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MEASUREMENT BOUND WATER MAXIMUM MOISTURE CONTENT AND DIFFUSION COEFFICIENT DETERMINATION OF BLOWN CELLULOSIC INSULATION MATERIAL IN LABORATORY CONDITIONS

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ABSTRACT

Mechanical properties, dimensional stability, and biological durability are affected by moisture in timber structures; however, moisture is necessary for hygroscopic insulation materials. With high moisture content, wood elasticity is reduced, corrosion of connectors is promoted, thermal conductivity and heat storage capacity are increased. Thanks to hygroscopic fibers, moisture is stored and redistributed in blown cellulose insulation, the hydrothermal balance within timber walls is enhanced. A laboratory method to determine the diffusion coefficient under variable surface fluxes is developed, and the maximum bound water content is measured using Archimedes' principle. 40 cm thick test specimens under controlled interior and exterior conditions were tested in the experiments. Moisture content fluctuation was monitored over three months. The diffusion coefficient was derived from Fick's law and from conservation principle using the inverse method. Results show a decreasing diffusion coefficient that stabilizes over time and a maximum bound water content of 34%. The findings indicate that effective insulation materials must combine a high diffusion coefficient and water storage capacity to manage water condensation and preserve structural durability.

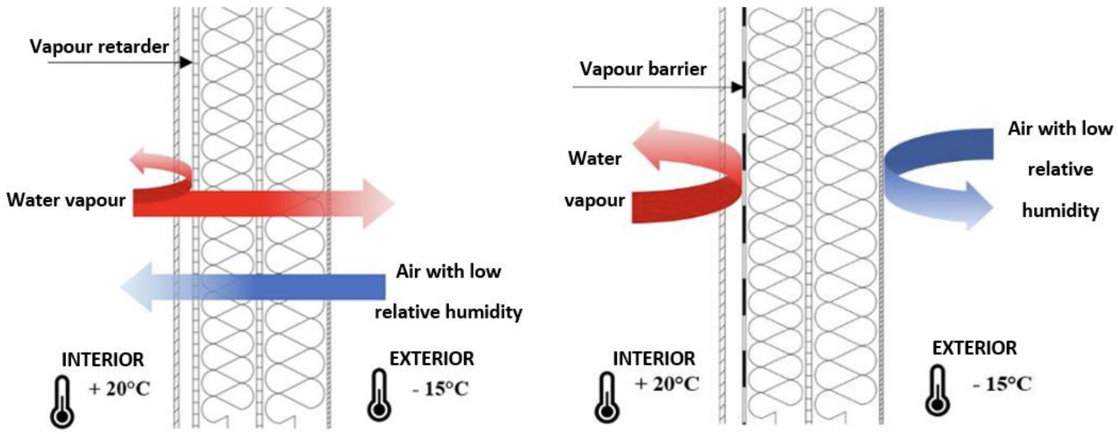
Keywords: cellulosic insulation material; bound water maximum moisture content; diffusion coefficient+.

INTRODUCTION

Water inside timber structures is undesirable from many perspectives, despite its necessity in wood and other natural and hygroscopic insulation materials (Babiak, 1990; Krieger and Srubar, 2019). The mechanical strength and modulus of elasticity of wood are reduced at higher equilibrium moisture contents (Fu *et al.*, 2022). The biological durability, dimensional stability, corrosion of steel connectors, thermal conductivity, and many other material characteristics show an undesirable trend with increasing moisture content (Zelinka *et al.*, 2014). From another perspective, the descending trend of thermal diffusivity with moisture content increases the lag time of the structure (USDA Forest Products Laboratory 2010). Specific heat and accumulation capacity of structures increase with moisture content (Szodrai and Lakatos 2017; Kotoulek *et al.* 2019). Despite some mentioned advantages for water presence in structures, the biological durability seems to be the most significant (Martín *et al.*, 2023; Thybring *et al.*, 2022) The moisture content changes significantly in wooden structures in real situations in contrary to set laboratory conditions (Jaskowska-Lemańska, and Przesmycka, 2021; Brandstätter, 2024). The boundary conditions are changing with relative humidity and temperature.

Also, the initial moisture content of the material is significant, as the production process can be wet or dry. The insulation may be installed within the wall, either in a dry state or with a specified initial moisture content. The structures are composed of blown materials serving as insulation. The importance of insulation lies in its large volume or thickness and its hygroscopic character (Slimani *et al*., 2019; Viljanen and Lu, 2019). The insulation material stores and conducts water within its volume. From a biological durability perspective, it is desirable to keep the insulation material as dry as possible. The entire wall assembly, and each of its parts, should reach equilibrium as quickly as possible (Mattila, 2017), and if condensation occurs within the insulation, it should accept water within its structure as bound water. Then, bound water will be transported to the wall surface by the structure of the insulation material, because the created capillaries where free water occurs are chaotic and not straightforwardly oriented perpendicular to the wall surface. That is the reason why the diffusion coefficient and the maximum bound water moisture content of the insulation materials studied

The evaporation of water vapor from its surfaces can be substantially enhanced by the wall construction, Figure 1:



a. **b.**

Fig. 1 The examples of insulation in different structures and possible fluxes at the surfaces.

The fluxes of water inside both structures (Figure 1a, b.) are different and can be different in one structure, also. The direction of the flux depends on the boundary conditions and the instantaneous moisture content within the structure. The equilibration of two different instantaneous moisture contents within the wall structure is described by the solution of the diffusion equation (Künzel, 1995; Hagentoft, 2001). The diffusion coefficient is the quantity that represents the rate of equilibration between two different instantaneous moisture contents within a material and is defined by Fick's law (Indekeu *et al.,* 2022; Künzel and Kiessl, 1997). The diffusion coefficient information must be supplemented with average moisture content values within the insulation to provide complete information on the velocity of the flux or a description of the moisture content field within the insulation. A method for measuring the above-mentioned needs to be developed. The method will help determine diffusion coefficients not only in laboratory conditions but also in situ in timber structures. The first aim of this contribution is to develop a laboratory method for determining the diffusion coefficient using variable fluxes at the opposing surfaces. The second aim is the measurement of the maximum bound water moisture content of blown insulation material using Archimedes' principle as described by Hrčka *et* al., 2020.

MATERIALS AND METHODS

STEICOfloc is a loose-fill thermal insulation material based on recycled cellulose fibers derived primarily from post-consumer newspaper. The material is produced through mechanical shredding and fiberization, resulting in a light, open, and flexible fibrous matrix suitable for pneumatic blowing into wall cavities, ceilings, and roofs. To ensure fire safety and biological durability, the fibers are treated with minerals, most commonly boric acid (H_3BO_3) as a fire retardant and biocide, and ammonium sulfate ($(NH_4)_2SO_4)_4$ as a fire retardant. Chemically, STEICOfloc consists of approximately 85–90% cellulose $(C_6H_{10}O_5)_n$, 0–15% inorganic preservatives, and maybe a small content of lignin. From a materials science perspective, cellulose fibers exhibit high hygroscopicity, allowing the insulation to absorb and release water vapor without structural degradation temporarily. The capillary and sorption characteristics facilitate moisture redistribution within the insulation layer, contributing to a balanced hydrothermal environment in timber-frame constructions. The vapor diffusion resistance factor (μ) ranges between 1 and 2, confirming the high vapor permeability of the material (STEICO, 2022).

The recommended installation density for vertical wall cavities is 55–65 $kg{\cdot}m^{-3}$ to ensure dimensional stability and minimize settlement, while horizontal applications can be filled with lower densities, around 45 $kg{\cdot}m^{-3}$ (STEICO, 2022).

Diffusion coefficient measurement

The method of diffusion coefficient measurement is based on solution of diffusion equation (1) (Crank 1975):

$$\frac{\partial c}{\partial t} = D\frac{\partial^2 c}{\partial x^2} \quad (1)$$

Where: c is concentration defined as the ratio of mass of water to initial specimen volume, t is time, x spatial coordinate and D diffusion coefficient. The solution is derived for zero initial condition and different fluxes, as the functions of time, at both surfaces, x=0 and x=R, equations (2) and (3):

$$\left.\frac{\partial c}{\partial x}\right|_{x=0} = f_0(t) \quad (2)$$

$$-D\left.\frac{\partial c}{\partial x}\right|_{x=R} = f_R(t) \quad (3)$$

The average concentration $\bar{c}$ can be only changed by fluxes at the surfaces and its time derivative is difference between fluxes:

$$\frac{\partial \bar{c}}{\partial t} = f_0 - f_R \quad (4)$$

If concentrations at some parts of thickness are determined, then diffusion coefficient is the only one unknown parameter in the solution of diffusion equation (1):

$$\frac{\bar{c}_{int}+\bar{c}_{ext}}{2} - \bar{c} = 2\sum_{j=1}^{\infty} \frac{\sin((2j-1)\frac{\pi}{2})}{(2j-1)\frac{\pi}{2}} e^{-(2j-1)^2\frac{\pi^2}{4}\frac{Dt}{\left(\frac{R}{4}\right)^2}} \int_0^t \frac{\partial \bar{c}}{\partial \tau} e^{(2j-1)^2\frac{\pi^2}{4}\frac{D\tau}{\left(\frac{R}{4}\right)^2}} d\tau \qquad (5)$$

Where: $\bar{c}_{int}$ is average concentration of adjacent parts to interior, $\bar{c}_{ext}$ is average concentration of adjacent parts to exterior,
(2j-1)π/2 is j_{th} root of characteristic equation, R is thickness of specimen and τ integration variable.

The left side of equation (5) is known by measurement. The least square method is applied on right side of equation (5) using criterion:

$$Q(D) = \sum_{n=1}^{\infty}\left(\left(\frac{\bar{c}_{int}+\bar{c}_{ext}}{2} - \bar{c}\right)\Big|_{theor} - \left(\frac{\bar{c}_{int}+\bar{c}_{ext}}{2} - \bar{c}\right)\Big|_{exp}\right)^2 \qquad (6)$$

Where: "theor" denotes right side of equation (5) and "exp" is experimentally determined values of average concentration and concentrations of adjacent parts to interior (int) and exterior (ext):

$$\bar{c}_{int}(t) = \int_0^{\frac{R}{4}} c(x,t)dx \qquad (7)$$

$$\bar{c}_{ext}(t) = \int_{\frac{3R}{4}}^{R} c(x,t)dx \qquad (8)$$

The method of determining the diffusion coefficient must fulfil the measurement's versatility requirements, as reflected in the initial and boundary conditions used to solve the diffusion equation. The solution of the diffusion equation will serve as a key factor in determining the diffusion coefficient and the moisture content field inside the wall. The potential for water diffusion in insulating materials, such as wood, is concentration, defined as the mass of water per unit volume of the dried insulation material. The dried substance was obtained by drying specimens of the insulation material at atmospheric pressure and 105 °C in an air environment. The experimental scheme is shown in Figure 2.

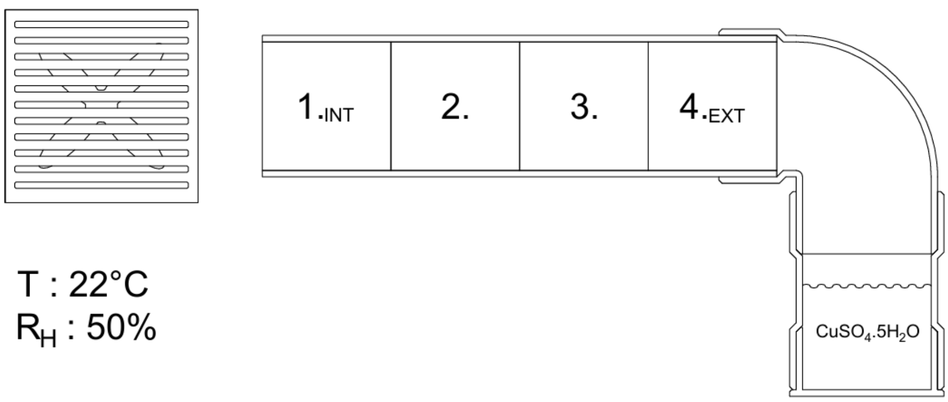


Fig. 2 The scheme of experiment.

The wall thickness was set to 40 cm. The temperature of 22 °C and humidity of 50% were set in the climatic chamber Binder KBF 720 (Tuttlingen, Germany) as interior conditions. The external conditions were exerted on a saturated aqueous solution of

$CuSO_4 \cdot 5H_2O$ at 22 °C. Mass measurements of the individual parts were performed daily for three months. Masses and times were recorded on the computer. Then, the average concentration in time was computed as the average of all four parts.

Bound water measurement

The measurement of the maximum bound water moisture content was performed using the method described by Hrčka *et al.* (2020). The method also uses the equilibrium between water and an arbitrary absorbing material, recycled cellulose fibers (STEICOfloc). The mass of the specimen was measured in water, and the apparent mass was determined. The ratio of apparent mass and oven-dried specimen is equal to the maximum bound water moisture content. The oven-dried mass was achieved in a dry-air environment at 105°C and normal pressure. The apparent density was measured at 21.5°C. The measurement was performed using the balances Radwag XA 60/220X (Radom, Poland) with the original density determination kit, and the mass of the oven-dried specimen was 40.0 mg. The maximum bound moisture content was reported when its value did not change, rounded to 3 significant digits.

RESULTS AND DISCUSSION

Equation (5) is the basis for the method of diffusion coefficient measurement. It contains only one unknown parameter – the diffusion coefficient on the right side of the equation. The left-hand side of the equation was calculated from the specimen masses and volume. Figure 3 presents the specimen moisture content change over time, and the experimental results were fitted using the method of least squares.

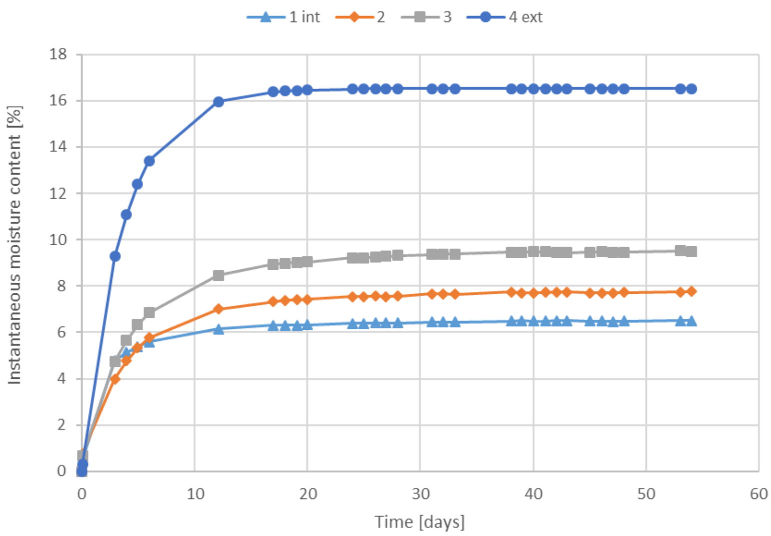


Fig. 3 Moisture contents of whole specimen four individual parts, the initial moisture content was zero.

After imbedding the oven-dried specimens in the climatic chamber, all four specimens began to increase their instantaneous moisture contents. As the experiment progressed, the rate of attaining higher instantaneous moisture content decreased. Finally, specimens' moisture contents reached stable values. As Figure 3 shows, the differences in moisture content between adjacent parts of the specimen did not reach equilibrium values. The differences could be due to non-constant flux throughout the specimen and to the material exhibiting a cosine-like instantaneous moisture content distribution in the spatial coordinate. The reason the equilibrium moisture content is taken into account does not seem valid in all parts, because it was not reached throughout the entire specimen.

The left side of the equation (5) was fulfilled in all details, because the right side contains the description of the flux evolution during the experiment. Then, the diffusion coefficient showed a decreasing trend throughout the experiment (Figure 4).

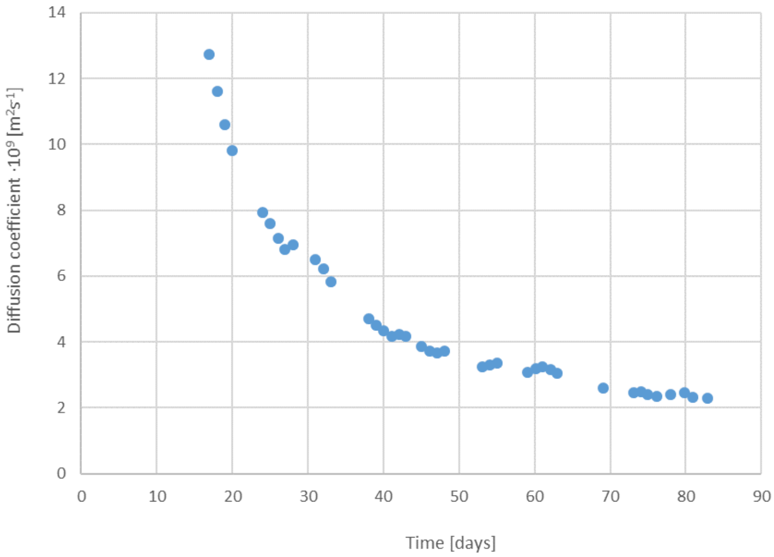


Fig. 4 The evolution of diffusion coefficient value during sorption experiment.

The curve depicted in Figure 4 showed a decreasing character and presumably reached a constant final value. It must be emphasized that the left side of equation (5), which included only measured instantaneous moisture contents and the average value of the insulating material, was fully satisfied, and diffusivity was the only unknown parameter. It is important to emphasize that equations (5) and (1) do not exclude the possibility of a variable diffusion coefficient during the experiment. Diffusivity is therefore difficult to treat as a property. It was assumed that the diffusion coefficient would reach a constant value under steady flux, that the insulation material would form a layer of water within it, and that the diffused water would move smoothly over it. If the created layer of water is modified with a versatile boundary, the diffusion coefficient will change until a new steady flux is reached.

The Nuclear Magnetic Resonance approach of Zou *et al*. (2023) was used to fit the parameters of their proposed model for the longitudinal transport diffusion coefficient of bound water in cellulose fibres. The value obtained for the diffusion coefficient was $3 \cdot 10^{-9}$ m^2s^{-1}. Thus, the longitudinal transport diffusion coefficient of bound water between cellulose microfibrils appears to be close to the self-diffusion coefficient of (bulk) water (i.e. $D = 2.3 \cdot 10^{-9}$ m^2s^{-1}) (Zou *et al.* 2023). This self-diffusion value and the diffusion coefficient of bound water in cellulose fibres in the longitudinal direction are in good agreement with the calculated values obtained by our method.

Diffusivity is not the only criterion for determining the suitability of a material for insulation. Another criterion is the bound water maximum moisture content. It was assumed that good insulation material should have high diffusivity and a high maximum moisture content within the wall structure, as shown in Figure 1b. If water vapor condenses, the insulation material must be able to diffuse the maximum amount of water possible. The results of the bound water maximum content measurement for STEICOfloc are presented in

Table 1. The initial density of the specimen inside the cuvette was 65 kg.m^{-3} in the dried state

Tab. 1 Maximum bound (B) and free (F) moisture contents of STEICOfloc measured using Archimedes' principle.

w_{Bmax} (%)	w_{Fmax} (%)
33.7	1400

The bound water maximum content value of 33.7% is comparable to the value reported by Hrčka *et al*. (2020) for cellulose, 38.9 ± 0.01%. The value is significantly lower due to the presence of some lignin in the STEICFloc insulation material.

CONCLUSION

The diffusion coefficient and the maximum moisture content of bound water are key factors for recognizing insulation materials. If condensation occurs inside a structure, the insulation material must be able to diffuse the maximum amount of water possible through the surface, which is normal to the flux in a given time interval. The STEICOfloc insulation cellulosic material exhibits a decreasing diffusion coefficient over time during sorption in constant climate conditions in laboratory conditions, at both surfaces. The maximum bound water moisture content of STEICOfloc was 33.7%. This value is almost equivalent to cellulose derived from wood. If a different insulation material is more suitable for the diffusion of water within a wall structure, the diffusion coefficient and the maximum bound water moisture content should be higher. Such an experiment must be performed with different commercially available insulation materials.

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