Determination of Instantaneous Moisture Content and Moisture Potential Profiles

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ABSTRACT

For hygrothermal assessment of building envelopes, numerical simulation is increasingly used by the engineering practice. As the applied material functions have a strong influence on precision and information value of the simulation results, it is important to assess them on the basis of meaningful measurement data.

One method to obtain this kind of measurement data is the Instantaneous Profile Measurement technique introduced by Plagge (1991). It allows not only to determine moisture content profiles at different sample positions versus time, but also to measure the moisture potential (relative humidity or capillary pressure) at the same time. Thus this method is very suitable for assessing existing material models as well as improving and validating them.

This study briefly introduces the measurement method and explains the chosen experimental setup and its course. With two building materials different adsorption and desorption runs were carried out within the hygroscopic and wet moisture range. The obtained measurement results are shown and discussed. Their application for assessment of material functions is indicated within the discussion of the obtained results.

INTRODUCTION

Since the application of building physical simulation is more widely used, the demands on quality are increasing. The hygrothermal behavior of building structures under defined boundary conditions should be predictable in a short time and also in a certain manner. Moreover, simulation results should be available in an acceptable time and cost relation.

Besides the simulation program itself and data sets for climatic conditions, material data is required, which normally plays a key role in numerical simulation. Numerous attempts have been undertaken in the past to characterize and verify material properties, usually by determining the storage and transport properties individually and using different methods. Since it is very difficult, time-consuming and tedious to measure these properties, great effort has been made to develop appropriate laboratory methods. Reviews have been presented by Hillel (1980) and Klute & Dirksen (1986), while a more recent discussion of the respective advantages and setbacks of the various laboratory methods available has been worked out by Plagge (1991).

The research reported in this paper is based on dynamic adsorption and desorption experiments, measuring moisture content and moisture potential simultaneously at different sampling positions versus time. Evaluation of the measured data alone and in combination with numerical modelling of the experiments comparing measured and calculated results delivers information about the influence of different effects such as hysteresis of moisture storage or the process dynamics. This can be used to verify limits of employed simplified material models, which are commonly based on measured data from steady-state experiments. Moreover, the applied method supports a physical understanding of transport processes and opens possibilities to develop new physical models and evaluate numerical transport models in describing the moisture behavior of capillary porous materials. Within this paper, this

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is to be shown on the basis of multiple experimental runs with two materials.

As follows, first, the Instantaneous Profile Measurement method (IPM), the experimental setup, the investigated materials and some special features of data evaluation when applying the Time Domain Reflectometry technology (TDR) will be introduced. Subsequently, a selection of results from measurement and numerical simulation are shown and explained. Thereafter, these results are discussed in the context of hygrothermal material modelling, and the potential for further investigations and applications of the used method are derived. Finally, a summary is given and conclusions are drawn.

MATERIAL AND METHOD

For the investigation reported here, the instantaneous profile measurement technique was applied. First, the method is introduced explaining the different setups for the hygroscopic and the overhygroscopic moisture range. Subsequently, an overview about the examined materials is given. Finally, some of the features of TDR data evaluation are outlined.

Measurement Method

To achieve the objectives, a sophisticated IPM technique was developed. A standard material container can be equipped by different types of sensors to investigate different ranges of the flow process within the material sample. The development of TDR as an accurate non-destructive laboratory method opened the possibilities to determine water content in a high temporal resolution. Therefore, a major effort was undertaken to incorporate the TDR technique with miniaturized probes having 0.8 mm diameter rods, (Malicki et al. 1992) as a basic tool of the method.

The basic idea for the development of the method is the use of the same material specimen in a series of experiments, where different flow regimes can be achieved by controlling the initial and boundary conditions. Miniaturized relative humidity sensors of 5 mm sensor diameter are used for the determination of the water potential in the hygroscopic moisture range. For the overhygroscopic moisture range, miniaturized psychrometer sensors were supposed to be used.

In general, the experimental set-up consists of several components. A material specimen with dimensions of 100x100x50 mm is used. The vertical measuring sampler is provided with five pairs of holes, into which guide nuts are screwed prior to drill holes and to insert the specially designed sensors. The material sample is sealed at all but one sides and equipped by TDR-probes and relative humidity sensors, aligned at equal distances of 20 mm along the 100 mm long vertical container. The specimen is connected with its unsealed side to the open top of a dessicator in which the relative humidity is controlled applying aqueous salt solutions. The whole setup is placed into a climatic chamber ensuring accurate temperature control. The boundary conditions inside the dessicator are also measured by a coupled temperature and relative humidity sensor. Figure 1 shows a schematic of this setup.

Movement of moisture can be achieved by controlling the vapor pressure inside the container, where a ventilator is used to control the evaporation rate and to realize the constant relative humidity boundary condition. To monitor, control and register material moisture content and water potential, a computer-aided automatic data acquisition system is used. Further information concerning the sensors use, their calibration and additional experimental details are given by Plagge et al. 1990, Plagge 1991, Plagge et al. 1999 and Plagge 2003.

Experiment. For measurement the material is sampled, sealed, fixed into a standard container and then prepared for sensor installation. The material sample is equipped with 5 temperature and relative humidity sensors and 5 miniaturized TDR probes. Measurements concerning the overhygroscopic moisture range were carried out using TDR probes only.

For the first experimental run, the relative humidity sensors are installed into the sample container. The top of the sample is sealed and the sampling container fixed upon the miniaturized climatic chamber box, where a small ventilator is used to mix the air volume below the specimen. The relative humidity is controlled by a vessel of silica gel as a drying agent or by aqueous salt solutions adjusting defined relative humidities. During the experimental run, the vessel can be removed to measure the increase or decrease of water in time. Water content and relative humidity within the sample and relative humidity and temperature in the chamber are continuously monitored and registered by the computer-aided data acquisition system. Different measurement steps were carried out to cover the whole hygroscopic moisture range. A list of these steps can be found in Table 1.

For the second experimental run, the relative humidity sensors are removed and the holes are sealed. A number of experiments with changing boundary conditions in the overhygroscopic moisture range are performed where phases of water contact, equilibration and drying are combined. The

![Figure 1](image-url)
Investigated Materials

The investigation reported in this paper was done for two different materials, a brick and an insulation material. The brick is a typical burnt brick from Northern Germany. The insulation material is calcium silicate which is highly capillary active and typically applied as inside insulation material. The hydrophysical properties of both materials were investigated at the laboratory of the Institute of Building Climatology at Dresden University of Technology. A list of typical basic material properties is given in Table 3 and the adsorption and desorption moisture storage functions in Figure 3 for both materials.

Data Evaluation

The actual measured quantity of time domain reflectometry sensors is the dwell time of an electromagnetic impulse from which the apparent dielectric constant of the porous medium around the TDR probe can be derived. As the dielectric constant differs strongly for porous media ($\varepsilon_{\text{air}} \approx 1$, $\varepsilon_{\text{material}} \approx 3–6$) and water ($\varepsilon_{\text{water}} \approx 80$), the apparent dielectric constant is very suitable to identify the water content in porous media.

There exist different physical, semi-empirical and empirical models of how to determine the water content from the measured apparent dielectric constant. The physical models (e.g. De Loor 1968) calculate the water content using the physical properties of the porous medium, which are, besides the basic properties of density and porosity, the dielectric constant of the material matrix, the inner surface area, the dielectric constant of bound water, the number of water layers at the inner surface to be regarded as bound and the dielectric constants of air and water.

Semi-empirical models, as the mixed dielectric models given by Tinga et al. (1973) and Birchak et al. (1974), introduce one additional parameter to be adjusted to reference measurements. This offers much more flexibility than the pure physical models but requires additional material information.

For the measurements reported here, the investigated materials were well explored and reference measurements for the whole moisture range were performed. Hence, the 4-phase dielectric model of Birchak et al (1974) has been applied to calculate the water contents.

In case the material properties are not known and reference measurements cannot be performed, which often occurs for field measurements, the empirical approaches can be applied (e.g. Malicki et al. 1994, Plagge et al. 2003). A good overview on TDR technology, including the different models.
for the calculation of water content and electric conductivity of the pore solution is given in Plagge (2003).

EXPERIMENTAL RESULTS

A large number of experiments was carried out which can be seen from Table 1 and Table 2. Showing and discussing all of them would go beyond the scope of this publication. Hence, for each moisture range, two significant experimental runs were selected to be shown and discussed here. For the hygroscopic moisture range, the calcium silicate material with Adsorption 5 and Desorption 4 (refer to Table 1) was chosen. For the overhygroscopic moisture range, the brick with phase 4 and phase 5 (refer to Table 2) was selected.

Hygroscopic Moisture Range

For the calcium silicate, Adsorption 5 was performed as a step of 35 days duration. The boundary condition at the bottom of the material specimen was changed from 32.9% to 97.4% relative humidity using a saturated K$_2$SO$_4$ solution. The whole setup (Figure 1) was placed inside a climatic chamber where the temperature was kept constant at 20°C.

During the experiment, temperature and relative humidity were measured by thermocouple sensors at 5 positions inside the material specimen (see Figure 1). At the same positions, the water content was measured by TDR sensors delivering a set of moisture-content and moisture-potential profiles versus time. These profiles can be evaluated as a dynamic sorption isotherm which is shown for the Adsorption 5 in Figure 4.

Due to the general chronology of boundary conditions, the experiment did not start with equalized conditions but with the hygrothermal stage reached after Desorption 4 which becomes apparent in Figure 4. The two continuous lines represent the functions of the adsorption and the desorption isotherm which were adjusted to data from static measurements according to ISO 11274 and ISO 12571. The data points mark the dynamic sorption isotherms for each position during the adsorption. Position 1 is at the bottom of the specimen which was exposed to the boundary conditions, position 5 is at the top of the specimen which was sealed.

It can be seen that the dynamic sorption isotherm during an adsorption process moves in direction of higher relative humidities (i.e., lower capillary pressures). At position 1,
where the strongest gradient in moisture potential occurs, this is most pronounced. However, all positions of the calcium silicate specimen show this behaviour.

Subsequent to Adsorption 5, Desorption 4 was run changing the boundary condition from 97.4% to 32.9% relative humidity using a saturated MgCl₂ solution. The other conditions were the same as before. In Figure 5 the results of this experiment are again displayed as a dynamic sorption isotherm for each measurement position.

It can be seen that the dynamic sorption isotherm does not follow the course of the static curve. The relation of moisture content and relative humidity again becomes somehow shifted as higher values of moisture content correspond to lower values of relative humidity. The influence is most pronounced for position 1 where the strongest gradient is valid. But for the other positions, the same behavior can be noticed.

Overhygroscopic Moisture Range

For the overhygroscopic moisture range, the experiments of Phase 4 and Phase 5 for the brick have been selected. Unfortunately, the moisture potential could not be monitored in this moisture range. Tensiometers can not be applied because they need to have water contact to measure correctly, which could not be ensured during the whole run. Instead miniaturized psychrometer sensors were supposed to be applied but failed due to technical problems.

However, meaningful results could still be achieved which is shown in Figure 6 and Figure 7. There, a comparison of measured and calculated moisture content profiles is shown. For numerical simulation, the program DELPHIN was used according to Grunewald (1997) and Nicolai (2006). The applied material functions were determined according to
Scheffler et al. (2004) distinguishing the moisture storage function for adsorption and desorption.

In Figure 6, the water content profile versus time is given for Phase 4 (see Table 2). There, the second water uptake after a first one and an equilibration time of 14 days is shown, followed by another time of equilibration with a duration of 14 days. The first day is shown enlarged in the upper corner.

The calculated data shows a quick water uptake leading to higher moisture contents than the measured data. Here, the simulation was done with the adsorption moisture storage function for the whole specimen. For the following equilibration, positions 1, 2 and 3 were calculated applying the desorption moisture storage function while positions 4 and 5 still used the adsorption function. From the results it can be seen, that the distinct moisture profile, which was measured, could not be reproduced by simulation. Instead, two levels of moisture content emerge belonging to the corresponding moisture potential values of the different moisture storage functions. The simulation leads to a higher moisture level than the measured data reaches. This observation will be discussed in the context of the applied material functions under paragraph Discussion.

Figure 7 shows a comparison of measured and calculated moisture content profiles versus time for the brick drying according to Phase 5 (Table 3). The first two days are given in a higher resolution in the small graphic in the upper corner of Figure 7. The drying was performed in the laboratory with boundary conditions of 20°C and around 60% relative humidity, that were measured and applied during simulation.

For simulation, the desorption moisture storage function was used in all positions. It can be seen, that the calculated course differs significantly from the measured one. In position 1, the calculated drying is much faster than the measured. In contrast to that, the water content of positions 3 to 5 increases at the beginning of the drying experiment. Only position 2 shows a rather good agreement between measured and calculated data.

Another interesting fact to be pointed out is that the measured water content decreases until a certain level (see position 1 in Figure 7) which is higher than the equilibrium moisture content according to the boundary conditions (see also the sorption isotherm in Figure 3 on the left). In contrast to that, the calculation dries out until the equilibrium moisture content with the boundary conditions is reached. The same kind of behavior could already be observed during the measurements in the hygroscopic moisture range, see e.g. Figure 5.

**DISCUSSION**

We distinguish between a discussion of the results itself and a discussion of perspectives how to use these results and how to further apply the IPM technology.

**Results of Measurement and Simulation**

It could be shown, besides others, by Topp et al. (1967), Smiles et al. (1971), Stauffer (1977), Hassanizadeh et al. (2002) and Scheffler et al. (2006), that there exists an influence of the dynamics on the relationship between moisture content and moisture potential. However, this influence could mostly only be proved for desorption processes. As shown for the calcium silicate – and although not pictured here, the brick shows the same behavior – this influence does also exist for the adsorption process. While for desorption processes, under strong gradients, the moisture content belongs to higher capillary pressures (i.e., lower relative humidities) than the static equilibrium, for an adsorption process, depending again on the moisture potential gradient, the same moisture content belongs to lower capillary pressures (i.e., higher relative humidities), see again Figs. 4 and 5.

Unfortunately, the measurements within the overhygroscopic moisture range lack data of the moisture potential. Therefore, a dynamic moisture storage function could not be displayed. However, as the purpose of the whole investigation was to receive data for material model validation, numerical simulation could be applied. The employed material model, given in Scheffler et al. (2004), is based on the bundle of tubes model described by Burdine (1953), using the moisture storage function to derive the capillary conductivity function. This function is further adjusted to data from water uptake and drying experiments according to Plagge et al. (2005) and Scheffler & Plagge (2005). As proved e.g. by Tzimas (1979), the liquid water conductivity in dependence to the moisture content is not hysteretic. Thus, only for the moisture storage function the hysteresis has to be taken into account which was done for the adjustment of the conductivity function. Figure 8 shows the final result of this calibration procedure. Both experiments were calculated with the same conductivity function, applying the adsorption moisture storage function for the water uptake experiment and the desorption moisture storage function for the drying experiment. Both storage functions are given in Figure 3. It has to be stated, that the desorption moisture storage function was adjusted to measured data over the whole moisture range whereas the adsorption function could only be adjusted to measured data within the hygroscopic moisture range. Above relative humidities of 97%, the measurement of adsorption moisture storage data is very difficult. Therefore, only few data points exist in the overhygroscopic moisture range. This is the reason why the slope of the adsorption moisture storage function was inversely adjusted by the help of the water uptake experiment within the wet moisture range.

The agreement between measured and calculated integral moisture is almost perfect. Also the surface temperature which was measured during the drying experiment giving a sensitive indicator to the chosen exchange conditions (see also Worch (2004) and Scheffler & Plagge (2005)) could be reproduced by simulation.
However, the comparison of measured and calculated moisture content profiles (Figures 6 and 7) shows significant deviations. One reason for that is the hysteresis of moisture storage. Up to now only the main drying and the main wetting curve can be applied, which does not allow us to follow one of the scanning curves. This becomes clear when looking at the measured moisture profile given in Figure 6 which could not be reproduced by simulation. We used the adsorption moisture storage function in all positions as long as water contact was applied (during the first 20 minutes). Then the desorption moisture storage function was assigned for the simulation of positions 1 to 3 whereas the adsorption function was used for positions 4 and 5. As a consequence, only two and not – as measured – five moisture levels are reached according to the equilibrium moisture content of either the adsorption or the desorption characteristic. Thus, the hysteresis in the moisture storage function is responsible for the developing steady-state moisture profiles.

This, however, does not explain the generally higher moisture level of the simulation results. From the authors point of view, this can only be explained with the influence of the process dynamics on the relation of moisture content and moisture potential, which is also not included within simulation. During desorption processes under strong gradients, the moisture potential moves to lower capillary pressures (i.e., higher relative humidities) for the same moisture content compared with the static case, see also Figure 4. The simulations were carried out with an adsorption moisture storage function which was partly adjusted inversely by simulating the water uptake experiment. Hence, the overhygroscopic bench of this adsorption curve includes static and dynamic effects. However, the dynamic effects included belong to the one case which was used for the adjustment. This was the water uptake experiment shown in Figure 8, which started with a dry material and took as long as the sample was saturated. These conditions do not necessarily correspond with those under which the data presented in Figure 6 was measured. The initial conditions were different and a distinct moisture profile was already established inside the material sample. The moisture potential gradients being responsible for the dynamic effect where thus not the same as during the water uptake experiment used for the calibration.

The liquid water flow is dependent on the liquid water conductivity and on the moisture potential gradient. As the moisture-content – moisture-potential relation is influenced by the process dynamics, this also influences the moisture potential gradient. In the case of an adsorption process, the corresponding capillary pressures will be lower than those of the static case. Since the capillary pressure is a logarithmic variable, this leads to moisture potential gradients which can differ by an order of magnitude for the static and the dynamic case. The higher moisture contents occurring in the simulation are thus attributed to the higher transport rates resulting from higher moisture potential gradients during the water uptake. Due to the dynamic effects, the corresponding potential gradients were lower during the actual measurements than they were calculated during the simulation. In the simulation, the dynamic effects were not taken into account correctly leading to an overestimation of the moisture transport rates.

Furthermore, long-range interactions are normally not taken into account in hygrothermal calculations. The measured drying data displayed in Figure 7 shows an immediate decrease of moisture content for all positions, although it is rather low for the upper positions. The calculated profiles behave completely different, which gives an indication also to this dependency.

Investigation of Moisture Transport Phenomena in Porous Media

As it was the original purpose of the reported investigations, the acquired data can be used to quantify options and
summarizes the data obtained by the IPM technology is very suitable. Thus, a hygrothermal material model validation is proposed on the basis of this data.

Associated with that, both, the measurement data and the comparison with numerical simulation results may lead to a further understanding of the observed phenomena. The hysteresis of moisture storage has been subject to research and modelling for more than 50 years but still most Building Physical simulation models do not include it. The obtained data is very suitable to further understand these phenomena and help to verify hysteresis models for building materials.

Moreover, new models regarding the influence of both hysteresis of moisture storage and the process dynamics, as proposed by Beliaev & Hassanizadeh (2001), gain an experimental basis. A model verification could be done with the help of this data. Further experiments should be carried out with special regard to this field of application.

SUMMARY AND CONCLUSIONS

A new application of the instantaneous profile measurement technique is shown for two materials, a brick and a calcium silicate insulation. With these measurements, a great deal of information concerning hygric material behavior under dynamic conditions could be obtained, especially when the moisture potential was also measured, as done in the hygroscopic moisture range.

In the hygroscopic moisture range, dynamic moisture storage functions can be derived from the measured data showing a clear change in the moisture-content – moisture-potential relation compared to static data. For the wet moisture region, the influence of hysteresis as well as the process dynamics can be shown using numerical simulation to reproduce the measured data.

The data can be further used for material model validation to quantify potential errors the implied model assumptions might cause. Furthermore, new approaches trying to include the hysteresis of moisture storage or the influence of the process dynamics can be verified. Only few data is currently available to show these influences, making the instantaneous profile measurement technology a very promising and innovative measurement method for further applications.

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REFERENCES


