A modified cup-method for lightweight and highly permeable materials.

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ABSTRACT: With an increasing interest for dynamic hygrothermal calculation models for short time periods, there is a need for uncomplicated and reliable methods to determine the moisture properties for materials used in the indoor environment. The traditional cup-method is commonly used for building materials to determine the water vapor permeability. When used on lightweight and highly permeable materials, the unidentified inner surface resistance of the cup-method will be problematic and fastening the sample to the cup can be troublesome. To address these problems, we have developed a modified cup-method based on standard laboratory crimp cap vials to determine the water vapor resistance for lightweight and highly permeable materials, such as textiles and paper. This modified cup-method together with a validation is presented in the paper.

1 INTRODUCTION

With an increasing interest for dynamic hygrothermal calculation models for short time periods, there is a need for uncomplicated and reliable methods to determine the moisture properties for materials used in the indoor environment. The traditional cup-method is commonly used to determine the water vapor permeability of building materials e.g. wood, flooring and board materials (e.g. ASTM 96-00, 2000, Hedenblad, G., 1993, McLean, R. et al. 1990, Joy, F.A. & Wilson, A.G., 1965). However, when used on lightweight and highly permeable materials, such as textiles and paper, the normally used cup-designs are difficult to use because thin materials cannot be fastened reliably in the cup and the high interior surface resistance is difficult to handle (Newns, A.C. 1950, Mackay, J.F.G., 1971).

Several attempts have been made previously to develop a method that deals with these problems. The draft to an international standard method ISO/DIS 15496 (2001) primarily to be used for quality control is an inverted cup-method for textiles where the cup is immersed into water. Nilsson, L. et al. (1993) used a diffusion cell where the test material was placed as a partition between two chambers with different relative humidity. Takada, S. et al. (2001) describe a modification of the wet-cup-method specialized for textiles.

The objective of this paper is to present a simple method for determining the water vapor permeability for lightweight and highly permeable materials using basic laboratory equipment.

2 METHOD

The modified cup-method to determine the water vapor permeability for lightweight and highly permeable materials is based on standard laboratory crimp cap vials and tear-away crimp caps. Saturated salt solutions inside the vials in combination with a well-controlled ambient climate create a gradient in vapor pressure. The material to be tested is placed on a highly vapor permeable, hydrophobic and micro-porous membrane as a lid over the vial opening and is held in place by the crimp cap. Figure 1, shows the details of the experimental configuration of the vial cup. The vial is placed upside-down in an airflow rack exposed to a controlled airflow and a constant climate, Figure 2.
Figure 1. The vial (a) with a diameter of 20 mm and a height of 75 mm is charged with a saturated salt solution (g) with an excess of salt (f) to insure that solution is saturated during the experiment. A Teflon coated silicone seal (b) is placed with the coating towards the glass rim. A vapor permeable membrane (c) (e.g. Durapore GVHP, Millipore, Bedford, USA) ensures that only water vapor is transported through the test material (d). The crimp cap (e) holds the seal, membrane and test material in place and provides an edge masking of the test material. The holes in the seal and in the crimp cap are 14 mm in diameter. A gasket ring (h) holds the complete configuration in place in the airflow rack.

Figure 2. The airflow rack with the vials mounted. The fan provides a controlled airflow. The section in the middle (a) is used for measurements.

The weight change of a vial over time is used to determine the moisture flow. By measuring the moisture flow for different number of sample layers under identical ambient conditions, we can calculate the water vapor resistance, $Z_m$, for one layer of the sample material, as the surface vapor resistance is the same in all vials. The evaluation is done as described by Huldén, M. & Hansen, C.M. (1985). Figure 3, illustrates the used nomenclature.

Figure 3.

The nomenclature used in the evaluation of the method. $Z_t$ the total vapor resistance, $Z_x$ the vapor resistance of the method including the inner and exterior surface resistance found in the intercept with the y-axis, $Z_m$ the vapor resistance for one layer of the sample material and $n$ the number of sample layers. Measurement for a textile in an experiment using MgCl$_2$ as an absorbent (*).

3 TESTS OF THE METHOD

We have made a series of test measurements to evaluate the modified cup-method.

3.1 Materials

The crimp cap used is a tear-away type leaving a circular area exposed (14 mm in diameter).

In the test measurements two types of Teflon coated seals were used, silicone and butyl rubber. The silicone seal is 1 mm thick and the butyl rubber 2.8 mm. The seals were made by using a hollow punch to make a center hole (14 mm in diameter) in a regular 20 mm vial septa. The measurements show a significant difference in leakage between the two types of seals; the butyl rubber seals had a leakage of salt solution in 67% of the vials compared with 13% for the silicone.

Two types of vapor permeable membrane were tested – Durapore GVHP (polyvinylidene fluoride) and Fluoropore (PTFE on a LDPE backing) both with a pore size of 0.22 µm and manufactured by Millipore, Bedford, USA.

Using a hollow punch the test material was cut into circular pieces 20 mm in diameter. For each measurement, we used two or three sets of different layer set-ups (e.g. 1, 2 and 4 layers). Table 1 presents a description of the test materials used in the test series.
Table 1. Test materials used in test series.

<table>
<thead>
<tr>
<th>Type</th>
<th>Textile</th>
<th>Paper</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type</td>
<td>Cotton/linen (58/42%)</td>
<td>Quantitative Filter paper 0OH</td>
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<tr>
<td>“Josef”</td>
<td>Almedahl AB, Dalsjöfors, Sweden</td>
<td>Ash content 0.007%</td>
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<td></td>
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<td>Munktell Filter AB, Grycksbo, Sweden</td>
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<td>Surface mass</td>
<td>273</td>
<td>88</td>
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<tr>
<td>(g/m²)</td>
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<td></td>
</tr>
<tr>
<td>Thickness</td>
<td>0.451 ± 0.001</td>
<td>0.132 ± 0.005</td>
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<tr>
<td>(mm)</td>
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3.2 Climate conditions

We used two climates (+20 ± 0.5 °C, RH 35 ± 1%) and (+20 ± 0.7 °C, RH 58 ± 1.5%) in the climate room where the airflow rack was placed.

Saturated salt solutions provide controlled relative humidity inside the vials. During the test measurements, we used MgCl₂ (RH 33% at +20 °C), Mg(NO₃)₂ (RH 54.4% at +20 °C), NaCl (RH 75.5% at +20 °C) and distilled water (RH 100%). In some of the experiments, the RH was higher in the vial than in the climate room, causing the vials to loose weight, in other experiments the RH was lower in the vials so that the vial increased in weight. A correction of the relative humidity inside the vial in relation to the actual temperature was done according to Greenspan (1977).

An airflow rack holding the vials gave a controlled airflow over the exposed area of the test materials, an elevated airflow also decreases the exterior surface vapor resistance. (Newns, A.C., 1950)

3.3 Measurements

The moisture flow was determined from the weight changes of the vials. Typical intervals for the weighing were every 6–8 hours during the first 2 days, thereafter once a day. During the weighing, the vial is placed right side-up on a balance. The weighing of each vial takes approximately 30 s.

3.4 Results

Since the measurements were done under constant conditions, the weight change plotted as function of time should be linear. Small deviations from this is seen in the beginning of the measurements and referred to as initial disturbances. (Fig.4)

From the gravimetric measurements, the total vapor resistance, Zₓ of the vial set up can be calculated, see Fig 5. Using the evaluation method described by Huldén, M. & Hansen, C.M. (1985) the vapor resistance, Zᵐ, for each layer can be determined. The magnitude of vapor resistance of the method, Zₓ (including exterior surface resistance etc.) was estimated to 1–2 times the vapor resistance Zᵐ for each layer for the present measurements.
The total vapor resistance, $Z_t$, plotted as a function of numbers of paper layers. Data from measurements on paper letting a salt solution dry through the sample and the vapor permeable membrane:

- (+) $4.5 \text{ g NaCl(s)} + 4.5 \text{ ml NaCl(aq, sat)}$,
- (o) $9 \text{ ml NaCl(aq, sat)}$ together with a small amount of NaCl(s).

The value $Z_t$ is calculated from the linear part of the measurements. An outlier is removed and not used in the figure or the regression.

The effect of the increased airflow over the surface was studied with paper as test material. One group of the samples was prepared in a normal manner, see Fig 1, one group was made with a double layer of membrane in the normal position and one group had one membrane in the normal position plus one additional membrane covering the exposed area (in that way protecting the sample material from penetrating airflow).

The evaluation shows no significant difference between the three groups, Fig. 6.

The masking of the edges of the sample will introduce an error in the determination of the water vapor resistance of the material, $Z_m$. With an increasing thickness of the sample e.g. increasing number of layers, this error will also increase (Claesson et al., 1994). We have made calculations to estimate the magnitude of the error due to masking, see Figure 7.

The maximum error was found to be approximately 6% for the paper (8 layers with a thickness 0.132 mm) and 10% for the textile (4 layers with a thickness 0.451 mm). The error calculations were made for an isotropic material.

The air speed was measured with an anemometer with a probe that was placed in a free vial place of the airflow rack while the other vials remained in their places. The air speed varied over the cross section as seen in Figure 8, similar distributions were seen for the complete test measurements. The moisture variations of the samples show a small variation over the cross section, Figure 9.

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Figure 9. The moisture variation of the sample as a function of the position in the airflow rack, 3 (+) and 7 (o) layers of paper using MgCl₂ as an absorbent. The measurements were made for the time period of one week.

4 DISCUSSION

Advantages with the method:

• The method avoids the problems caused by uncertain interior surface resistances.

• The use of crimp caps makes it easier to fasten lightweight material and provide an edge masking.

• Standard laboratory equipment makes the method simple and inexpensive.

• It is a rapid method. Optimized measurements should probably not take more than two days.

Sources of error and special considerations for the method:

• Variations in the ambient climate. The relative humidity of the climate room varied by up to 1.5% over a measurement period. Such a change in RH results in up to 9% changes in the flow rates and is a factor that should be taken into account when this type of measurements are evaluated (this has not been done in the present measurements).

• Initial disturbances giving non-linear plot over the weight changes as a function of time. This is most probable caused by starting the measurements without conditioning the vial set-up properly. For experiments where the salt solution absorbs water and it could be beneficial to use a saturated salt solution with a high or even very high degree of undissolved salt this question needs careful considerations. This should be taken into account when optimizing the method of measurement.

• Disturbances caused by the measurement procedure were the vial during weighing is turn from upside-down to right side-up and back again. This is can be minimized by using a stand on the balance allowing the vial to weighed in an upside-down position.

• Fluctuations and disturbances in the air flow in the airflow rack.

• Air penetration of the sample material because of the elevated airflow in the airflow rack. Our test indicates that this is a minor source of error.

• The area determination needs special attention and especially the risk of large spread due to the variations from the opening of the tear-away crimp cap.

• A thick and stiffer seal, as the butyl rubber, seems to increase the risk for leakage and limits the number of layers of test materials that to be used.

• Salt crystals growing through the membrane. This is known to be a risk for measurements where salt solutions are being dried through a vapor permeable membranes. Special care should be taken to avoid this, mainly by performing the measurement over a short time (1-2 days).

• As an indicator for leakage due to capillary transport, a color marker of the salt solution (e.g. ordinary food color) can be used.

• Due to the small area exposed the more heterogeneous a material is the larger spread can be expected. Using an increased number of identical sample set-ups will help to obtain a more correct value for the water vapor resistance.

Future work:

• To optimize the method of measurement so that it will be rapid and reliable. Improve the evaluation method so that e.g. leaking vials are more easily detected. There is also a need to develop some aid to minimize the differences between different persons performing the preparations and measurements.

• Continue the validation of the method with an increased number of test materials.
Investigate the possible problem caused by pressure changes in the vials.

5 CONCLUSIONS

We have developed a modified cup-method for lightweight and highly permeable materials that has the potential to be reliable, simple and practical to use. The method deals with the disturbances and errors caused by the inner surface resistance in the air gap of the traditional cup method and provide an easy fastening of thin and lightweight sample. The improvement and validation of the method will continue.

REFERENCES


