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▶ **To cite this version:**

Floran Pierre, Mehdi Ayouz, Patrick Perre. An original method for the determination of mass diffusivity in unsteady state. Eurodrying'2013, Oct 2013, Paris, France. hal-01829073

HAL Id: hal-01829073
https://hal.science/hal-01829073

Submitted on 3 Jul 2018

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An original method for the determination of mass diffusivity in unsteady state.

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ABSTRACT. This work proposed a new method for the determination of mass diffusion coefficient in hygroscopic materials. The experiment consists in submitting one side of the sample to a variation in time of the relative humidity (RH) and to collect the RH on its back side. The imposed RH is measured during the test and serves as boundary condition in a comprehensive computational code solving heat and mass transfer in porous media. This model is the physical engine embedded in the inverse procedure implemented to determine the mass diffusion coefficient. Compared to classical methods, this new method has several advantages:
- it allows several samples to be measured simultaneously simply by multipexing the RH sensors,
- accurate values can be obtained even when starting and ending out of equilibrium, which allows the characterization time to be drastically reduced,
- the external mass transfer coefficient has negligible effect on the identified value,
- non-standard Fickian behaviours can be detected by the disagreement between the measured and the simulated curves.
The results show that the diffusivity obtained for spruce and for medium density fibreboard (MDF) are in good agreement with the ones found with classical methods. On the opposite, result obtained for fibreboard give unrealistic values which confirm the dual scale nature of mass transfer occurring in this kind of material for unsteady regimes.

KEYWORDS: Transfer; Hygroscopic materials; Identification; Green building.

1 Introduction

Reliable data on the bound water diffusion coefficient of building materials are very important for accurate description of materials drying as well as for the proper design and use of structures and buildings made with these materials. The problem of moisture in buildings has always generated great interest. The investigation of moisture transfer in building materials were mostly investigated regarding building durability (Sereda and Litvan 1980; Derluyn *et al.* 2012). During last decades, the global warming has led to a growing interest in renewable materials and energy savings in buildings construction. Improving the energy performance of buildings became the main motivation in the study of heat and mass transfers in building materials (Piot *et al.* 2011).

The conception of green building is strongly linked to the simulation of air quality inside the envelope. Numerous models have been developed for these purposes (Perré and Turner 1999; Crawley et al. 2001; Woloszyn and Rode 2008) which need materials properties to be known with good accuracy. The determination of bound water diffusion coefficient at a sample scale is especially required. During the last century, two groups of methods for measuring this coefficient were developed. The first one, traditionally called the cup method, uses data from steady state experiments of bound water transfer and is based on Fick's first law of diffusion. Unfortunately, the method is not valid for the bound water diffusion coefficient determination in wood because it cannot satisfy the requirements of the boundary condition of the first kind and the constant value of the diffusion coefficient (Olek *et al.* 2005). The second group of methods is based on the unsteady state experiments and Fick's second law of diffusion. The common name of this group is the sorption method and it was developed to overcome the disadvantages of the cup technique. But studies in this group of method gave unsatisfactories results. Sometimes the diffusion coefficient was assumed as a constant leading to significant errors (Chong and Skaar 1972), sometimes the method induces significant simplifications of the solution (Hrcka and Babiak, 1999) or sometimes experimental procedure is not sufficiently accurate leading to difficulties in the inverse analysis (Koc *et al.* 2002).

The objective of coming works will be to expand the earlier developed inverse procedure (Olek *et al.* 2005) for the diffusion coefficient identification by introducing : the improved optimization procedure, the alternative submodel for the diffusion coefficient dependency on the bound water content, parameterization of the submodel as well as the improved experimental procedure. This paper is a part of these coming works as it is devoted to the presentation of the new experimental procedure for the determination of mass diffusion of hygroscopic materials in unsteady regime.

2 Materials and Methods

2.1 Experimental set-up

The principle of measurement is inspired by Zohoun *et al.* (2003). It consists in imposing a relative humidity on one side of the sample in order to create a transient diffusive flux through the sample.
The special feature of the present study is to determine the diffusivity from the measurement of the relative humidity on both sides of the sample. The sample is put in a sample support made from a PVC tube. An air tube is put inside the tube to insure air

tightness around lateral face of the sample without preventing its dimensional variation. At the backside of the sample (Fig. 2), a sealing disk (PVC) is put against to sample to get a very small gaseous cavity, which includes a Temperature/Relative Humidity sensor (*Sensirion SHT25*). The airtightness of the support was checked by using a PVC sample.

Eight sample supports are placed inside the climatic chamber (*Weiss WKL100*). This is a clear benefit in terms of time saving because such tests require long experience durations. The relative humidity at the outer face of the sample is generated by the thermodynamic conditions of air and water being in the climatic chamber. The dry temperature of the air of the climatic chamber may range from 10 to 95°C and the relative humidity from 10 to 98%. A sensor (*Sensirion SHT25*) is placed inside the chamber to record these two values. The samples supports were subjected to the following air conditions:
- Isothermal condition (35°C):
- Relative humidity of 40% until stabilization,
- Sudden increase of relative humidity to 75%.

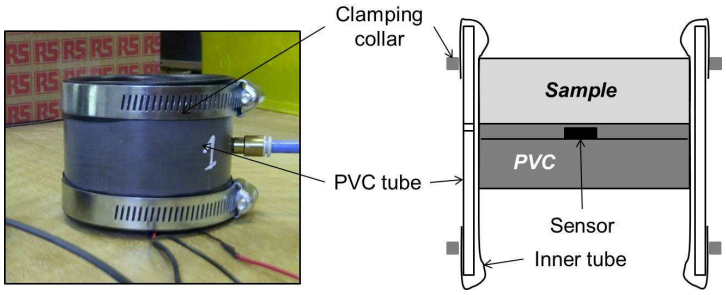


Fig. 1. Sample support

2.2 Materials

Several renewable buildings materials were tested in this study: fibreboard, Medium Density Fiberboard (MDF) and spruce wood (*Picea Abies*) oriented in radial and tangential direction. The material was carefully and slowly air-dried to an equilibrium moisture content close to 12%. The disk-shaped samples, with a diameter of 72 mm, were cut from the equilibrated material. The sample thickness ranges from 10 mm to 20 mm. The lateral surface of the samples were sealed with two layers of an epoxy resin. Using samples with fully coated faces, this resin was previously proved to be air-tight. Because of the propensity of fibreboard samples to absorb the epoxy resin, the preparation protocle was slightly different in this case. The resin was first applied on a thin strip of aluminium coated, which was then applied around the sample.

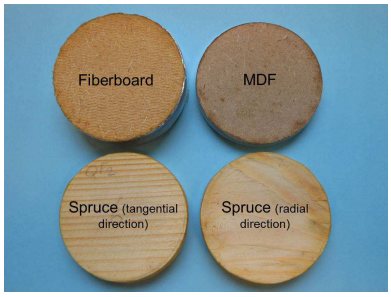


Fig. 2. Tested sample

2.3 Methods

Figure 3 presents the general principle of identification. The known sample characteristics (thickness, density, porosity ...) and estimated values of unknown parameters (diffusion coefficient, initial water content) are supplied to the comprehensive model (*TransPore*) to predict the relative humidity at the backside of the sample. The difference between the predicted values and experimental values is quantified by an objective function *S* defined as the sum of the squared residues :

$$S = \sum_{i=1}^{NT} w_i \left[E_{exp}(t_i) - E_{pred}(t_i) \right]^2 \quad (1)$$

where E_{exp} is the experimental value, E_{pred} is the predicted value, w_i the weight function, t the time and NT the number of time intervals.

An optimisation algorithm (*Simplex*) is used to minimise this function by modifying the unknown parameters. The diffusivity is given as the value reached after convergence.

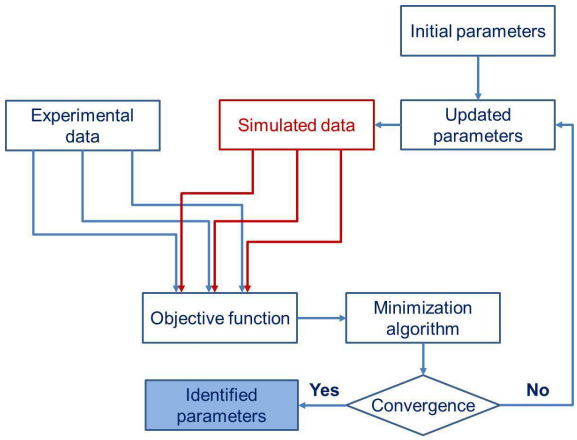


Fig. 3. General principle of the inverse identification method.

Compared to classical methods (Zohoun *et al.* 2003), this new method does not need to wait for equilibrium of the moisture field inside the sample. Actually, the equilibrium relative humidity on the backside is already known at the beginning of the experiment: this is the constant relative humidity imposed in the climatic chamber after the sudden change. Therefore, it becomes possible to determine the diffusivity as soon as the backside RH changes significantly, which results in huge time saving. Keeping in mind that the stabilisation of some hygroscopic materials such as OSB can take several weeks, we can realize the importance of this issue.

2.4 Physical model

The transport equations used by the physical model are presented hereafter. In the hygroscopic range the total flux of mass is expressed by the sum of two parallel fluxes (2). Note that at a microscopic scale these phenomena are more complicated. Indeed, a complex system of parallel and serial fluxes is developing at the microscopic scale due to the pore morphology.

Total mass flux : $$\overrightarrow{q_m} = \overrightarrow{q_v} + \overrightarrow{q_b} \quad (2)$$

Water vapour diffusion : $$\overrightarrow{q_v} = \rho_g D_{eff} \vec{\nabla}(w_v) \text{ with } D_{eff} = f.D_{vap} \quad (3)$$

Bound water diffusion : $$\overrightarrow{q_b} = \rho_s D_b \vec{\nabla}(X) \quad (4)$$

where q_m is the total moisture flux, q_v the water vapour diffusion, q_b the bound water diffusion, D_b the bound water diffusivity, D_{eff} the effective diffusivity, D_{vap} the water vapour diffusivity, f the dimensionless diffusivity, ρ_g is the bulk density of the total gas, ρ_s the bulk density of the lingo-cellulosic part, X the moisture content (dry basis) and w_v the bulk density of water vapour.

Nevertheless, at the macroscopic level, it is difficult, if not impossible, to distinguish the contribution of each flux. From an experimental point of view, this would at least impose two indepednant values to be collected (for example the temperature field in addition to the information regarding moisture content). In this study, the total flux is supposed to be mainly due to the water vapour diffusion. Only equation (3) is used.

3 Results and discussion

In this study, the water migration was assumed to only occur in the gaseous phase. Thus the gradient of bulk density of the water vapour was taking as driving force and the reduced diffusivity f was determined. Note that f ranges from 0 (sample impervious to any moisture transfer) to 1 (open sample having the same behaviour of an air layer at rest.

Figure 4a depicts the evolution of relative humidity at the backside of different samples following a sudden increase of the RH in the climatic chamber from 40% to 75%. The temperature was kept constant (35°C). As expected, the time constant depends on the nature and thickness of the sample. It is about 250 hours for spruce and much faster for MDF and fibreboard (20 and 10 hours, respectively). This previous observation is confirmed by the values of reduced diffusivity (table 1). Note that figure 4b confirms the excellent agreement between simulated and experimental values.

		f (unsteady regime)	f (steady regime) (Zohoun, 2003)
MDF		0.285	-
Fiberboard		>> **1**	-
Spruce	Radial direction	0.017	0.013
	Tangential direction	0.024	0.014

Tab. 1. Reduce diffusivity (f) obtained for each material (average of two repetitions).

The values obtained for spruce are in good agreement with literature data (Agoua 2001). Note that for this material f is similar in steady and unsteady experiments. Agoua (2001) also reported this observation with Norway spruce (*Pinus sylvestris*), teak (*Tectona grandis*) and beech (*Fagus silvatica*).

The identified values for fibreboard give a non-physical value of f. This confirms the double-scale character of mass diffusion for this material in unsteady regime. Ongoing studies will improve the method to account for the non-local balance by considering mass

transfers at a microscopic scale. A dual-scale model is required for this purpose (Perré 2007, 2010).

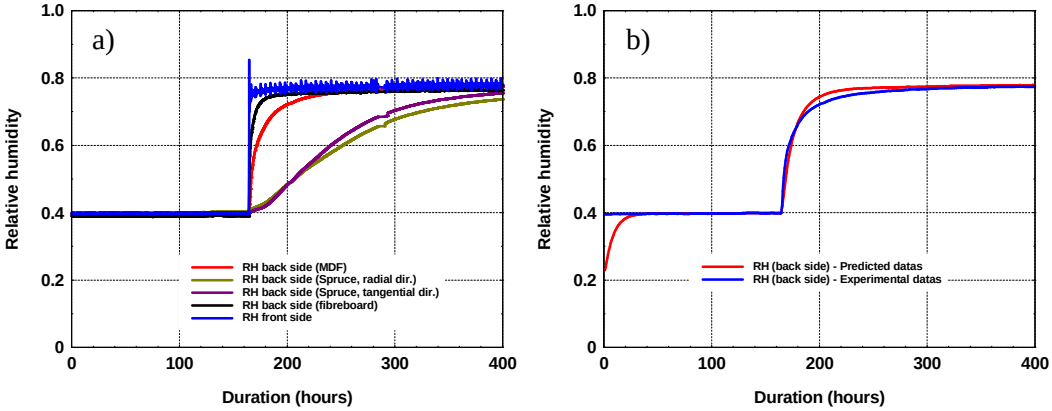


Fig. 4. Experimental values obtained (a); Comparison between experimental and predicted values (MDF) (b).

4 Conclusions

This study presented an original method for the determination of mass diffusion of hygroscopic materials in unsteady regime. Compared to classical methods, it presents many advantages including a drastic reduction of experience time. The obtained data confirm the reliability of the method for wood and MDF: reduced diffusivities obtained are in good agreement with previous studies. It allows also non-Fickian behaviour to be detected. In this case, the method can still be used, provided a dual scale model is used as physical model in the identification procedure.

5 Acknowledgments

This work was financially supported by the ANR project HYGROBAT.

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