapor diffusion resistance factor (or μ-value) for the corresponding 100 h segment.



OPTIMIZING THE SEALING METHODS

We found that the sealing methods were extremely important in both the vapor diffusion and capillary absorption testing. For the capillary absorption measurements, the neoprene paint provided an excellent seal preventing liquid water absorption. Additionally, the neoprene paint was easy to use and was opaque, which helped in determining proper coverage.

For the vapor diffusion tests, even with the best sealants, we found a leakage rate of 3–7 mg per day. However, this was not caused by the permanent seal of the sample to the lid of the cup, but rather the gasket between the outside of the lid and the canister. We found that both butyl tape and paraffin wax could provide vapor tight seals in the vapor diffusion test. However, neither of these materials were easy to work with, with the butyl tape being the slightly easier of the two. No matter the sealing material, the leakage rates should be characterized as part of the measurements by running an additional cup with an impermeable material. ASTM already recommends that leakage rates should be routinely characterized and reported with measurements with gasketed seals [4]. However, as a good practice, this characterization should be performed with all measurements. If no leakage is detected, then there is confirmation of a good seal. If leakage is measured, then those measured values can be used to correct the data for the other samples in the experiment.

ADVANTAGES AND DISADVANTAGES OF AUTOMATION

The major advantages of the automated measurements are that data can be collected at an arbitrarily high frequency, with a higher resolution, with no labor after the test has started. This is especially advantageous in the capillary absorption experiments where the linear region is small and happens very shortly after

the beginning of the test. Because the sample does not need to be removed from the water bath, an arbitrarily large amount of data can be collected; furthermore, measurements are not affected by blotting prior to weighing. Interestingly, three out of the four materials tested exhibited a rapidly decreasing slope within the first few seconds of the test prior to having a constant slope (**Fig. 4**). This region of the curve would not be accessible in manual tests, and may suggest that the absorption mechanism exhibits non-Fickian behavior at extremely short times.

For the vapor diffusion experiments, the advantages of automation are less pronounced. Since these tests take much longer than the capillary absorption experiments, the abundance of data is of limited use. The ASTM E96 [4] vapor diffusion standard specifies that the test can be concluded when six continuous points form a straight line or when the plot of the weight versus time becomes linear [14]. With sparse data, determination of a linear region can be dependent on the person interpreting the data. While the data collected from the method herein provides enough data to ensure that a linear region exists, it comes at the cost of time. Whereas a single operator could run several tests in parallel, the automated methods require serial processing unless several balances are available. Furthermore, running the balance for several weeks without re-zeroing it causes a small amount of drift in the zero point. While the capillary absorption tests must also be run in series, this is less of an issue as the tests are much more rapid.

Conclusions

This paper examined errors in water vapor transmission and capillary water absorption tests caused by sealing methods and also whether automating these measurements can help give more reliable data. Several different sealants were tested for both the water vapor transmission test and the capillary absorption test. We draw the following conclusions from the data:

- Neoprene paint is an excellent sealing material for the water absorption test. It provided the least water penetration of the five sealants tested. It is also opaque, which is useful in confirming that the entire surface is covered.
- Butyl tape and paraffin wax are both good sealants for the vapor diffusion tests, and showed negligible leakage around the sample. The measured leakage was attributed to the fluoroelastomer gasket, which allowed mounted samples to be easily moved between different conditions. No matter the sealing system, for optimal results, the cup test should be run with an extra cup, run with an impermeable material sealed to the top. By using the impermeable material, the leakage rate of the seal can be measured and corrected in the cup test results.
- It takes a long time for a true steady-state condition to be achieved for certain materials in the vapor diffusion tests (i.e., **Fig. 5**). For these tests, the automation is not necessarily helpful since it would require serial processing. Whether the data collection is automated or not, the experiment should not be terminated until the rate of change of mass with time is constant.
- For the capillary absorption measurements, the automation does appear to have the advantage of rapid data collection near the origin. Additionally, the sample is constantly in contact with the water and does not need to be blotted, which could be another source of systematic errors. Automation appears especially promising for these measurements since the experiments do not need to be continuously observed (as in the manual method) and the tests are relatively short so parallel processing is not necessary.

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