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Wadsö, Lars

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LUND UNIVERSITY

PO Box 117
221 00 Lund
+46 46-222 00 00



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Division of Building Materials

A METHOD TO MEASURE SORPTION ISOTHERMS

Lars Wadsö

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A method to measure sorption isotherms

Lars Wadsö
Building Materials
Lund University
Box 118
S-221 00 Lund
Sweden

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1 Introduction

Sorption isotherms, i.e. the equilibrium moisture content of a material as a function of the relative humidity, are important parameters in building materials research. Data of sorption isotherms is often produced by placing material samples in closed containers with saturated salt solutions and weighing the samples until they show a stable weight, i.e. are in equilibrium with the surrounding relative humidity. The moisture content (m) is then found as:

$$m = \frac{M - M_0}{M_0}$$

Here M and M_0 are the weights of the sample at equilibrium and at dry conditions, respectively. Drying is in most cases made at 105°C until the weight is stable.

Saturated solutions keep the relative humidity constant in the closed air volume above them. Different salts give different relative humidities so it is possible to measure the equilibrium moisture content at different relative humidities by using different salts. Greenspan (1977) have compiled relative humidity data for a large number of salts at different temperatures.

The above described common procedure is often acceptable, but has some drawbacks as the samples regularly has to be taken out of their relative humidity containers for weighing:

- It will be difficult to weigh samples that rapidly can adsorb or desorb moisture, as the relative humidity of the room is often quite different from that of the saturated salts,
- The relative humidity of the containers will be disturbed for a long time after each weighing. The salt solutions are often not very quick in re-creating the proper relative humidity.

This paper concerns a simple and inexpensive, but still accurate method of measuring sorption isotherms.

2 Method

The measurement takes place in a glass jar with a vapour tight lid as shown in Fig. 1. In our experiments we used ordinary household glass jars with metal lids, but any jar with a removable lid can be used. To the lid a metal plate is fastened (e.g. with a heat resistant adhesive). A 4 mm hole is drilled through the sheet and the lid. The sample is then hanged in a wire through the hole in the lid. A light transparent washer (outer diameter 10 mm, inner diameter <1 mm in excess of wire diameter) tightens the 4 mm hole, and a stop is fixed on the wire, so that the sample does not fall onto the bottom of the jar. The stop also acts as an extra barrier to vapour flow when the sample is not being weighed.

During a measurement the bottom of the jar is covered with a saturated salt solution and the lid with the sample is drawn tight. Measurements of weight can now take place without the sample leaving the container as shown in Fig. 1. The balance is raised so that the weighing can take place by hooking the wire with the sample to the balance from below. During each weighing it is essential that the wire does not touch the lid or the washer. It is quite easy to see when this is not the case as the wire and the sample then makes a small pendulum movement. We have used a mg-balance and did not find that the described arrangement decreased the precision of the measurements when the wire was free, e.g. not touching the lid or the washer.

When regular weighings show that the sample weights are stable the samples should be dried to get the dry weight. The lids with the samples are then transferred to empty glass jars. This time the lids are not tightened, but put loose on the top of the jars leaving a slit for the vapour to escape from the jar. The samples and the jars are then dried in a laboratory oven. When the samples are considered dry the lids are screwed tight on the jars inside the oven. The jars are then taken out of the oven to cool. When they are at room temperature again, the samples are weighed a last time to get their dry weight.

A drying agent (e.g. silica gel) may be placed in the glass jars to guarantee that the sample is dried and stays dry during the cooling. Due to the contraction of the air in the jar during cooling some room air will be sucked into the glass jar. We have found that it is not possible to make accurate weighings unless the jar and the sample is cooled to near room temperature, but this problem may probably be solved by protecting the balance from the heat radiation and convection from the jar.

As it is easy to fit a number of jars into a laboratory oven it is possible to make measurements at other temperatures than room temperature. Three problems with measurements at other temperatures than room temperature should be mentioned:

1. The temperature of the jar and the sample must not change during the weighings,
2. As discussed above, the warm jar must not disturb the weighings,
3. If the jars are kept in a laboratory oven it is essential to check that the temperature is constant. A fluctuating temperature will give a fluctuating relative humidity.

As one does not only weigh the sample, but also the wire and the stop, the weights of these must be recorded and subtracted from the measured weights to get the true weights of the samples.

3 Results

We have used the above described jars to measure the sorption isotherm of pure cotton wool. This material consists of very thin cellulose fibers that

quickly reacts on changes in relative humidity. For such a material it is impossible to make good measurements if the relative humidity containers are opened during the measurements, or the samples taken out of into the room air for weighing. Figure 2 shows the results which compare well with values from the literature.

4 Conclusions

A new method for the determination of sorption isotherms has been developed. It is simple and inexpensive at the same time as it is accurate. The relative humidity around the samples is not disturbed during the weighings. Therefore, even very thin and small samples can be weighed with high precision. The method would seem to be a good method to use at laboratories that do not have the possibility of investing in expensive equipment.

5 References

- Greenspan, L., J. Res. National Bureau of Standards 81A, 89-95 1977
- Urquhart, A.R. and Williams, A.M. according to p. 157 in Stamm, A.J., "Wood and Cellulose Science", The Ronald Press Comp., New York 1964

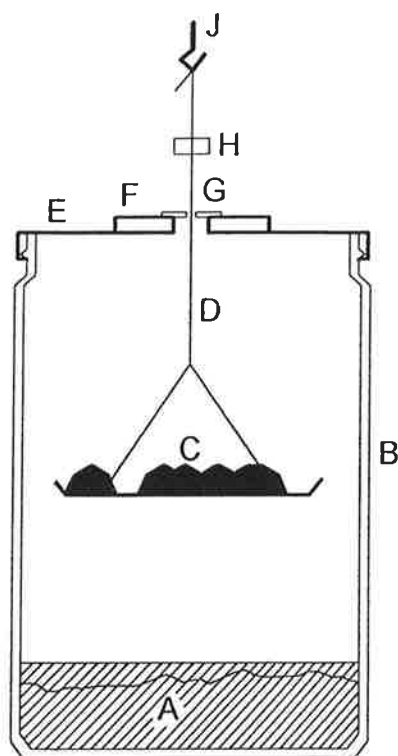


Figure 1. Drawing of the jar used in the measurements of sorption isotherms. A: saturated salt solution; B: glass jar; C: material to be investigated; D: wire; E: lid; F: metal plate; G: transparent washer; H: stop; J: hook on balance for under-weighing.

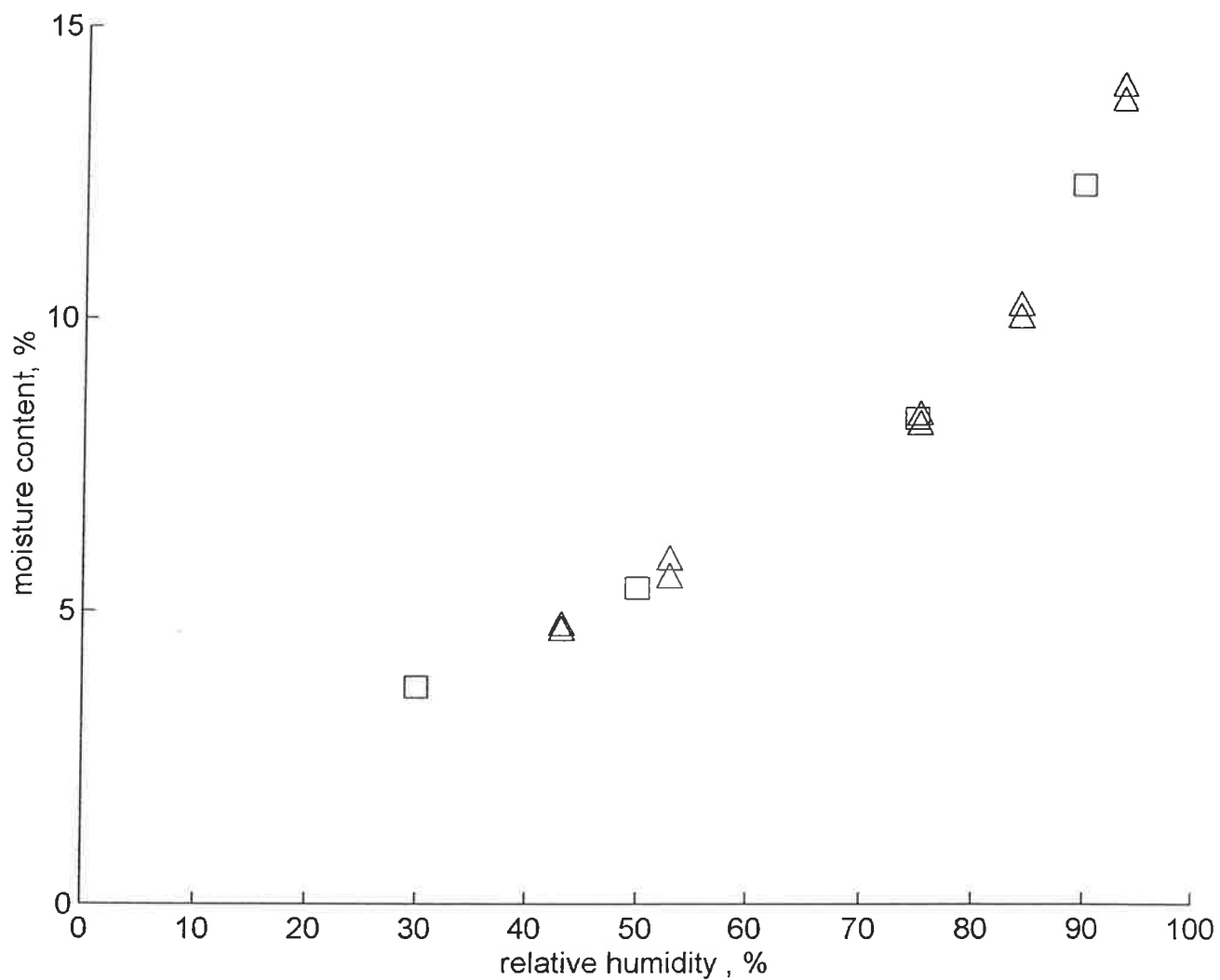


Figure 2. The result from a measurement of the sorption isotherm of cotton wool by the present method (triangles) and by Urquhart and Williams (squares). All measurements were made at 25°C.