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Determination of moisture properties and verification of step-responses for materials and material combinations.

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Report TVBH-7229 Lund 2005

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The paper presents an overview of the research work within the School of Civil Engineering at Lund Institute of Technology focusing on moisture properties. The focus is put on the JAR-method developed at the Division of Building Materials and the Department of Building Physics, but some the other methods used for determination of various moisture properties are also briefly presented.

The JAR-method is a sorption method based on standard glass jars and salt solutions and can be used for both determination of sorption isotherms and for monitoring the step-response of a material subjected to a step-change in relative humidity. The moisture variations in the sample are determined gravimetrically and the balance can be connected to a computer to enable continuous measurements. In the JAR-method the sample is kept inside the desired ambient climate for the complete experiment and therefore the disturbances are small. Result from several experimental series with various materials and material combinations are presented. The method has shown to be a robust and inexpensive method for a wide range of materials.

1 INTRODUCTION

One of the basic moisture properties of a material is the sorption isotherm— the equilibrium water gain as a function of the relative humidity (RH). To measure sorption isotherms is therefore a common practice in building material science as well as in many other fields of science e.g. food science, soil science and pharmaceutical science. There are a number of methods to do this and many of these are based on equilibration over saturated salt solutions (Svennberg, 2003). The use of automated sorption balances increase (Arlabosse et al., 2003) and even more sophisticated methods using NMR (Leisen et al., 2002) or sorption calorimetry (Wadsö & Markova, 2002) have come into use.

At the School of Civil Engineering at Lund Institute of Technology we use the basic simple methods based on saturated salt solutions as well as both automated sorption balances (DVS1000, Surface Measurements Systems, London, UK) and sorption calorimetry. The sorption calorimeter is developed by PhD Lars Wadsö, Division of Building Materials in collaboration with Thermometrics AB, Järfälla, Sweden. A simple but yet precise and practical method based on saturated salt solutions have been developed by PhD Lars Wadsö in collaboration with Kaisa Svennberg. (Wadsö, Svennberg and Dueck, 2004). The method called the JAR-method will be presented more in detail in section 2.

For determination of water vapor transport (WVT) we use a traditional cup method, a modification of the ASTM 96-00 standard (Hedenblad, 1996). An inverted cup method for thin and highly permeable materials such as textiles and paper has

been developed by Kaisa Svennberg, Department of Building Physics (Svennberg & Wadsö, 2003).

For determination of moisture profiles in mineral based materials a number of techniques have been developed and evaluated at the Division of Building Materials over the last years. It includes destructive methods as thermal imaging and non-destructive methods as x-ray detection and Magnetic Resonance Imaging (MRI). A destructive gravimetric method where the sample is split into well defined slices has been developed by Peter Johansson, Division of Building Materials and is soon to be published.

2 THE JAR-METHOD

2.1 Method

Saturated solutions of different salts give different constant relative humidities (RH) over their surfaces (Greenspan, 1977) (unsaturated solutions may also be used if one keeps control of the concentration of the solution (Clarke et al., 1985)).

When a sorption isotherm is to be measured with saturated salt solutions each sample is kept over a salt solution and weighed regularly. When the mass of the sample does not change anymore according to the equilibrium criterion being used that mass is noted as the equilibrium mass of the sample at that RH. Thereafter the sample may be taken to another RH where the procedure is repeated. The moisture content of a sample is usually based on the dry mass

of the sample and the sample is therefore dried before or after the measurement.

In simple methods relying on conditioning above saturated salt solutions that keep constant relative humidities (RH) and gravimetric methods to determine the moisture content in the sample the RH around the sample is disturbed during each weighing because the sample has to be taken out of the controlled RH for each measurement. A solution to this is to keep both the balance and the sample at the same RH, but this is impractical, as many different RH are needed to determine a complete sorption isotherm.

Gustafson & Hall (1974) and Zuritz et al. (1979) tried to address this problem with new methods. The JAR-method is an improvement of Gustafson & Hall and Zuritz ideas.

The JAR-method is based on standard glass jars (750 ml) and the desired relative humidity inside each jar is created by using well-defined salt solutions, usually saturated salt solutions. In the JAR-method the sample is suspended in a steel thread that runs through a rather large hole in the airtight lacquered steel lid. The hole in the lid is covered with a transparent polycarbonate washer with a minute hole in it only letting the steel thread to run through. The washer is free to move on the upper surface of the lid. On the steel thread a piece of silicone rubber tubing is mounted to both tighten the hole in the washer when not in weighing position and to keep the sample in correct vertical position. The details are shown in Figure 1.

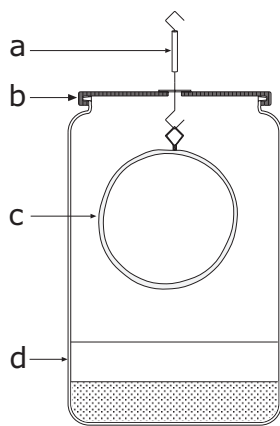


Figure 1. The JAR method. A ordinary glass jar for foods with screw lid (b) with saturated salt solutions (d) was used. The hole in the lid was covered with a thin plastic washer with a minute hole in it that only allows the wire to pass through. A small silicone rubber tube was placed over the wire (a) to seal the hole in the washer when measurements were not being done and to keep the sample in a vertical position. Determination of the moisture absorption-desorption was made with below balance weighing by connecting the wire (a) to the balance.

The weight change of the sample is determined gravimetrically through below balance weighing as shown in Figure 2. If the balance is connected to a computer, continuous measurements can be performed. This together with the easy manner to facilitate a step change in RH simply by moving the lid with the attached sample to a jar with the desired RH makes the JAR- method well suited for monitoring the step response for a material or a material combination.

Both the jars and the balance are kept in a climate room with a constant temperature to assure that the fluctuations in RH inside the jars are kept at minimum.

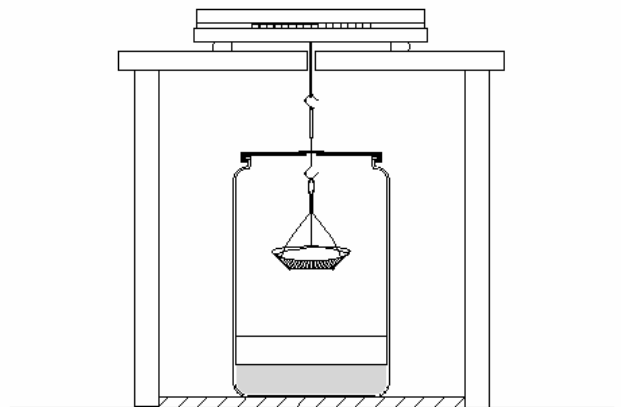


Figure 2. The measurement set-up with the sample in weighing position using below weighing. The sample hangs in the wire that goes through the hole in the lid. This hole is made quite tight by the washer floating on top of the lid (and the rubber tubing on the wire that tightens the hole when weighing is not being made). Note that the glass jar stands on a glass plate so it is easy to place it in the correct position for a weighing.

2.2 Sorption isotherm measurements

The JAR-method have been used to determine sorption isotherms for a broad variety of materials. The method was validated by using microcrystalline cellulose and bentonite clay. These studies are presented together with results from investigations on concrete and expanded clay.

Micro crystalline cellulose (MCC)

The JAR-method was validated for sorption isotherm determination by measuring the absorption isotherms for microcrystalline cellulose (MCC, Avicel PH-101, SERVA Electrophoresis, Heidelberg, Germany) as a reference material. MCC is a material with a uniform aggregate size and a very low influence of hysteresis. In the fields of food sci-

ence and pharmaceutical science MCC have been used often as a reference material. Therefore there are good literature values available.

The MCC measurements were done at 20 °C and six different RH (33, 43, 54, 75, 85 and 95%) using saturated solutions of six salts (MgCl₂, K₂CO₃, Mg(NO₃)₂, NaCl, KCl, KNO₃). Samples of 3.5 g MCC were used and placed in aluminum sample pans that were hung about 2 cm above the salt solution surfaces. Three samples in individual jars were used at each RH. To increase the sample mass a stainless steel weight of about 20 g was attached to each sample pan. This was done to minimize leakage. The dry mass of the MCC samples was determined by conditioning over drying agent (0.4 nm molecular sieves) in the method jars for ten days until equilibrium was reached.

The measurements were compared with literature values from Wolf et al. (1984) measured at 25 °C. The agreement was found to be satisfying, see Figure 3.

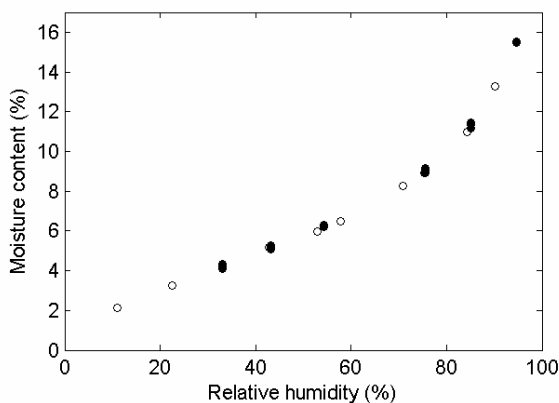


Figure 3. Measurements of the sorption isotherm for microcrystalline cellulose (MCC) made with the present method at 20 °C (filled circles) compared to literature values from Wolf et al. (1984) measured at 25 °C (open circles). Moisture contents are calculated on a dry basis.

Bentonite clay

Bentonite clay (commercial bentonite clay with the quality symbol MX-80 from American Colloid Co., which is a blend of natural Wyoming bentonite horizons) was also used as a reference material in the validation of the JAR-method for sorption isotherm determinations. The bentonite clay is slow sorbing natural material and makes it more difficult to determine the sorption isotherm for bentonite clay than for MCC.

The bentonite clay measurements were done at 6 different RH (11, 33, 59, 75, 85 and 93%) using saturated salt solutions of five salts (LiCl, MgCl₂, NaBr, NaCl, KCl) plus one aqueous unsaturated NaCl solution for 93% RH. Only one sample was used at each RH. The unsaturated aqueous solution was a two molal solution of NaCl, the 2 molal NaCl solution at 20 °C gives RH = 93% (Clarke et al.,

1985). The jar with the unsaturated aqueous solution was exchanged once a week to minimize the effect of evaporation and leakage. Each solution was weighed before and after being used and the mass loss was found to be less than 2% of the total weight of the solution.

Samples of 10 g, with an initial moisture content of 0.4%, were placed a couple of cm above the saturated or unsaturated aqueous solutions in the same type of sample pans as used for the MCC. The initial state of the bentonite was a result of conditioning over drying agents for 28 days. Stainless steel weights, as mentioned above, were also used here. After 82 days the tests were finished. The samples were dried at 105 °C for 24 hours and then placed over molecular sieves (0.4 nm) in the glass jars and weighed. The weight after oven drying was regarded as the dry mass.

The bentonite clay measurements were compared with literature values as well as measurements performed in the automated sorption balance (DVS 1000, SMS, London, UK). The agreement was good and the deviation seen in the upper part of the sorption isotherm can most probably be explained by the slow attainment of equilibrium for bentonite clay, see Figure 4.

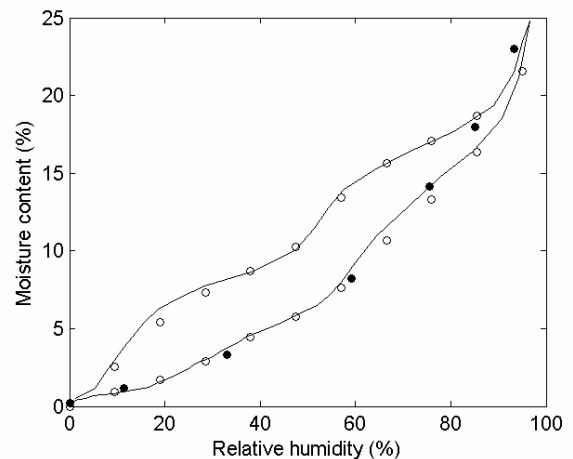


Figure 4. Measurements of the sorption isotherm for bentonite clay made with the present method at 20 °C (filled circles) compared to sorption balance measurements at 25 °C (open circles) and literature values from Kraehenbuehl et al. (1987) and Kahr et al. (1986) at 20 °C (solid line). Moisture contents are calculated on a dry basis.

Concrete

Sorption isotherms were determined for two concretes having, w/c-ratios of 0.60 and 0.40 both of them without air entrainment. Eight samples (7.2 cm³) were placed over saturated salt solutions contained in jars. The relative humidities (RH) were 11% (LiCl), 33% (MgCl₂), 59% (NaBr), 75%

(NaCl), 85% (KCl), 91% (BaCl₂), 94% (KNO₂) and 97% (K₂SO₄). The samples were placed in aluminum sample pans of the same type as used in the MCC and bentonite experiments.. The dry weights were determined after the samples had been dried at +105°C, the jars then containing 0.4 nm molecular sieves.

In another part of the project the influence of drying was investigated for samples that were tested in the differential scanning calorimeter (frost resistance tests), the sorption isotherms were determined for samples treated in the same way. A sawn prism (20x20x170 mm) was used in constructing each of the isotherms, the treatment for each isotherms being as follows:

Absorption (B)

A five-year-old sample was sawn into 8 pieces (~18x20x20 mm). Each piece was weighed in both air and water. The pieces were dried at +105°C for six days.

Each piece was crushed separately in a plastic bag and placed in a jar over a saturated salt solution to obtain the RH that was desired. Prior to being crushed, the pieces were cooled off in a box containing a drying agent.

When the final weight was determined, the samples then being dried at +105°C, cooled off and weighed again.

Absorption (C)

Using the same procedures as in (B), except for drying was changed to: the pieces were placed in an airtight box containing a drying agent (silica gel) for a month, and no cooling being necessary..

Desorption (A)

Using the same procedures as in (B), with the drying being excluded and no cooling being necessary.

Desorption (D)

The same procedures were followed as in (B), with the except the drying being changed to the pieces being placed in an airtight box containing a drying agent (silica gel) for a month, thereafter the pieces were placed in a box containing deionised water, where they absorbed water for a month, no cooling being necessary.

Desorption (E)

The procedures here were the same as in (B), but after the drying the pieces were placed in a box of deionised water, where they absorbed water for a month, no cooling being necessary.

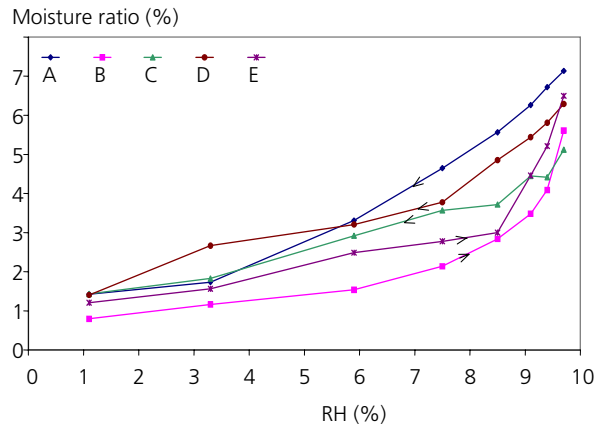


Figure 5. The influence of drying conditions on the sorption isotherms for concrete with a w/c-ratio 0.60 and a natural air content. By courtesy of Katja Fridh, LTH.

Expanded clay

The isotherm was determined for expanded clay LWA 4-10 mm at 20 °C using saturated salt solutions (11% (LiCl), 33% (MgCl₂), 59% (NaBr), 75% (NaCl), 85% (KCl), 91% (BaCl₂), 94% (KNO₂) and 97% (K₂SO₄) and 97%). The expanded clay aggregates were placed in baskets made of stainless steel grid allowing maximum moisture exchange between the sample and the ambient air.

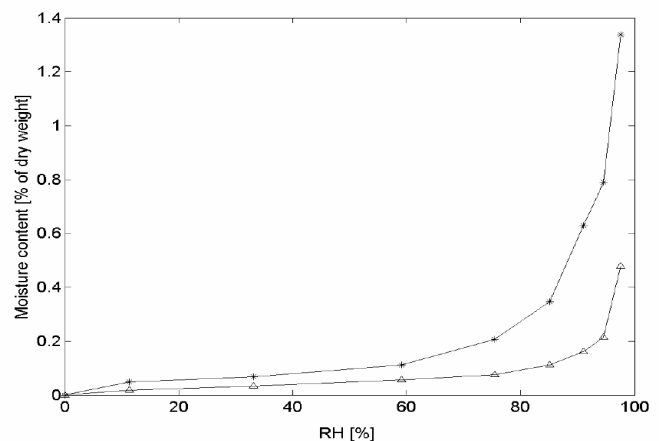


Figure 6. Sorption isotherm of expanded clay LWA 4-10 mm. By courtesy of Anders Anderberg, Optiroc & LTH.

2.3 Step-response measurements

The Jar-method can also be used to monitor the step-response for materials subjected to a step change in RH. The step change is easily achieved by simply moving the lid with the attached sample from a jar the original RH to a jar with the new RH. This can be quickly done in a well controlled manner.

The transient application of the method is still under validation and the validation procedures are briefly described below. Two series of experiment are presented: plasterboard with a dispersion paint system and wood with surface coating. An ongoing study for textile composite materials (fabric and padding) is used as example of the development of spe-

cific sample holders for different materials and purposes.

Plasterboard with a dispersion paint system

Plasterboard for indoor use (density 720 kg/m³, thickness 12,5 mm) lined with paper on both sides was used. The surface coating system was of dispersion paint type. The system consisted of: an oil emulsion primer, a water-soluble adhesive, one layer of glass fibre fabric and two coats of acrylate dispersion paint. The first coat of paint had a matt sheen and the topcoat a semi-matt sheen.

10 samples were prepared from each of the six stages in the surface coating system resulting in a total of 60 samples. The samples were sawed out of the plasterboard with hole saw. The raw paper edges were trimmed and a thin stainless steel wire used for hanging was applied.

A 50/50 mix of bees wax/paraffin wax was used for sealing. A thin plastic sheet was cut and centred on the open side to mask it from the wax. Thereafter, the backside and the edges were sealed with the wax. The seal extended approximately 1 mm from the outside edge over the open side.

After the samples were sealed they were conditioned at RH 33% respectively 54% for at least 6 weeks. The conditioning took place in the same type of equipment as used for the measurements see Fig. 1 above.

Five samples from each sample group were subjected to a step change (RH 33-54 %) resulting in a moistening (absorption) and the other five to a drying step change (desorption) in the humidity interval. The temperature was held constant (+ 20°C) throughout the whole experiment (Svennberg & Harderup, 2002).

The weight change was determined gravimetrically and the measured weight differences were used for calculating the moisture absorbed and desorbed respectively. The absolute values of these moisture variations were plotted as shown in Fig. 7.

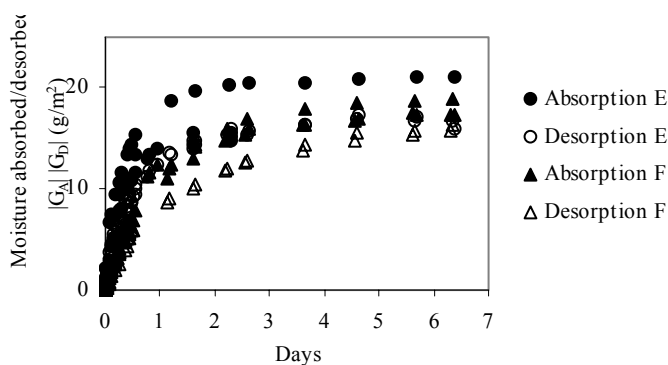


Figure 7. Absolute values of the moisture absorbed / desorbed for samples with 1 layer of paint (E) respectively 2 layers of paint (F).

When the samples have reached equilibrium the moisture capacity, ξ , can be calculated, see Figure 8. The most conspicuous result from this study is that the moisture capacity, ξ , increases with the increasing number of stages in the surface coating system (Figure 8.). The assumption was that the surface coating had a negligible moisture capacity. Our study shows that for the investigated system with a glass fiber fabric backing the dispersion paint there is probably a significant moisture capacity. A probable cause is the asperity and porosity of this type of surface coating.

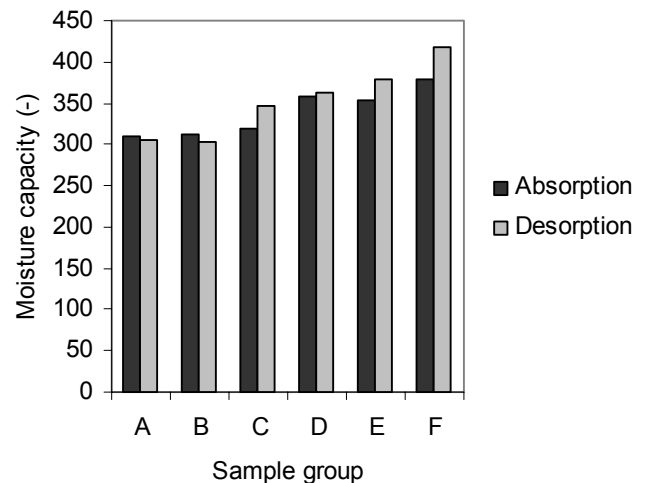


Figure 8. Moisture capacities, ξ , mean values for each sample group (A-F). It is worth noting that the moisture capacity, ξ , increases with an increasing number of layers in the paint system. The magnitude of the increase was not anticipated.

Wood with surface coating

On cylindrical (~60 mm diameter and ~5 mm thickness) test specimens of different woods/wooden material, absorption and desorption was studied for step-changes in RH. The moisture capacity for each step change in relative humidity was calculated for the different woods/wooden material. The test specimens have their fibres oriented in the plane of the exposed surface.

They were untreated, stained or treated with beeswax. The investigation covers birch, beech, pinewood, oak, spruce and mahogany, glulam as plywood and stave glued birch moreover veneer of birch, beech and oak on particle board.

Massive birch exhibits the highest absorption of moisture while massive spruce and beech have the lowest hysteresis effect of the examined woods/wooden material. The veneered samples have higher absorption of moisture than corresponding massive woods. After each change to a new relative humidity the untreated test specimen exhibits faster moisture absorption than those with treated surfaces. This was expected, as the surface treatments shall give an increased diffusion resistance. The differences in

moisture capacities between test specimens of the same woods/wooden material but with different surface treatments are small compared to the differences between woods/wooden materials. This is according to the expectations as the surface treatment only marginally should influence the moisture capacity.

A comparison with numerical calculation model based on Luikov's analytical model was performed and showed good correlation, see example in Figure 9 (Freij-Andersson & Heimsäter, 2000).

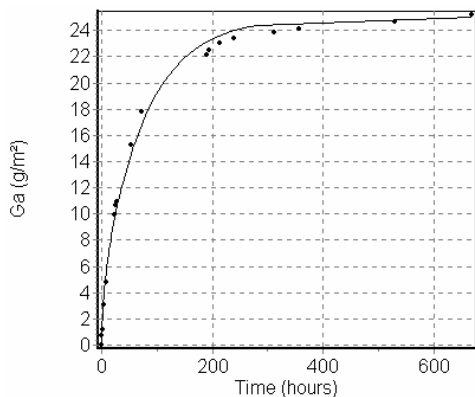


Figure 9. Comparison between measured and calculated moisture uptake as a response to a change in RH for birch wood without surface coating. (Freij-Andersson & Heimsäter, 2000)

Wool fabric in combination with foam plastic

Since the JAR-method have been used for so many different materials, several different solutions to the sample holder dilemma have emerged.

In Figure 10 a and 10b a composite textile material consisting of a wool fabric (A) and foam plastic (D). For optimal measurements, one-dimensional moisture flow is desired. This is accomplished through sealing the sample on the sides and in the back (B).

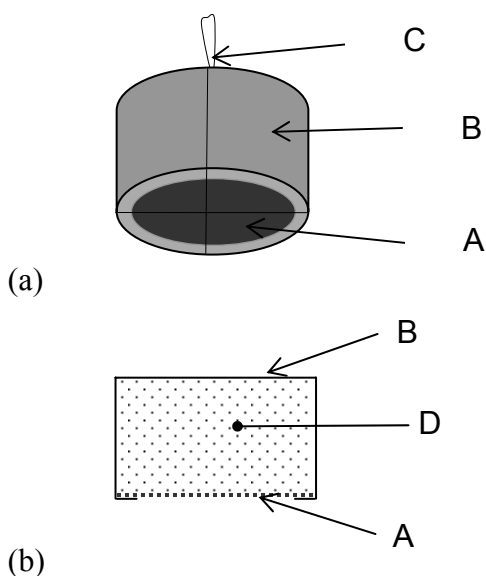


Figure 10 a & b. Sample holder (Fig 10 a) for textile composite materials –fabric (A) and padding (foam plastic) (D). The sides and the back of the sample is covered with sturdy aluminum foil (B) of the type used in disposable cake pans. A

stainless steel thread (C) is mounted on the sample so it can be hung on the hook in the jar. Figure 10 b shows the sample holder in section.

Validation experiments with calcium silicate as reference material

The JAR-method is to be validated carefully for step-response measurement and the validation process includes step-response measurements where calcium silicate is used as a reference material.

A modified JAR-method with a small membrane pump installed on the lid will also be tested. The membrane pump will provide a more controlled air flow inside the jar; hopefully this will reduce the influence of the surface resistance due to a stagnant air layer around the sample. The position and direction of the sample will also be carefully investigated.

3 SORPTION ISOTHERMS FOR TEXTILE MATERIALS

The sorption isotherms for a wide variety of textile materials have been determined using an automated sorption balance (DVS 1000, Surface Measurements Systems, London, UK). The samples have been relatively small (circular with a diameter of 20 mm for the fabrics and cubes with a side of 7-8 mm for the other materials). The samples were initially dried in the instrument with dry nitrogen. Both absorption and desorption was determined in steps of 9.5 % RH.

The results are being analyzed and only preliminary data are available so far. Figure 11 show the sorption isotherms for two textile furnishing materials. The wool fabric shows a more hysteresis than the cotton fabric. Something that may reduce the moisture buffering capacity in practical applications.

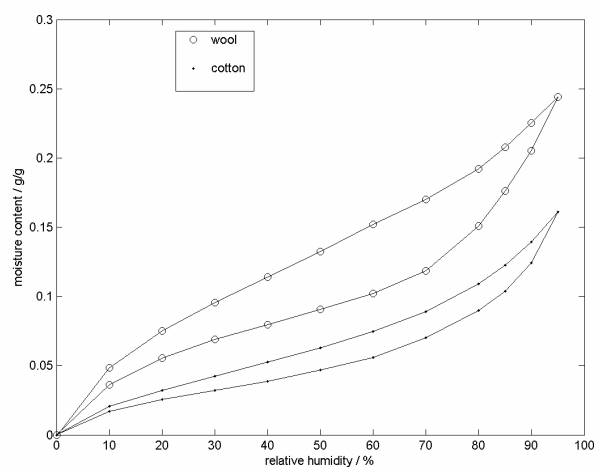


Figure 11. Sorption isotherms for a wool fabric (used on a chair) and a cotton fabric (used for curtains). Both sorption isotherms are determined in an automated sorption balance (DVS 1000, SMS, London, UK)

4 CONCLUDING REMARKS

The area of determination of moisture properties has a long tradition in the School of Civil Engineering at Lund Institute of Technology and range from traditional building materials as wood and concrete to furnishing materials as textiles and paint. We have several climatic chambers where the temperature and RH can be controlled in a precise way.

The JAR-method developed at Lund Institute of Technology is a simple, inexpensive method and yet a precise and practical method for determination of sorption isotherms and for monitoring and verification of step-responses for a wide range of materials.

The automated sorption balance and the sorption calorimetry are also used successfully and can aside from the traditional materials also be used for thin and highly permeable materials.

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