7th International Building Physics Conference

IBPC2018

Proceedings

SYRACUSE, NY, USA

September 23 - 26, 2018

Healthy, Intelligent and Resilient Buildings and Urban Environments

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Psychrometer method to measure the moisture retention curves of porous building materials in the full humidity range

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ABSTRACT
Moisture retention curves describe the moisture storage property of porous building materials. Currently available experimental methods for measuring moisture retention curves are often demanding in equipment and moreover not always suited for the adsorption process in most of the capillary moisture content range. This paper proposes a novel and simple technique – the psychrometer method – and validates it by the tests on calcium silicate and ceramic brick. In this experiment the moisture content of the samples experiencing adsorption and desorption processes is determined gravimetrically, while the corresponding capillary pressures in the samples are measured with a chilled-mirror dew-point psychrometer. Comparisons are made with results from mercury intrusion porosimetry, pressure plate/membrane and desiccator tests. It is shown that this psychrometer method is simple and reliable for both adsorption and desorption processes for capillary pressures below $-1\cdot10^5$ Pa. When the capillary pressure is around or above $-1\cdot10^5$ Pa, the psychrometer method is no longer very accurate, and other experimental techniques should be employed.

KEYWORDS
psychrometer, capillary pressure, moisture content, retention curve, porous building material

INTRODUCTION
Hygric properties of porous building materials are indispensable input parameters for analyzing the hygrothermal performance of building envelopes and the built environment (Defraeye et al. 2013; Zhao and Plagge, 2015). In general, hygric properties can be classified as moisture storage and transport properties, characterizing how much moisture can be stored in and how fast moisture can be transported through a material, respectively. Moisture storage properties are not constant; instead, they depend on the ambient humidity. Moisture storage properties are normally depicted either by moisture sorption isotherms, i.e. moisture content (w, kg/m^3) as a function of relative humidity (RH, -), or by moisture retention curves, i.e. moisture content in function of capillary pressure (p_c, Pa). As RH and p_c are interchangeable through the Kelvin-Laplace equation, sorption isotherms and retention curves are equivalent. In this paper we use moisture retention curves to describe the moisture storage properties.

Because of hysteresis, moisture retention curves are usually not a single-valued function but process-dependent. As shown in Figure 1, for a complete characterization in the full humidity range, there are at least three important curves: the adsorption curve starting from the dry state, and two desorption curves, starting from the saturated moisture content (w_{sat}, kg/m^3) and the capillary moisture content (w_{cap}, kg/m^3) respectively.

In the hygroscopic range – where water vapor dominates – the static gravimetric method (e.g. the manual desiccator test (Feng et al. 2013) or the automatic sorption balance (Garbalińska et al. 2017)) is simple and reliable. In the over-hygroscopic range – where liquid water is more important – the currently available experimental techniques are unfortunately less satisfactory. Table 1 summarizes the characteristics of several of these methods. Clearly, none of them can measure the moisture retention curves completely. Even when combined, much information for the adsorption process in the capillary range is still not measurable.

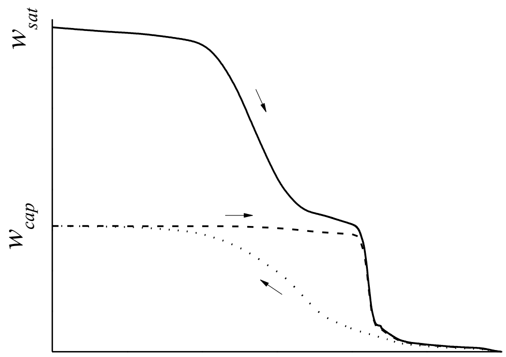


Figure 1. Moisture retention curves.

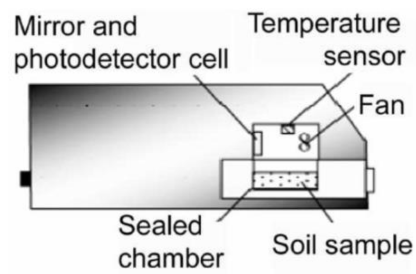


Figure 2. A schematic chilled-mirror dew-point psychrometer (Leong et al. 2003).

Table 1. Characteristics of several methods for measuring moisture retention curves

Method	Mercury intrusion porosimetry (Roels et al. 2001)	Traditional pressure plate/membrane (Hansen, 1998)	Modified pressure plate (Fredriksson and Johansson, 2016)
Equipment price	Very expensive	Expensive	Expensive
Availability	Common	Common	Rare
Toxicity	High (mercury)	Low	Low
Procedure	Simple	Complicated	Complicated
Data processing	Complicated	Simple	Simple
Applicable p_c range	$0 \sim -2 \cdot 10^8$ Pa	$0 \sim -1 \cdot 10^7$ Pa	$0 \sim -5 \cdot 10^5$ Pa
Applicable process	Desorption from w_{sat}	Desorption from w_{sat} and w_{cap}	Adsorption
Duration	Hours ~ days	Weeks ~ months	Weeks ~ months
Others	May not suit cement-based materials	Measurements easily fail	Measurements easily fail

To solve these problems, this paper proposes a novel experimental method – the psychrometer method. It features in simplicity, reliability and most importantly, the capability to do both adsorption and desorption measurements in the full humidity range. In the following sections, we will first introduce the principle of the psychrometer method. Then the validation measurements on calcium silicate and ceramic brick are explained. Finally, our experimental results are presented and compared with extra data from other methods.

METHODS

The key to obtaining moisture retention curves is to determine the corresponding w and p_c in the sample. Moisture content w can be easily obtained through the gravimetric method, while capillary pressure p_c can be obtained by holding the sample in a sealed chamber and measure the humidity of the air inside caused by the water evaporation from the sample. In the over-hygroscopic range the air humidity is close to saturation, thus the widely used RH sensors are no longer reliable and psychrometers instead become a much better choice. Cardoso et al

(2007) studied different types of psychrometers profoundly, and found that many factors – such as temperature, hysteresis, calibration and equilibrium time – all have an impact on the results. After comprehensive comparisons, the chilled-mirror dew-point psychrometer was recommended.

In this study, we adopted the chilled-mirror dew-point psychrometer for the humidity measurement. The instrument model is WP4C, produced by Decagon Devices, Inc (a schematic is illustrated in Figure 2). It is reported by the manufacturer that this psychrometer has an accuracy of $\pm 5 \cdot 10^4$ Pa in the range of 0 ~ $-5 \cdot 10^6$ Pa and ±1% for $-5 \cdot 10^6$ ~ $-3 \cdot 10^8$ Pa. More details about the psychrometer can be found in (Leong et al. 2003).

During the test, calcium silicate (density: 271 kg/m^3) and ceramic brick (density: 1818 kg/m^3) were cut into samples with a diameter of 3 cm and a thickness of 0.5 cm. The dry mass of each sample was determined with a balance with a resolution of 1 mg after oven drying at 70℃ for at least one week. For desorption measurements, the samples were first vacuum saturated and then exposed to 97% or 94% ambient RH (controlled by saturated salt solutions) at 23±0.5℃ . At certain points in time, the desorption process was interrupted by sealing samples into the small sample cups. Trial tests showed that after several hours, the capillary pressure and moisture content distributions within the samples reached equilibrium, indicated by stable psychrometer readings. For this reason, we always carried out the measurements on samples having been isolated overnight. The capillary pressure was measured with the WP4C psychrometer and the wet mass was measured with the balance. Resultantly, the desorption curve starting from w_{sat} in the capillary range was determined. Reversibly, we measured adsorption curve in the capillary range by putting samples pre-conditioned by adsorption at 97% ambient RH above pure water in a closed container, with a similar process for the determination of capillary pressure and moisture content.

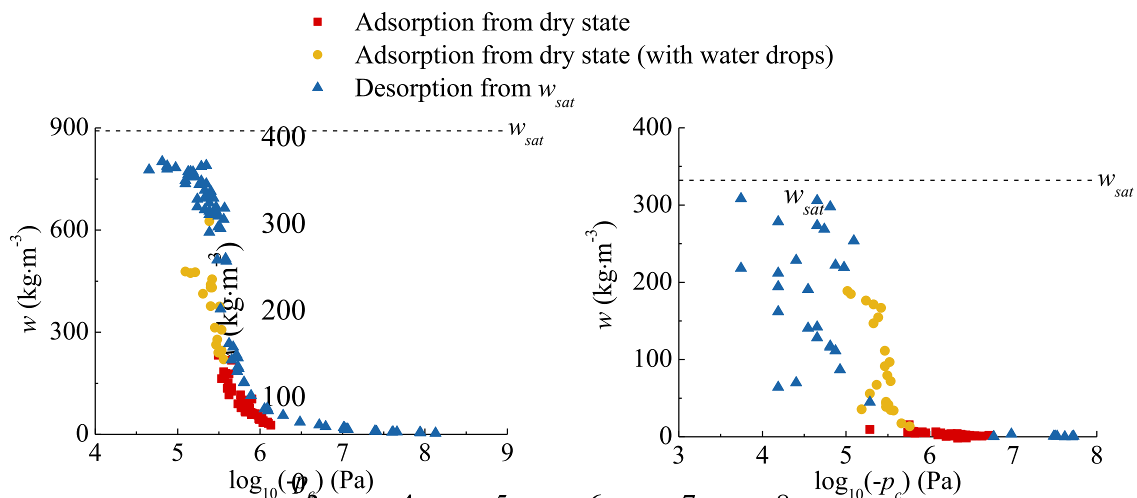
It should be mentioned that for calcium silicate the adsorption progressed very slowly when the humidity is extremely high, while for ceramic brick the adsorption directly from air can hardly result in an observable change in moisture content. For those cases, we applied some tiny water drops directly on the samples for acceleration of the conditioning. To check the applicability of the psychrometer method in the hygroscopic range, we also exposed samples to lower RHs for measurements, as in the desiccator test.

It should also be noted that according to the working principle of the chilled-mirror dew-point psychrometer, an underestimation of w is inevitable due to the evaporation. However, we estimate the total volume of the sealed chamber at 50 mL. A simple calculation reveals that under our experimental conditions 1.1 mg water vapor could yield 100% ambient RH in such volume. Our sample size is roughly $3.5 \cdot 10^{-6}$ m^3 in size, hence the underestimation of w is just around 0.3 kg/m^3, which is completely negligible.

RESULTS

Figure 3 illustrates the experimental results obtained from the psychrometer method described in the previous section. For calcium silicate it is clearly reflected that both the adsorption and desorption curves have been obtained throughout the full humidity range. The results obtained by applying tiny water drops on the samples for accelerating the adsorption process also seem reasonable. It should be noted that even with the tiny water drops on the samples, the adsorption still progressed very slowly, as the whole adsorption process took more than 6 months. Consequently, for capillary pressures above $-1 \cdot 10^5$ Pa ($\log_{10}(-p_c) < 5$), we failed to obtain the adsorption curve.

For ceramic brick the general shape of the water retention curves can be observed. However, the data points display large scatters for the desorption process, and the hysteresis phenomenon also seems illogical (the desorption curve should stay above the adsorption curve). This should be explained by the fact that the ceramic brick used in this study has relatively large pores, with a median radius around $4 \cdot 10^{-6}$ m. Thus, a large drop/rise of the retention curves around $-3.2 \cdot 10^4$ Pa ($\log_{10}(-p_c) \approx 4.5$) is expected. The psychrometer used in this study has an accuracy of $\pm 5 \cdot 10^4$ Pa in that range. Consequently, the measured p_c here are not very reliable.



a) Calcium silicate b) Ceramic brick

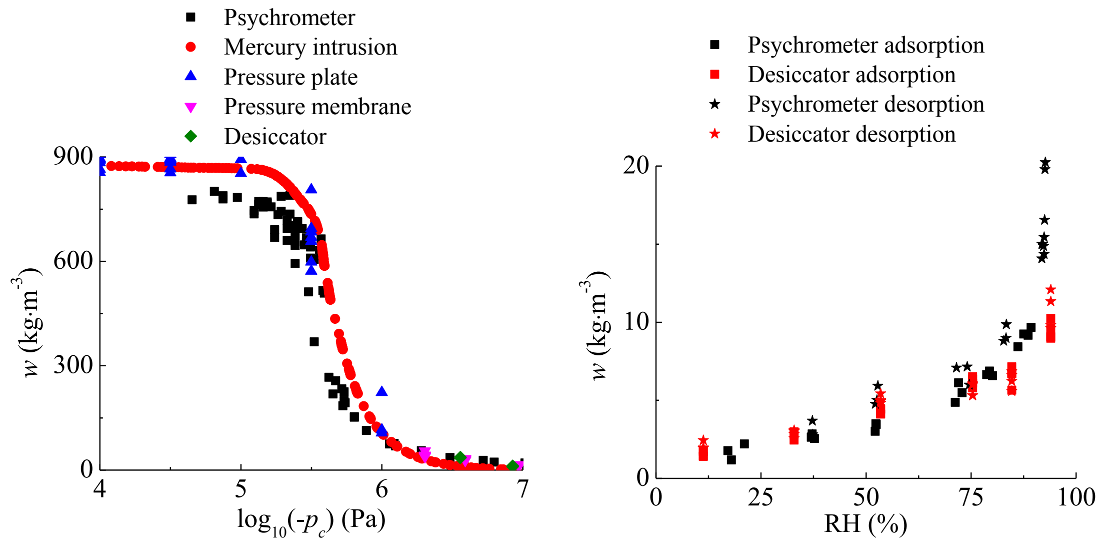
Figure 3. The moisture retention curves from the psychrometer method.

DISCUSSION

To validate the accuracy of the psychrometer measurements, we performed mercury intrusion porosimetry (Roels et al. 2001), pressure membrane (Hansen, 1998) and desiccator (Feng et al. 2013) experiments on the same batch of calcium silicate for comparison. Pressure plate results were also cited from the HAMSTAD project (Roels et al. 2003). As is clearly reflected in Figure 4.a, in the over-hygroscopic range the desorption results from the psychrometer method are generally close to the results from the other methods, albeit that the deviations around $-1 \cdot 10^5$ Pa ($\log_{10}(-p_c)=5$) appear to be more significant. Similarly shown in Figure 4.b, in the hygroscopic range for both adsorption and desorption processes the psychrometer method provides slightly different but reasonable results when compared to other methods.

For ceramic brick the situation is less promising. As illustrated in Figure 5, when compared to results from mercury intrusion and pressure plate tests, the psychrometer method results in a cloud of results – scattering irregularly in the range $\log_{10}(-p_c)$=4-5, reflecting the limitation of the psychrometer method, due to its limited accuracy in this p_c range. However, it should be noticed that the results from mercury intrusion and pressure plate tests are neither very close, and it is difficult to judge which method is more reliable here. Further experimental methods – such as the hanging water column method (Plagge et al. 2007) – are hence necessary to be developed and validated for the p_c range around and above $-1 \cdot 10^5$ Pa.

Last but not least, in this study we just performed the measurements starting from dry state and w_{sat}. The principle of the proposed psychrometer method is obviously also valid for the adsorption and desorption processes starting from other initial conditions.



a) In the over-hygroscopic range (desorption from w_{sat}).

b) In the hygroscopic range (adsorption from dry state and desorption from w_{sat}).

Figure 4. Comparison of the experimental results from different methods on calcium silicate.

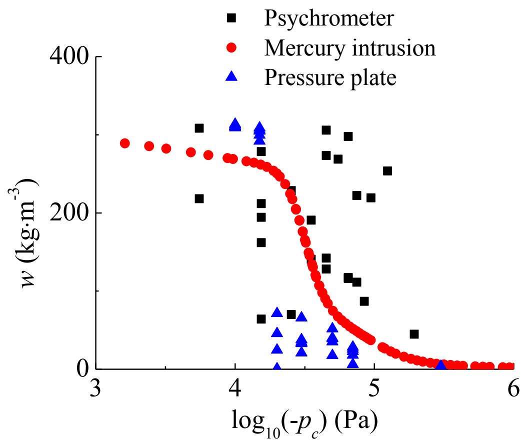


Figure 5. Comparison of the experimental results from different methods on ceramic brick (desorption from w_{sat} in the over-hygroscopic range).

CONCLUSIONS

This paper proposes a novel experimental approach – the psychrometer method – for determining the moisture retention curves of porous building materials. Measurements on calcium silicate and ceramic brick were conducted and the results were validated by comparing with experimental results obtained from the mercury intrusion porosimetry, pressure plate/membrane and desiccator tests. The psychrometer method demonstrates its simplicity, reliability and wide applicable range for capillary pressures below $-1\cdot10^5$ Pa, covering almost the full humidity range. When the capillary pressure is around or above $-1\cdot10^5$ Pa, other more reliable experimental techniques should be developed and validated.

ACKNOWLEDGEMENT

This project is supported by the FWO Odysseus grant "Moisture transfer in building materials: analysis at the pore-scale level" (No. G.0C55.13N).

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