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# A method for determination of absorption isotherms at high relative humidity levels: measurements on lime-silica brick and Norway spruce (*Picea abies* (L.) Karst.)

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## Abstract

The relationship between the relative humidity and the moisture content of a material for relative humidity levels close to 100% can be studied using the pressure plate technique. However, this method is designed for desorption experiments and modifications are necessary in order to perform absorption experiments. This paper presents a method to condition samples to a uniform moisture content by absorption with the pressure plate technique. The specimens were suspended in magnetic clamps until the ceramic plate had equilibrated with the applied pressure and modifications were made so that water could circulate through the pressure plate cell.

*Keywords:* (3-5 keywords) sorption isotherm, suction curve, method development, pressure plate, hysteresis

## 1. Introduction

Many degradation mechanisms are linked to high moisture contents; for example, high moisture contents in combination with low temperatures can cause frost damage in porous materials and wood is degraded by rot fungi if the moisture and temperature conditions are favourable. In order to predict how fast the degradation will occur (see e.g. Isaksson et al. <sup>[1]</sup>), predictions of which moisture contents that are reached in a structure are necessary. For rain exposed structures, such predictions require moisture transport models as well as material data in the high moisture range. In the field of wood drying, moisture transport models can be used to optimize drying schedules<sup>[2]</sup> and various drying models have been presented in literature e.g. <sup>[3-8]</sup>. Hysteresis, i.e. that the moisture content during desorption is higher than during absorption at the same relative humidity (RH) level, is seldom included in wood drying models<sup>[9]</sup>. However, Salin <sup>[9]</sup> emphasized the importance of inclusion of hysteresis and the significance of inclusion of hysteresis was also illustrated by Cloutier and Fortin <sup>[10]</sup> and Salin <sup>[11]</sup>.

Sorption isotherms in the hygroscopic range are commonly determined by equilibrating samples over saturated salt solutions<sup>[12]</sup> or by using a sorption balance<sup>[13]</sup>. However, freshly felled wood and building materials that are used in rain exposed structures reach moisture contents above this range and it is therefore of interest to study this relationship also for high RH levels. For wood, this range is of particular importance, since the growth of rot fungi generally occurs when the moisture content is above the fibre saturation point<sup>[14]</sup>, the growth of rot fungi is thus limited to the high moisture range.

It is theoretically possible to condition samples above salt solutions also for relative humidity levels close to 100% but it requires extreme temperature stability in order to avoid condensation and changes in RH. Another method, the pressure plate method, is therefore commonly used in the high moisture range. Here, specimens are placed on a ceramic plate in

an extractor where a pressure up to 15 bar is applied. For higher pressures, up to 100 bar, a pressure membrane extractor with a cellulose membrane is used. The pressure to which the samples are exposed presses out the water in the pores in which the capillary forces are unable to hold the water, i.e., water in which the capillary pressure is lower than the applied pressure. These pressures correspond to certain moisture states that can be given as for example RH. The RH that corresponds to the applied pressure in the extractor is given by:

$$\ln(\phi) = -\frac{\Delta PM_w}{RT\rho_w} \quad (1)$$

Here,  $\phi$  is the relative humidity,  $M_w$  is the molar weight of water (0.018 kg/mol),  $R$  is the gas constant (8.314 J/(mol·K)),  $T$  is the temperature (K),  $\rho_w$  is the density of water (kg/m<sup>3</sup>) and  $\Delta P$  is the pressure relative to the atmosphere pressure (Pa). For example, 1.5 MPa (15 bar) corresponds to 98.90% RH and 0.9 MPa (9 bar) corresponds to 99.34% RH. With the pressure plate method it is thus possible to equilibrate samples to high moisture states and determine the sorption isotherm in the superhygroscopic range. The method was originally used for soils [15], but it has also been widely used for several materials such as wood [16-18, 10, 19-26], lime and cement mortar [27-29], sand stone [30], lime-silica brick [28] and brick [27, 28].

In literature, most sorption isotherms for high RH levels are determined as desorption isotherms. This is probably due to the method adjustments that are necessary for absorption experiments. One of the challenges with absorption experiments is to ensure that all parts of the specimen reach equilibrium state from absorption. If a dry specimen is put in contact with the water saturated ceramic plate, there is a risk that a higher moisture content than the equilibrium moisture content is reached in the lower parts of the specimen. If that is the case, the lower parts of the specimen reaches equilibrium state from desorption instead of absorption. Consequently, the moisture content of the lower part of the specimen becomes higher than the upper part due to hysteresis effects. It is therefore important that the specimen does not come in contact with the ceramic plate until the plate has equilibrated with the applied pressure. In addition, the ceramic plate needs to be supplied with water which requires modifications of the pressure plate cell. The latter was considered by Cloutier and Fortin [10], Fortin [17] and Leong et al. [31] who rebuilt the pressure plate cell and attached a plastic base below the ceramic plate in order to enable water flow. In addition, Fortin [17] preconditioned the ceramic plate to the desired pressure before the specimens were placed in the extractor. However, this requires that the pressure is released and that the extractor is opened in order to place the specimens in the extractor. This means that there will be no pressure gradient over the ceramic plate. If there is water below the ceramic plate the RH will be 100% and the lower parts of the specimens might therefore still reach a higher moisture content than the equilibrium moisture content.

Penner [16] measured absorption isotherms by first performing a desorption experiment; wet specimens were placed in the extractor and when they had equilibrated, the pressure was lowered and the specimens started to absorb water. However, during the desorption experiment it is not possible to dry the specimens lower than the RH corresponding to the maximum pressure of the pressure plate/pressure membrane cell which is 15 bar (approx. 98.9% RH) and 100 bar (approx. 92.3% RH) for the pressure plate and pressure membrane cell respectively. Since the specimens start to absorb water from a RH close to the desired equilibrium RH there is a substantial risk that the specimens are conditioned to a scanning curve rather than the absorption isotherm. In addition, since a desorption experiment has to be performed before the absorption experiment can start the experimental time is doubled.

This paper presents a method to determine absorption isotherms using the pressure plate technique. To ensure that the whole specimen reaches equilibrium state by absorption, the

specimens were suspended in magnetic clamps and were let down when the ceramic plate had reached equilibrium with the applied pressure. The pressure plate cell was modified to enable inflow and outflow of water and the water was circulated by peristaltic pumps to ensure a continuous water flow. The measurements were performed on lime silica brick and Norway spruce (*Picea abies* (L.) Karst.) sapwood. The results for the lime silica brick were compared to desorption pressure plate measurements on the same material from a previous study<sup>[28]</sup>. In addition, in order to compare the data obtained for the wood specimens, pressure plate desorption measurements were performed and sorption isotherms were determined in the hygroscopic range using a sorption balance.

## 2. Pressure plate absorption method

### 2.1 Experimental set-up

The experimental set-up used in the absorption experiment is shown in Fig. 1. The design of the pressure plate cell was slightly different compared to those used in the desorption experiments. Here, the pressure plate cell had one outlet stem and one inlet stem, connected to an outflow and an inflow tube so that water could be circulated between the ceramic plate and the neoprene diaphragm. To facilitate the water circulation, the internal screen was modified; the grids between the ceramic plate and the neoprene diaphragm were displaced in relation to each other to make sure that water could pass between the inlet and the outlet. The inlet tube was connected to two peristaltic pumps which provided the ceramic plate with deionised water from a flask. The outlet tube was connected to a T-bore stopcock so that the outflow could be led to either burette 1 or burette 2. Burette 1 was used in the initial state until the ceramic plate had equilibrated to the applied pressure (see below). However, when the absorption experiment started, the water was led to burette 2 which was connected to the flask with deionised water. The inlet of burette 2 was placed on the side at the upper part of the burette so that air bubbles were separated from the outlet water. The air was let out through a hypodermic needle inserted in a rubber stopper placed on top of the burette. Both burettes had a resolution of 0.05 ml.

### 2.2 Experimental procedure

The modified pressure plate cell (maximum pressure 15 bar) was vacuum saturated with deionized water and was then placed in the pressure plate extractor (Model 1600 5 Bar Pressure Plate Extractor, Soilmoisture Equipment Corp., Santa Barbara, CA, USA). To prevent absorption before the pressure was applied the specimens were hanged in magnetic clamps (25 mm HT-D-25-F-12VDC, Kuhnke, Malente, Germany) above the ceramic plate. The extractor was then closed and a pressure was applied using nitrogen gas. The outlet water was led to burette 1. When the ceramic plate had reached equilibrium state, i.e. when no water outflow was registered in burette 1, the outlet water was instead led to burette 2 and the peristaltic pumps were started. When the water from the flask had reached the ceramic plate, the power to the magnetic clamps was turned off and the specimens were dropped onto the ceramic plate. The specimens then started to absorb water and the water level in burette 2 decreased. Equilibrium state was reached when there was an unchanged linear decrease in the water level; since water evaporates through the tubing, the water level in the burette never becomes constant. Therefore, to know which leakage to expect, the leakage through the tubing was determined, see section 3.1.2. When the water level in the burette decreased linearly similar to the leakage in the control experiment setup (see section 2.3) the experiment was ended.

### 2.3 Determination of leakage through the tubing

The leakage of water vapour through the tubing needs to be known in order to define the equilibrium state; a leakage through the tubing lowers the water level in the burette even though the specimens have reached equilibrium state. An additional experiment was set up to determine the leakage through the tubing. Three different types of tubing were used in the experimental setup: tygon tubing (R3603), PVC-tubing and silicon tubing. Two peristaltic pumps were connected to a burette and a flask with deionized water similarly as in the pressure plate set up but without the pressure plate extractor and without burette 1. The same amount and types of tubing were used. Water was then circulated and the water level in the burette was registered regularly. The experiment was performed in the same room as the pressure plate experiment, i.e. the RH and temperature in the room was identical.

### 3. Test measurements

#### 3.1 Material

##### 3.1.1 Wood

Specimens with dimensions  $40 \times 40 \times 5 \text{ mm}^3$  (radially  $\times$  tangentially  $\times$  longitudinally) were cut from fast grown mature sapwood of Norway spruce (*Picea abies* (L.) Karst.). The growth ring width was in the range of 1-4 mm/2-5.5 mm (latewood/earlywood) and the dry density was about  $340 \text{ kg/m}^3$ ; the material is further described by Fredriksson et al. <sup>[32]</sup>. All specimens were cut from one board.

The specimens were dried at  $30 \text{ }^\circ\text{C}$  (approx. 20% RH) before the experiment started. An anti-corrosion treated steel screw (Essve outdoor mounting screw CorrSeal,  $4.5 \times 13$ ) was fastened in the middle of each specimen so that the specimens could be attached to the magnetic clamps. This screw was chosen since it was both magnetic and would withstand corrosion despite of the high RH in the pressure plate extractor.

##### 3.1.2 Lime silica brick

Four specimens of lime silica brick was sawn to dimensions  $40 \times 40 \times 5 \text{ mm}^3$ . The lime-silica brick specimens were dried at  $50 \text{ }^\circ\text{C}$  before the experiment started. Since it was not possible to fasten the anti-corrosion treated screw directly in the specimen as for the wood specimens, a stainless steel wire (thickness 0.5 mm) was winded around each specimen. In order to be able to suspend the specimen in the magnetic clamps an anti-corrosion treated steel screw was attached to another stainless steel wire which in turn was attached to the wire that was winded around the specimens.

The lime silica brick that were used in the present measurements was cut from the same material that was used by Johansson <sup>[28]</sup>. This material was chosen since the complete desorption isotherm was available and the new absorption data could thus be compared to the desorption data in Johansson <sup>[28]</sup>. To ensure that the specimens in the present study were fully comparable with the material in Johansson <sup>[28]</sup>, 10 extra specimens ( $40 \times 40 \times 5 \text{ mm}^3$ ) were cut and material properties in terms of density and porosity were determined as follows. Dried specimens ( $50 \text{ }^\circ\text{C}$ ) were vacuum saturated and the mass of each vacuum saturated specimen was determined both in air and in water. The volume  $V$  ( $\text{m}^3$ ) of the specimens could thus be determined by:

$$V = \frac{m_{\text{vac, in air}} - m_{\text{vac, in water}}}{\rho_w} \quad (2)$$

where  $m_{\text{vac, in air}}$  (kg) is the mass of the vacuum saturated specimens,  $m_{\text{vac, in water}}$  (kg) is the mass of the vacuum saturated specimens under water and  $\rho_w$  ( $\text{kg/m}^3$ ) is the density of water. The density was then determined as dry mass ( $50 \text{ }^\circ\text{C}$ )  $m_{\text{dry}}$  (kg) divided by the volume  $V$  ( $\text{m}^3$ ) and the porosity was determined by:

$$P = \frac{m_{vac, in air} - m_{dry}}{\rho_w V} \quad (3)$$

The porosity and density of the specimens in the present study as well as the material properties measured by Johansson <sup>[28]</sup> are shown in Table 1. As seen in Table 1 the results were similar and the absorption data obtained in the present study should therefore be fully comparable to the desorption data in Johansson <sup>[28]</sup>.

### 3.2 Experimental procedure

The experiments were performed as described in section 2. The pressure levels used as well as the number of specimens used on each pressure level is shown in Table 2. When equilibrium state was reached, i.e. when the leakage in the control experiment was the same as the leakage in the pressure plate absorption experiment, the pressure was released, the screws/suspension devices were removed from the specimens, and the mass of each specimen was determined. Finally, all specimens were dried at 105°C (wood specimens) or 50°C (lime-silica brick specimens), the dry mass was determined and the moisture content was calculated by:

$$u = \frac{m - m_{dry}}{m_{dry}} \quad (4)$$

Here,  $m$  (g) is the equilibrium mass and  $m_{dry}$  (g) is the dry mass of the specimen. The temperature at two positions in the pressure plate extractor as well as the temperature and the RH in the room was logged during the experiments. The temperature in the room was 21.5 ±0.5 °C for the measurement on wood and 21.5±1.5 °C for the measurements on lime-silica brick (the equipment was moved between these measurements due to renovation).

## 4. Comparative measurements

### 4.1 Pressure plate desorption measurements

Since desorption data in the high moisture range was not available for the wood material that was used in the present study, pressure plate desorption experiments was set up in order to enable comparison with the absorption moisture contents. The experimental set-up was the same as described by Fredriksson et al. <sup>[33]</sup>. Wood specimens with dimensions as described in 3.1.1 were used. Both the specimens and the ceramic plate were vacuum saturated as described by Fredriksson et al. <sup>[33]</sup>. The specimens and the ceramic plate were then placed in the pressure plate extractor (Model 1500 15 Bar Pressure Plate Extractor, Soilmoisture Equipment Corp., Santa Barbara, CA, USA). To ensure hydraulic contact, a piece of cotton cloth was placed between the saturated ceramic plate and the specimens <sup>[28]</sup>. The extractor was then closed and a pressure was applied in the extractor using nitrogen gas. The experiment continued until no outflow of water was registered in the burette (0.05 ml resolution) for at least 12 days. A desorption experiment was also performed in a pressure membrane extractor (Model 1020 Pressure membrane extractor, Soilmoisture Equipment Corp., Santa Barbara, CA, USA). Here, a cellulose membrane is used instead of a ceramic plate. Apart from that, the experimental procedure is the same. Which pressures that were applied in the experiments in the two extractors are shown in Table 2.

When equilibrium was reached, the specimens were removed from the pressure plate extractor/pressure membrane extractor and the mass of each specimen was determined. Finally, all specimens were dried at 50 °C (lime silica brick specimens) or 105 °C (wood specimens) and the moisture content was determined by Eq. 4.

### 4.2 Sorption isotherms for wood in the hygroscopic range

For comparison, sorption isotherms for wood in the hygroscopic range was determined using a sorption balance (DVS Advantage, Surface Measurement Systems Ltd., London), which monitors the mass of the specimen with a resolution of 0.1  $\mu\text{g}$  while the relative humidity is incrementally changed <sup>[34, 13]</sup>. The relative humidity is regulated by changing the proportions of dry and water-saturated nitrogen gas.

A piece of wood with the approximate dimensions  $2 \times 2 \times 7 \text{ mm}^3$  (radial  $\times$  tangential  $\times$  longitudinal) was cut so that it contained both latewood and earlywood. The sample was placed in water for about 30 min and was then placed in the sorption balance. The sample was then equilibrated to 97%, 96%, 95%, 85%, 75%, 65%, 55%, 40%, 20% and 0% RH first during desorption and then during absorption. The 30 minutes in water was enough to achieve an initial moisture content that was substantially higher ( $>180\%$ ) than the equilibrium moisture content at 97% RH.

## 5. Results

The results from the absorption pressure plate measurements for lime silica brick are shown in Fig. 2 together with pressure plate desorption data from Johansson <sup>[28]</sup>. For the lime silica brick, the moisture content measured during absorption using the pressure plate technique are significantly lower than the moisture content measured during desorption as expected.

The results from the absorption pressure plate measurements for the spruce wood are shown in Fig. 3 together with the data from the comparative measurements. The moisture contents obtained from the pressure plate absorption experiment is significantly lower than the desorption isotherm also for high relative humidity levels. Note that the number of data points is not always clear from Fig. 2 and 3 since some data points are very close to each other (e.g. the pressure plate absorption data in Fig. 3). The number of specimens on each pressure level is given in Table 2.

## 6. Discussion

### 6.1 Comparison of results

For the lime-silica brick the pressure plate absorption data is lower than the data for desorption (Fig. 2) which indicates that the method provides absorption data. This is true also for wood where the data from the pressure plate absorption experiments is significantly lower than the desorption data which is expected since also previous studies has indicated that the hysteresis is large in the high moisture range for wood <sup>[16, 10]</sup>. In addition, the absorption moisture contents measured in the present study is in the same range as the absorption data presented by Penner <sup>[16]</sup> and Fortin <sup>[17]</sup> for spruce and western hemlock respectively. The desorption data from the present study, to which the absorption measurements were compared, are in the same range as desorption results from previous studies performed on softwoods around  $20^\circ\text{C}$  <sup>[16, 17, 20]</sup> as well as data for aspen sapwood <sup>[18, 19]</sup>. However, there are two exceptions: Thygesen and Engelund <sup>[26]</sup> and Stone and Scallan <sup>[35]</sup>, in both these studies the desorption moisture contents are significantly lower. However, Stone and Scallan <sup>[35]</sup> used very thin samples (microtomed cross sections) and the equilibrium moisture content for desorption should then be lower since the thickness of the sample is significantly smaller than the length of a tracheid cell. When using a thicker sample including full length cells with the interconnecting bordered pits, the cell lumen will be emptied when the applied pressure correspond to the radius of the bordered pits openings <sup>[36]</sup>. However, when a thin sample is used, it should be the radius of the lumen that is decisive. Since the lumen radius is larger than the radius of the bordered pits, lumen will be emptied at a lower pressure, i.e. at a higher

relative humidity, when thin samples is used. The desorption isotherm for high relative humidities should thus be lower for thin samples than for thicker samples which explains the lower desorption isotherm obtained by Stone and Scallan <sup>[35]</sup>. The reason for the lower values measured by Thygesen and Engelund <sup>[26]</sup> is more difficult to explain. They used specimens of thicknesses 10 mm. The only difference between the study by Thygesen and Engelund <sup>[26]</sup> and the present study is that the specimens were dried at 102 °C before the vacuum saturation in the study by Thygesen and Engelund <sup>[26]</sup> and the vacuum saturation procedure was slightly different. It does however not seem likely that these differences would results in such a large difference in equilibrium moisture content.

For both absorption and desorption one run was performed at each pressure level and consequently all specimens at a certain pressure level were included in the same run. The small difference between specimens seen especially for the wood pressure plate absorption data, is thus due to small variation in material properties between individual specimens and is not related to the reproducibility of the method.

## 6.2 Methodical aspects

Compared to pressure plate desorption experiments, two main modifications have to be made in order to perform absorption experiments. First, the whole specimen needs to reach equilibrium state from absorption and second the ceramic plate needs to supply the specimens with water. In the present method, the specimens were hanged in magnetic clamps until the ceramic plate had equilibrated with the applied pressure. This is an improvement from the method used by Cloutier and Fortin <sup>[10]</sup> and Fortin <sup>[17]</sup> where the extractor needs to be opened in order to place the specimens on the ceramic plate after it has equilibrated. Since the plate then is exposed to atmospheric pressure, i.e. 100% RH if there is still water below the ceramic plate, there is a risk that the lower parts of the specimen reaches equilibrium state from desorption instead of absorption. In addition, since Cloutier and Fortin <sup>[10]</sup> and Fortin <sup>[17]</sup> preconditioned their specimens before they were placed in the extractor this risk increases since the initial moisture content then is close to the intended equilibrium moisture content. The method presented by Penner <sup>[16]</sup> should result in that the specimens are conditioned to a scanning curve rather than the absorption isotherm since the absorption starts from an RH-level close to the equilibrium RH-level. However, the error in moisture content using Penner's method depends on the slope of the scanning curves which are not known in the high moisture range; horizontal scanning curves would give a smaller error than if the slope is steep. Since the absorption results from Penner <sup>[16]</sup> are in the same range as the moisture contents obtained in the present study this is an indication of that the scanning curves are horizontal as suggested by Nilsson and Sandberg <sup>[37]</sup>. However, since the material properties of the wood used in the study by Penner <sup>[16]</sup> is not known, it is not possible to compare the results.

In the present method, the specimens and the ceramic plate were supplied with water using peristaltic pumps that circulated water in a closed system. Modifications of the ceramic plate are then necessary since it needs to be provided with both an inlet and an outlet. In addition, the internal screen needs to be modified in order to enable water flow below the ceramic plate. Since peristaltic pumps were used, the water was circulated below the ceramic plate continuously. In addition, any air trapped in the system was let out through the hypodermic needle in the stopper on top of burette 2. Therefore, the water level in the burette was not affected by accumulated air as in the method presented by Fortin <sup>[17]</sup> where the water was circulated and air was let out manually. In the present study air bubbles were seen in the tubing for some runs. This was most probably due to a leakage in the connection between the ceramic plate and the outflow tube in these runs. The continuous water circulation in the present study also ensures that there is always water below the ceramic plate. This may not be the case if the circulation is performed manually.



The magnetic clamps generate heat which means that the temperature in the pressure cell is elevated during the period of time when the magnets are powered. In Fig. 5 the temperatures inside the pressure extractor are shown. To minimize the time during which the magnets were powered, and thereby also elevated temperatures, the time to equilibrium for the ceramic plate was tested. The ceramic plate was equilibrated in less than 24 hours and the power to the magnetic clamps was therefore turned off as soon as equilibrium state was reached since drying of the ceramic plate would result in leakage through the emptied pores. No such leakage was observed if the specimens were dropped within 24 hours. Since the temperature in the extractor was elevated the RH was lowered during this period of time. A temperature of 25 °C, which occurred during a short period of time when the magnetic clamps were powered, means that the RH is lowered from 99.9% to about 84% RH. However, since this RH is substantially higher than the RH corresponding to the initial moisture content of the specimens, the specimens still absorbed moisture and were therefore still on the absorption isotherm. In this experiment a ceramic plate designed for a maximum pressure of 15 bar ceramic plate was used. A pressure of 15 bar correspond to a RH of 98.898%. If the RH is lower than this, the largest pores in the ceramic plate will be emptied and there will be a gas leakage through the pressure plate cell. Since no leakage occurred through the plate, this means that the RH of the ceramic plate was not lower than the RH corresponding to 15 bar despite of the elevated temperature. However, in a test experiment, when the magnets were powered during several days, an air leakage was seen through the plate. It is therefore important to determine the time required to equilibrate the plate in advance so that the specimens can be let down as soon as the ceramic plate has equilibrated. In this case, the equilibration time for the ceramic plate was shorter than 24 hours. Future improvements can possibly be made by using magnetic clamps that generate less heat.

In the present study, a significant leakage was seen through the tubing as shown in Fig. 4. This leakage (0.01 ml/h) was subtracted from the measured change in water level in the absorption experiment in order to determine equilibrium state. Examples of the change in water level from the burette reading with and without compensation for the leakage through the tubing are shown in Fig. 5. Fig. 5b show the change in water level for the measurement on wood presented in this paper while Fig. 5a show the change in water level in a pre-experiment where the water level was monitored more frequently. In Fig. 5 it is seen that the change in water level in the burette is constant during the end of the measurement period when the leakage is taken into account. The dominating leakage was through the silicone tubing used in the peristaltic pumps and the leakage may therefore be reduced if peristaltic pumps with for example tygon tubing are used instead. Apart from the experiment described in section 2.3, the leakage through the tubing was also tested by placing water filled sealed pieces of tubing in a room with constant climate (20°C/55% RH) and weighing them regularly. However, the leakage was then significantly lower than when the tubing was used in peristaltic pumps.

A flow resistance between the ceramic plate and the neoprene diaphragm can cause a pressure gradient that may affect the moisture balance of the ceramic plate and thus also the moisture balance of the specimens. The pressure difference was therefore measured by connecting an additional piece of tubing to the outlet tubing by a T-bore stopcock. The piece of tubing was placed in a vertical position and the water level was measured. The highest registered water level in the vertical tube was 39 cm which correspond to a pressure of 0.04 bar. This means that the pressure difference over the ceramic plate was 0.04 bar smaller by the outlet stem than by the inlet stem. This difference in pressure means that the RH close to the outlet stem may be 0.003% higher and the specimens close to the outlet stem may therefore equilibrate with a 0.003% higher RH. This pressure difference may be reduced by further modifying the internal screen so that the resistance to water flow is decreased.

The present pressure plate experiments were performed in a pressure plate extractor with a maximum pressure of 5 bar. This means that using this equipment, absorption measurements between 99.63% RH and 100% can be performed. However, since the design of the pressure plate equipment used up to 15 bar (i.e. from 98.90% RH) is similar it should be possible to use the method in this extractor as well. This would enable pressure plate absorption measurements between 98.90% RH and 100% RH. Even though the design of the pressure membrane extractor that is used up to 100 bar is different, it should be possible also to modify that cell in a similar way. However, this is a more challenging task because of the high pressure.

## 7. Conclusions

This paper presents a method for determination of absorption isotherms in the high moisture range. The major differences compared to previous methods is that the specimens were suspended in magnetic clamps and were let down when the ceramic plate had reached equilibrium with the applied pressure. This is advantageous since the specimens can be dried to a low RH before the experiment starts which ensures that they are conditioned to the absorption isotherm rather than a scanning curve. The test measurements performed on lime-silica brick and Norway spruce wood showed that the moisture contents from the absorption experiment were significantly lower than the desorption moisture content obtained by pressure plate measurements. In addition, the absorption moisture contents obtained by the pressure plate method seemed to follow the same sorption isotherm as the absorption data measured in the hygroscopic range using a sorption balance. The test measurements thus show that the method presented here can be used to measure the absorption isotherm in the superhygroscopic range.

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## Figures and Tables

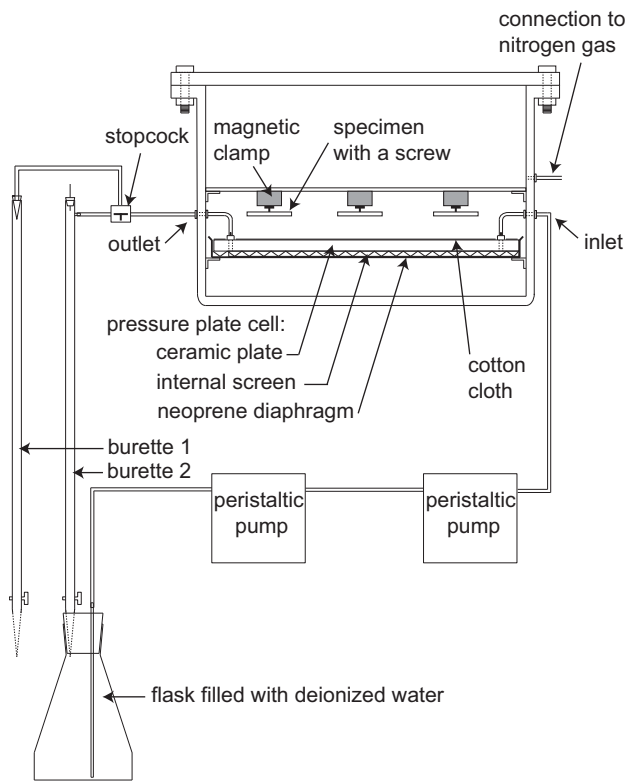


Figure 1. Schematic illustration of the experimental setup in the pressure plate absorption experiment.

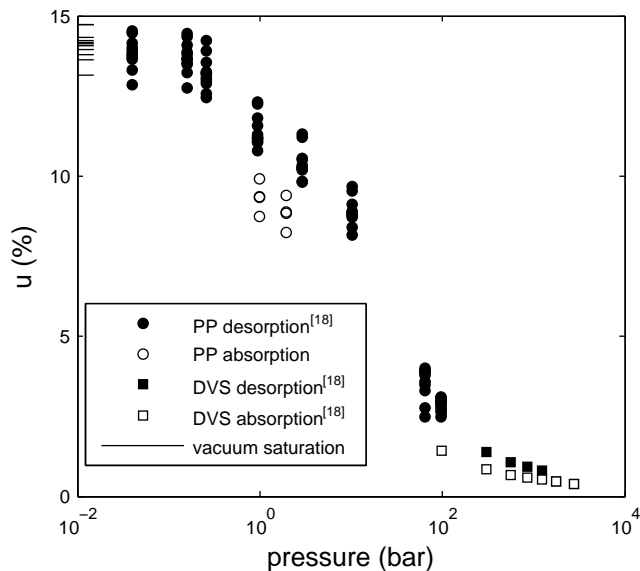


Figure 2. The results for lime-silica brick from the pressure plate absorption measurements (PP absorption) together with pressure plate desorption data from Johansson<sup>[18]</sup> (PP desorption) and data from Johansson<sup>[18]</sup> in the hygroscopic range (DVS desorption and DVS absorption). The horizontal lines on the y-axis indicate the moisture content at vacuum saturation.

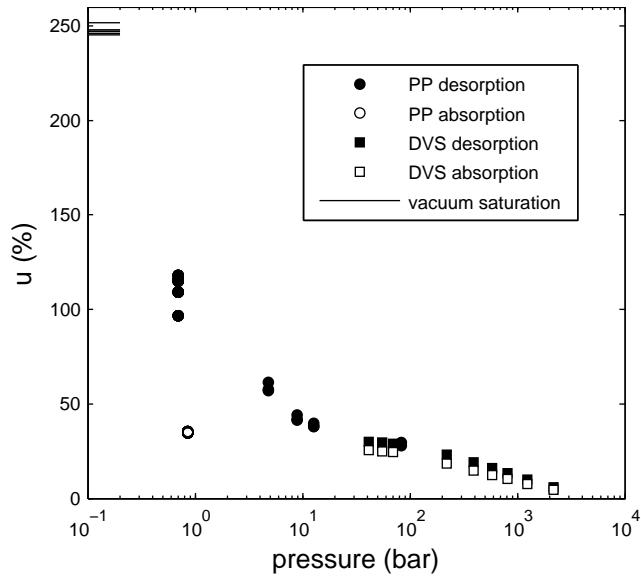


Figure 3. The results for the spruce wood from the pressure plate absorption measurements (PP absorption), pressure plate desorption (PP desorption) and sorption isotherms in the hygroscopic range (DVS desorption and DVS absorption). The horizontal lines on the y-axis indicate the moisture content at vacuum saturation.

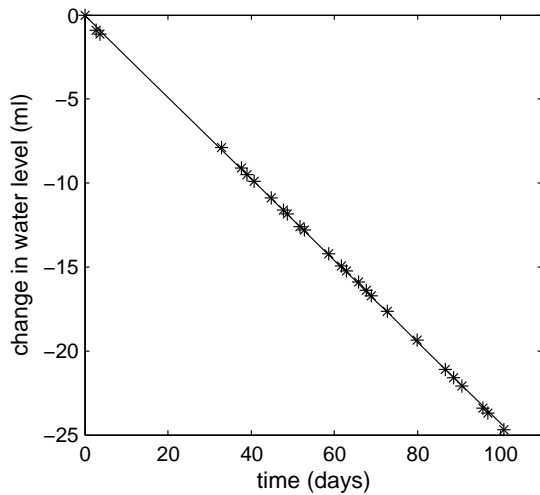


Figure 4. The leakage through the tubing used in the experiments, measured in the control experiment (see section 2.3).

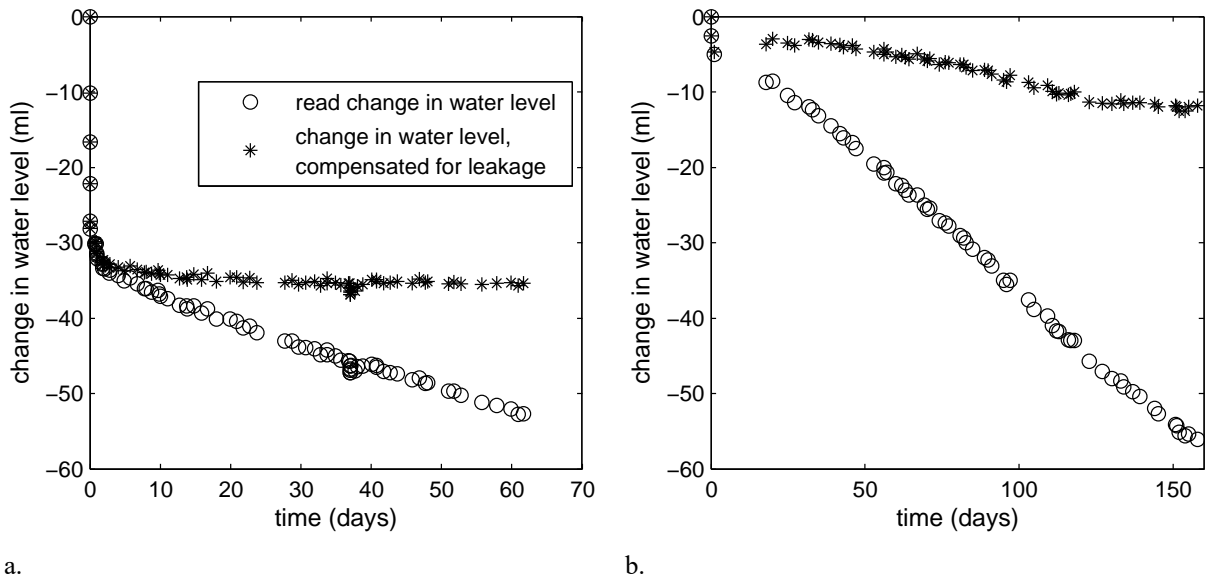


Figure 5. The change in water level in a pre-experiment (a) where the water level was monitored more frequently and the change in water level for the wood absorption pressure plate measurements described in the present paper (b). The initial change in water level was smaller in b since all tubing was filled with water from the start of the experiment. Note that modifications were made during the pre-experiment in a and there are therefore some disturbance at for example  $t = 35$  h.

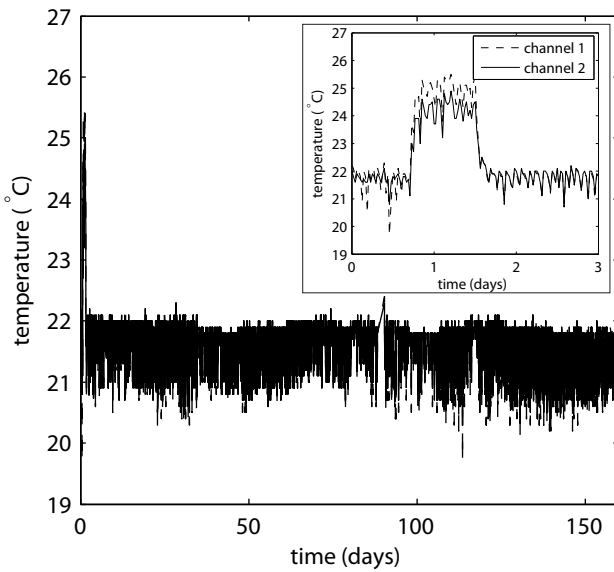


Figure 6. The temperature inside the pressure chamber measured at two positions. The temperature during the first three days including the period when the magnetic clamps were powered is enlarged in the upper right corner.

Table 1. The density and the porosity for the lime-silica brick used by Johansson<sup>[18]</sup> and in the present study. Mean values and the standard deviation (std) are given.

	density (kg/m <sup>3</sup> )		porosity (%)	
	<i>mean</i>	<i>std</i>	<i>mean</i>	<i>std</i>
Johansson (2005) <sup>[18]</sup> (batch 1)	1911	15.6	27.7	0.6
Johansson (2005) <sup>[18]</sup> (batch 2)	1898	31.4	27.7	1.2
the present study	1890	35.7	27.1	0.7

Table 2. Overview of the pressure plate experiments performed in the present study.

	Maximum pressure, extractor  (bar)	Applied pressure  (bar)	Applied pressure  (MPa)	RH  (%)	Number of samples
<i>wood</i>					
absorption	5	0.85	0.085	99.937	6
desorption	15	0.70	0.070	99.948	6
	15	4.8	0.48	99.646	3 <sup>1</sup>
	15	8.9	0.89	99.345	3 <sup>1</sup>
	15	12.7	1.27	99.066	3 <sup>1</sup>
	100	33.5	3.35	97.555	3 <sup>2</sup>
	100	82.6	8.26	94.079	3 <sup>2</sup>
<i>lime silica brick</i>					
absorption	5	0.99	0.099	99.927	4 <sup>3</sup>
	5	1.93	0.193	99.858	4 <sup>3</sup>

<sup>1,2,3</sup> same number indicates that the same specimens were used on these pressure levels