

# Methods for moisture storage and transport property determination of autoclaved aerated concrete

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**Abstract:** The hygric performance of autoclaved aerated concrete is a key determinant for many other material properties as e.g. Thermal conduction, carbonation or shrinkage behavior. Laboratory determination of hygric material properties, i.e. Moisture storage and moisture transport, is hence a prerequisite and a standard in production and process supervision.

In that context, prediction and simulation of the hygric material performance based on numerical calculation models has become a widely used research and design tool. However, for assessment of the material behavior under variable climatic conditions, the hygric material properties have to be determined in a first step. In a second step, these properties have to be transformed into the non-linear coefficients required by these numerical calculation models.

This paper is the first out of two focusing on the first step. It introduces different methods for moisture property determination. The methods cover the full range of possible moisture stages. Moisture storage and moisture transport is distinguished. For moisture transport, vapor and liquid phase transport is considered by different direct and indirect methods.

The methods are applied for an aerated autoclaved concrete. The obtained data is shown and discussed. In a second step, described in a second paper [30], this data is used to derive the non-linear material functions required for sophisticated numerical simulation of the hygric material performance.

**Keywords:** moisture storage, moisture transport, measurement methods

## 1. INTRODUCTION

Moisture is a key determinant for the performance of AAC with regard to strength, shrinkage, carbonation and thermal insulation [1]. For understanding of the governing processes as well as for modeling and predicting the hygric material behavior under arbitrary boundary conditions, a complete set of hygric material properties is essential. This set of material data has to be determined by measurement from different experiments.

## 2 METHODS

The relevant material properties comprise moisture storage and moisture transport properties where moisture transport further distinguishes between vapor and liquid transport.

### 2.1. Moisture storage

Moisture storage data comprises the hygroscopic and the overhygroscopic moisture content range. Different procedures and measurement technologies are applied in both moisture regions. Therefore a further distinction is made for moisture sorption and moisture retention.

Whereas the sorption methods use the relative humidity as the moisture potential, the retention methods employ the capillary pressure. Both of them are linked via the Kelvin equation (1) where  $p_c$  is the capillary pressure,  $\rho_l$  is the liquid water density,  $R_v$  is the gas constant of water vapor,  $T$  is the temperature and  $\varphi$  the relative humidity.

$$p_c(\varphi) = \rho_l \cdot R_v \cdot T \cdot \ln(\varphi) \quad (1)$$

In a strict sense, the Kelvin equation is not valid for the whole moisture range since a continuous liquid phase, at least inside the small pores, and thus more than a few molecular water layers at the inner surface is required. However, it offers a good illustration of the complementary moisture storage data within the different moisture ranges.

### Moisture sorption

The measurement of moisture storage within the hygroscopic moisture content range is widely done by exposing a material specimen to a defined relative humidity, waiting until equilibrium (constant mass) is reached, and gravimetrically determining the corresponding moisture content.

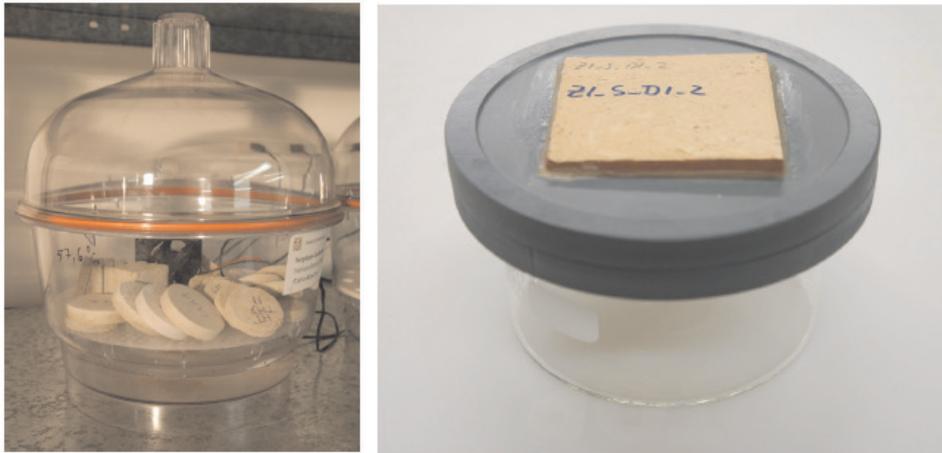


Fig. 1. Left: Desiccator with material samples above a saturated aqueous salt solution. Right: Measurement cup for vapor diffusion experiments according to [21]

There exist two ways of doing such experiments. First is to use a complete device, as a micro balance system [10] or a sorption calorimeter ([1],[3]). They adjust the relative humidity and perform the gravimetric measurements automatically. The second way is to employ the conventional desiccators with saturated aqueous salt solutions ([26], [3]), see Fig. 1 (left). This method is also standardized in ISO 12571.

To adjust a defined relative humidity inside a desiccator, saturated aqueous salt solutions are used. A fan inside the desiccator causes quick equilibration of the humidity conditions. To keep the temperature constant, either a water bath for the desiccators, or a temperature controlled room can be used. The material specimens are placed into the desiccator and left there for a certain time, normally two to four weeks, until equilibrium is reached. The criterion for the equilibrium is defined in ISO 12571: The mass change of the

specimen of three subsequent measurements within 48 hours has to be less than 0.1% of the dry mass of the specimen. Once the equilibrium is reached, the material specimen is weighed. From the ratio to the dry weight, the moisture content can be derived.

### Moisture retention

The moisture storage within the overhygroscopic moisture range is frequently called moisture retention due to the most common measurement techniques used for its determination. There, a pressure is applied to an initially saturated specimen and the retained water mass is measured after equilibrium is reached. The most common device employed for such measurements is the pressure plate apparatus.



Fig. 2. Pressure plate apparatus consisting of two 5bar and two 15bar pressure chambers

The pressure plate apparatus consists of a pressure chamber to which an overpressure can be applied, a set of ceramic plates and a gas pressure supply with a regulation system, see Fig. 2. The ceramic plate holding a certain air entry value, i.e. a certain resistance to the pressing air to penetrate the plate, is placed inside the pressure chamber. At the left hand side of Fig. 3, a ceramic plate with an air entry value of 0.1 MPa is shown. Upon the ceramic plate, saturated specimens are set providing hydraulic contact by a special kaolin-silt mixture. To prevent the specimens from drying out by evaporation, it is recommended to cover them with a wet towel [11]. The bottom side of the ceramic plate is connected to atmospheric pressure via an outlet in the pressure chamber, where the water pressed out of the specimens is allowed to leave. By applying a controlled overpressure, a certain amount of pores will be drained according to their size. When equilibrium is reached, the specimens are removed and their moisture content is determined gravimetrically, before the next pressure step is started. This measurement procedure is standardized in ISO 11274.

The criterion for equilibrium according to ISO 11274 is, that the mass change between measurements at two subsequent days is less than 0.02% of the sample mass. The time required for equilibration generally depends on the investigated material and the height of the specimens. The authors' experience from a large number of measurements shows that for most building materials equilibrium is reached after three to four weeks, for

materials with a very low liquid water conductivity (as e.g. concrete) six weeks are sufficient (for a sample height of 1 cm). For different applied pressures, different pressure chambers and different ceramic plates according to their air entry value are used, where it is recommended to use always the plate with the smallest possible resistance ([11], [12]). The accessible pressure range of the measurements reported here is shown in 0.

Table 1. Pressure steps and air entry pressures of available ceramic plates used for our measurements

<b>Applied pressure (MPa)</b>	0.003	0.01	0.03	0.06	0.1	0.2	0.5	0.8	1.4
<b>Used ceramic plate (MPa)</b>	0.05	0.05	0.05	0.1	0.2	0.3	0.5	1.5	1.5

Normally, only the desorption characteristic is measured by this method due to the difficulty to ensure perfect water contact at the bottom side of the ceramic plate. With specially designed and flushable ceramics, this would be possible, though very laborious. The idea of the pressure plate apparatus goes back to Richards & Fireman [25] who first build such a device and compared their measurement results with those from suction measurements obtaining good agreement. For an overview on the pressure plate method, see [12]. A general instruction for both, suction and overpressure methods, can be found in ISO 11274.

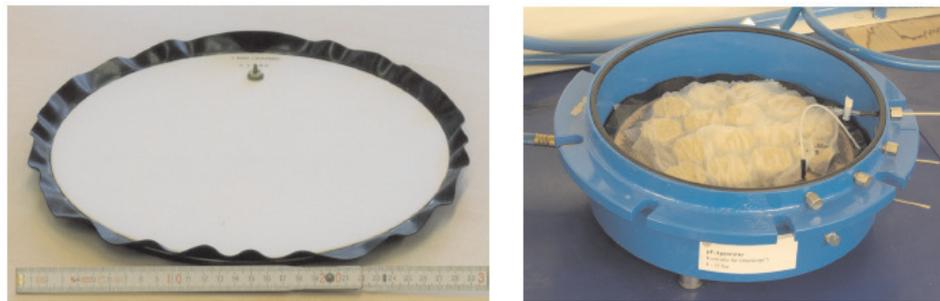


Fig. 3. Details of the pressure plate apparatus. Left: ceramic plate for the hydraulic contact of the specimen. Right: open pressure chamber with specimens covered by a wet towel

## 2.2. Moisture transport

### Vapor diffusion

The basic principle of the vapor diffusion measurement procedure is standardized in ISO 12572. Different measurement devices have been introduced in the literature ([4], [7], [22]), all referring to the same principle. This principle is based on a steady state flow under a constant potential gradient where the flow rate is measured. The potential, i.e. the vapor pressure gradient, can be adjusted by controlling temperature and relative humidity.

For vapor diffusion measurements, so called cups are used, see Fig. 1 (right). Inside the cup, the relative humidity is adjusted whether with a drying agent or with a salt solution. A material specimen is sealed into the lid of the cup providing one-dimensional transport. The cup is closed with the lid allowing vapor to enter (or leave) only through the investigated material. The whole cup is placed either into a climatic chamber or into a desiccator, where another relative humidity is adjusted. Depending on the vapor pressure gradient, vapor will enter or leave the cup. From gravimetric measurements, the flow rate

can be obtained and, after a steady state flow has been developed, the vapor diffusion coefficient can be derived.

With the choice of different relative humidity conditions, the vapor diffusion coefficient can be determined for different potential gradients and correspondingly for different moisture contents. With this regard, the experiment is often called dry-cup or wet-cup experiment. For higher relative humidities, not only vapor diffusion proceeds, but also liquid conduction.

To achieve reliable measurement results, it is recommended to keep the relative humidity difference rather small (i.e. 10% to 20% RH). By preconditioning the material specimens at a mean relative humidity of the planned experiment, the time the steady state flow conditions require to develop can be significantly reduced. This is the reason for the special cup design shown in Fig. 1 (right), where the lid can be easily removed for that purpose. Moreover, the mean moisture content of the specimen can be determined at the beginning and at the end of the experiment.

### Liquid Transport

#### *Water uptake experiment*

The water absorption or water uptake experiment measures the mass increase during a one-dimensional imbibition of a material specimen. It is specified in ISO 15148. To provide one-dimensional transport, the lateral sides of the specimen have to be sealed. After preconditioning at normal laboratory conditions, the specimen is placed into water with its bottom side. With increasing time intervals, the weight of the specimen is determined. For this purpose, the specimen is taken out of the water, its bottom side is dabbed off with a towel and it is placed on a balance. This has to be repeated several times to obtain a number of measurement points forming a characteristic water-uptake versus time curve. According to the standard, the experiment is completed after 24 h.

For homogeneous and isotropic materials, the water absorption experiment consists of two distinct phases, see Fig. 4 curve (a). The first is characterized by a linear mass increase versus the square root of time. The transition from the first to the second phase proceeds when the moisture front reaches the top side of the specimen. A further mass increase is due to redistribution processes involving dissolution of entrapped air and typically proceeds very slowly.

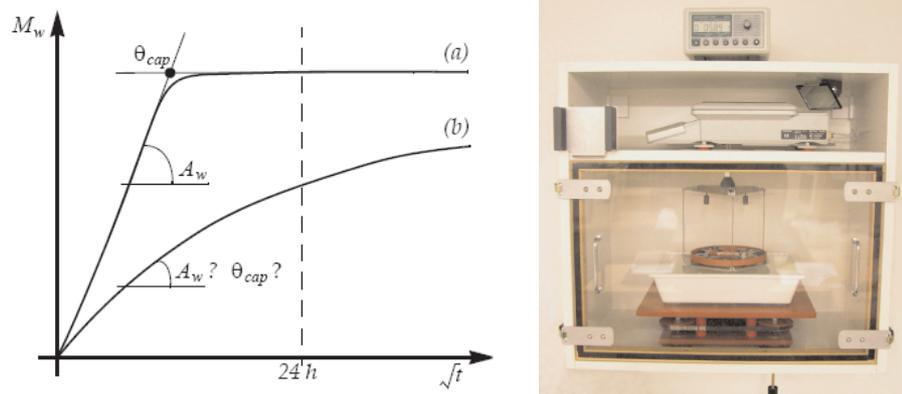


Fig. 4. Illustration of water absorption data of different types of material (left) and automatic water absorption device developed according to [8] and [23] (right)

From this experiment, the water uptake coefficient and the capillary moisture content can be derived. The water uptake coefficient is determined as the slope of the mass increase versus square root of time during the first phase of the experiment. The capillary moisture content is usually taken as the moisture content which is reached when the slope of the mass increase deviates from the linear behavior in the square root of time scale or as the moisture content at the end of the experiment.

The standard ISO 15148 is a specification of minimum requirements. Ahead of these, two details should be taken into account with more care:

- duration of the whole experiment and amount of data points collected during measurement,
- sealing of the upper side to prevent evaporation / moisture absorption due to boundary conditions.

The duration of the experiment is seen most problematic. Since the time the moisture front requires to reach the top of the specimen is dependent on both, the transport properties of the material and the height of the specimen, it is much too general to stop the experiment after 24 hours, see illustration in Fig. 4, curve (b). Therefore, the material behavior should be taken as the criterion when to stop the experiment.

To overcome this problem, an automatic device was developed according to [8] and [23], see Fig. 4 (right). The mass increase is continuously measured by an automatic balance delivering the whole curve. The duration of the experiment can be adjusted exactly to the material behavior. Moreover, the water absorption behavior of materials not following the ideal square root of time course, see curve (b) in Fig. 4, can be observed and evaluated, too.

To prevent a material specimen from drying by evaporation through the upper surface, it is recommended to cover it. The cover must, however, enable air pressure equilibration with the atmosphere [5], [31]. Therefore, the top sealing should be pierced or some gaps should be left for the air pressure to equilibrate.

For more information about the water absorption experiment and different influences on the absorption behavior of materials, refer to [2], [3], [5], [6], [9], [14], [17], [19], [23] and [28].

#### *Drying experiment*

The drying behavior of building materials reveals information about the moisture transport properties in the lower unsaturated moisture content range. By that drying plays an important role for masonry structures [18]. Moreover, drying data is frequently used to identify the transport parameters required in simulation models ([3], [15], [16], [19], [28]).

The general principle of a drying experiment is rather simple. A laterally sealed, saturated material specimen is exposed to certain boundary conditions while its weight change is measured versus time.

The drying is a three-dimensional heat and moisture transport phenomenon that includes evaporation cooling which leads to a temperature and also to a moisture profile. The drying behavior of porous materials depends on:

- The material properties (moisture storage and transport)
- The climatic conditions (temperature and relative humidity)
- The transfer conditions for heat and vapor (air flow conditions [33])

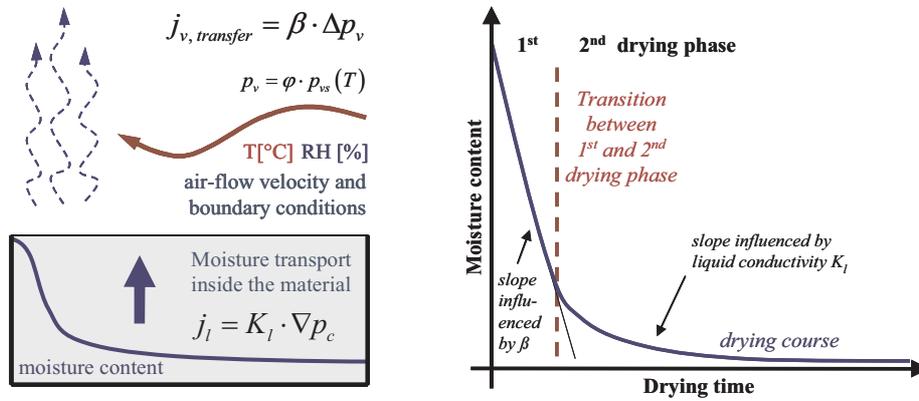


Fig. 5. The drying process: boundary conditions and material properties influencing the drying process (left) and example integral drying curve with a distinction of the two drying phases indicating its influencing parameters (right).

The material properties influence, how quickly moisture can be transported inside the material. The combination of climatic and transfer conditions, i.e. the boundary conditions, defines the speed and quantity of moisture that can be transported to the surrounding atmosphere. The left figure of Fig. 5 illustrates these influences. They become also visible in a material's drying curve, see the figure on the right of Fig. 5. Two distinct phases, called first and second drying phase, can be distinguished. The first drying phase is characterized by an almost linear weight-loss in time. During this phase, the material transports moisture faster to the evaporation surface than what can evaporate. During the first drying phase, the drying is limited by the boundary conditions [27], [29].

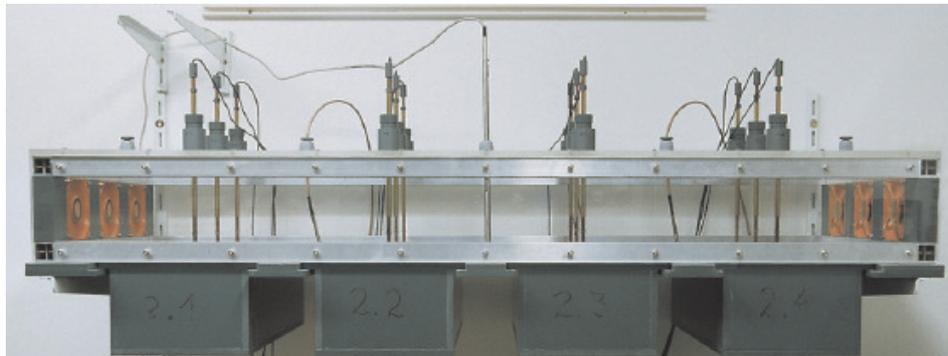


Fig. 6. Device for air flow control and boundary condition measurement during the drying process according to [27].

In the second drying phase, this physical phenomenon reverses. The moisture transport becomes slower and the boundary conditions would allow more moisture movement towards the surrounding air than actually arrives at the surface. The process is slowed down and a distinct moisture content profile develops inside the material. During the second drying phase, the drying is limited by the material properties.

Until now, a standard on drying experiments does not exist. As the drying is influenced by many conditions, it is important to conduct drying experiments under laboratory conditions and measure all influencing parameters, i.e. relative humidity and temperature of the environment and surface temperature of the evaporation surface. For that purpose, a special drying device was developed fulfilling these requirements, see Fig. 6. For more information on the drying as well as on the drying device, refer to [27], [28], [29].

#### *Saturated and unsaturated flow experiments*

Within the capillary pressure range close to saturation, steady state flow measurements at different pressure gradients can be performed to determine capillary conductivity data. In principle, the experimental setup is based on the same idea as introduced for vapor diffusion measurements. A potential gradient is applied to a laterally sealed, saturated material specimen and the development of steady state flow conditions is awaited. From this steady state flow rate, the transport coefficient under the corresponding potential gradient can be derived and related to the water content.

A comprehensive review on different steady state and unsteady state laboratory methods can be found in [11], [13] and [20]. The application of two direct water conductivity measurement devices was reported in [22] and [24]. The principle ideas of both methods are briefly explained as follows.

For the head permeameter device, shown in Fig. 7 (a), a material specimen is sealed with epoxy into a Perspex ring, see Fig. 7 (b). The ring fits into the permeameter head where it is tightly fixed by a rubber pressure membrane (Fig. 7 (c)). One side of the specimen is now accessible to boundary conditions. The other side is, together with the head, connected to a glass container and a vacuum pump. After the permeameter head with the specimen is placed into a water bath, suction is applied to the system connected to the other side causing a water flow through the material sample. The applied suction pressure, the water temperature and the water flow rate are continuously measured enabling the liquid water conductivity to be derived once steady state flow conditions have developed.

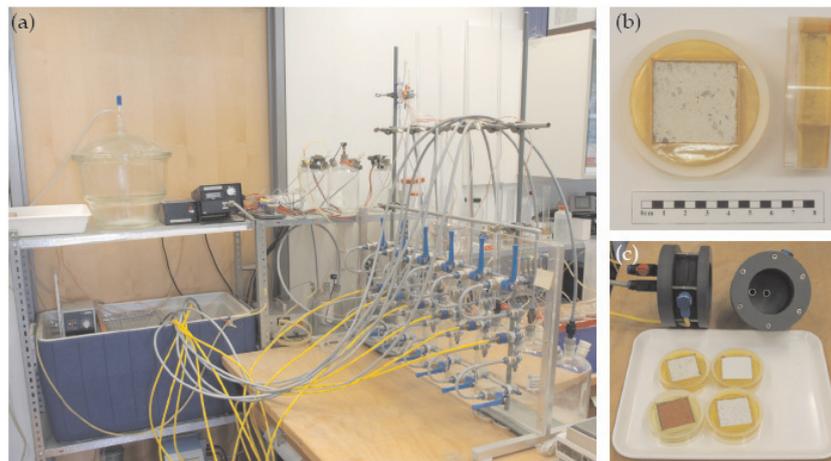


Fig. 7. Head permeameter: complete device (a), material sample sealed into a Perspex ring (b), and permeameter head with material specimens (c)

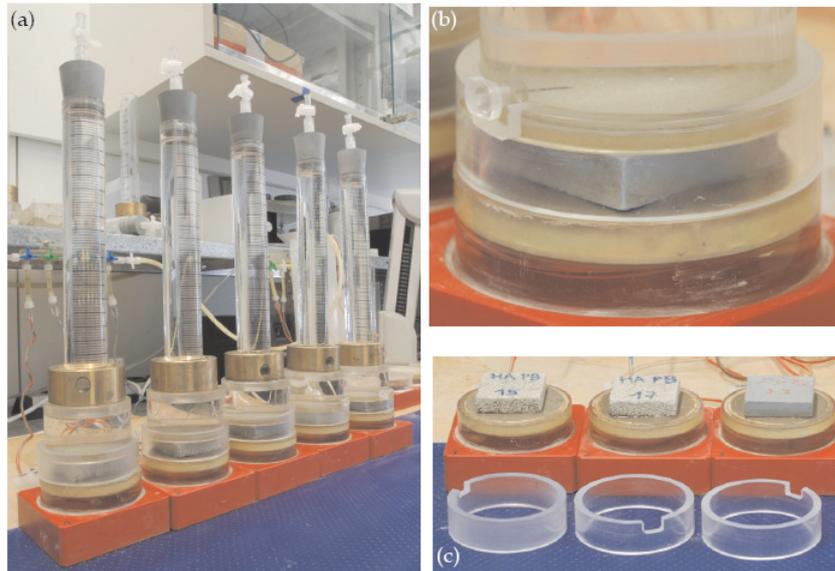


Fig. 8. View on the tension infiltrometer device (a) with connection detail (b) and placing material samples on the flushed ceramic with a silt-kaolin mixture (c)

The second device, called tension infiltrometer, is shown in Fig. 8 (a). It uses a flushed ceramic in combination with a vacuum pump to apply a suction value at the bottom side. A material specimen is set on the ceramic providing capillary contact by the help of a kaolin-silt mixture as shown in Fig. 8 (c). At the top side of the specimen, another suction value is adjusted by the help of a closed water reservoir with a small capillary allowing air to enter in case the pressure falls under a certain value (Fig. 8 (b)). By that, the water from above stays under a defined tension which is dependent on the size of the small capillary, while a higher suction value is applied to the bottom side of the material sample. The rest of the principle is the same as before: steady state flow is awaited, the flow rate is measured and the corresponding conductivity value is derived in dependence to both applied suction values.

### 3. RESULTS

The investigated AAC is a P2 with a density of  $400 \text{ kg/m}^3$ . A list of basic material properties is given in 0.

Table 2. List of basic material properties of the investigated AAC

Parameter	Symbol	Unit	Value
Dry bulk density	$\rho$	$[\text{kg/m}^3]$	400
Total open porosity	$\theta_{\text{por}}$	$[\text{m}^3/\text{m}^3]$	0.87
Capillary saturation	$\theta_{\text{cap}}$	$[\text{m}^3/\text{m}^3]$	0.26
Dry cup vapor diffusion resistance	$\mu_{\text{drv}}$	$[-]$	7.5
Water uptake coefficient	$A_w$	$[\text{kg}/(\text{m}^2\text{s}^{0.5})]$	0.043

The measured moisture storage data is presented in Fig. 9. The relative humidity and the capillary pressure are converted into each other via the Kelvin equation (1). That way, the complementary data from different experiments can be shown in one chart. The dark red dots are data from sorption experiments. The orange dots are data from pressure plate experiments.

The data in the right chart of Fig. 9 shows that the AAC contains pores of many different pore diameters. There are two pore maxima at lower capillary pressures, i.e. two distinctly sharp negative slopes in the data. At higher capillary pressures, i.e. at lower moisture contents, there is no distinct pore maximum. Instead, there are many pores and, thus, a high moisture capacity, in the transition to the hygroscopic moisture content range, which is typical for an AAC.

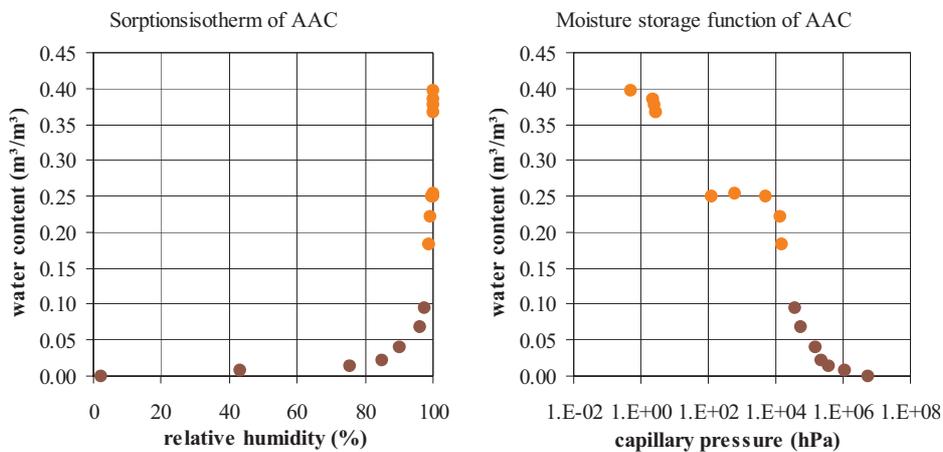


Fig. 9. Moisture storage data of the investigated AAC. Dark red dots represent data from sorption measurements, orange dots data from retention measurements

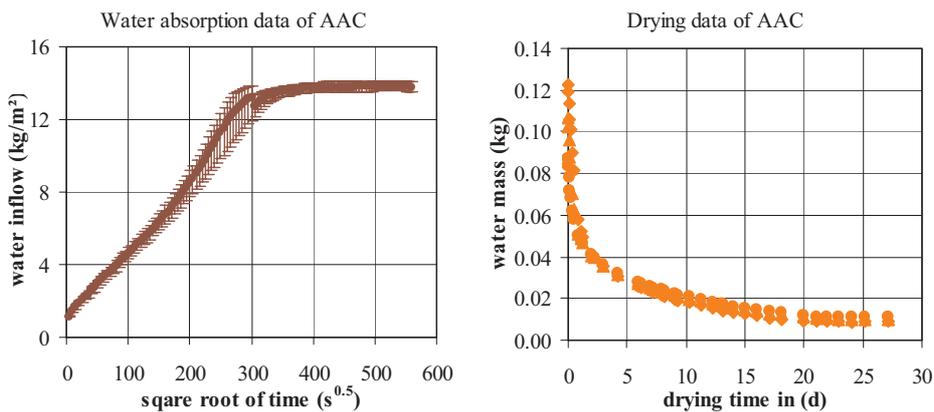


Fig. 10. Water absorption data (left) and drying data (right) of the investigated AAC

Fig. 10 presents data from water absorption (left chart) and drying experiments (right chart). The water absorption behavior follows in principle the expected linear behavior in the square-root of time scale with small deviations. After the moisture front has reached the top end of the specimen (bend in water absorption curve), a small further increase in moisture content is measured. The water absorption coefficient specified in 0 is derived from the linear slope.

The drying curve (right chart in Fig. 10) shows a clear distinction between first and second drying phase. From the slope during the second drying phase one can see, that the AAC dries relatively fast. This is a consequence of the low vapor diffusion resistance and the liquid transport properties in the unsaturated moisture range, which are relatively high.

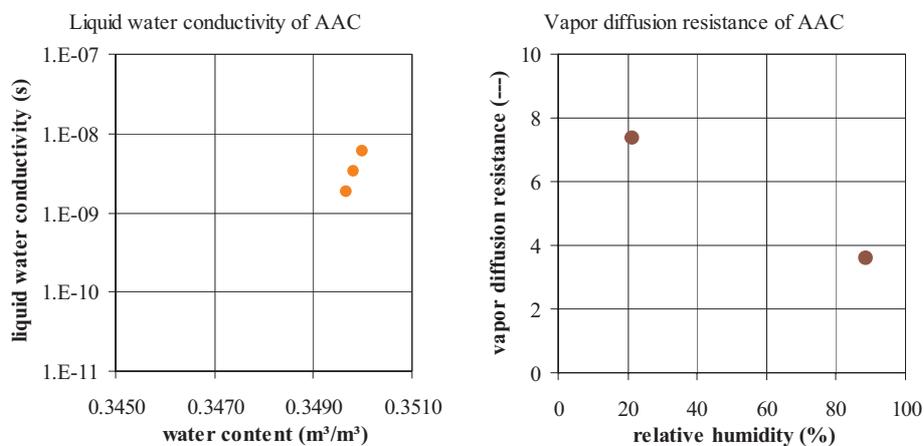


Fig. 11. Saturated liquid water conductivity data (left) and dry-cup and wet-cup vapor diffusion resistance factors (right) of the investigated AAC

Fig. 11 shows results of the vapor diffusion and liquid permeability tests. In contrast to the water absorption and drying experiment, these two methods allow a direct measurement of the transport properties. The left chart of Fig. 11 presents data of the saturated conductivity measurements. This data confirms that a fully saturated AAC has a rather high permeability. The same becomes apparent when looking at the right chart of Fig. 11 where measured dry-cup and wet-cup vapor diffusion resistance factors are plotted versus mean measurement relative humidity. The dry-cup value specified in 0 is the one at a low relative humidity. The wet cup value is significantly smaller since the measurement conditions at higher relative humidity comprise vapor diffusion and liquid transport combined. The significant difference between dry-cup and wet-cup value is hence an indication for reasonable liquid transport properties of the AAC already at low moisture contents. The same indication could be derived from the drying data.

#### 4. CONCLUSIONS

The set of basic experiments for hygric material investigation was introduced. It forms a proper methodological foundation for an advanced but still relatively simply applicable hygric material characterization. The methods were applied for an AAC and

measured data was presented. This dataset forms the basis for material modeling, i.e. the conversion of measured properties into material functions to be used in moisture transport simulation models.

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