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Capillary pore-size distribution and equilibrium moisture content of wood determined by means of pressure plate technique

Abstract: This paper deals with the determination of the capillary pore-size distribution (CPSD) and equilibrium moisture content (EMC) of untreated and thermally modified (TM) Norway spruce [*Picea abies* (L.) Karst.] by means of the pressure plate technique (PPT). Desorption experiments were conducted at very high values of relative humidity (RH) in the range between 99.2% and 100%. The thermal modification of spruce results in an alteration of the CPSD, owing to the formation of intercellular cracks in the middle lamella, as a result of cell-wall compression. The desorption curves for both untreated and TM spruce show an extremely upward bend at 99.97% RH. This step reflects an EMC of 38.1% for untreated spruce and 33.8% for TM spruce. None of the samples shrunk during the PPT measurements. Following desorption experiments at 97.4% RH, all samples shrunk. This step reflects an EMC of 27.9% for untreated spruce and 21.7% for TM spruce.

Keywords: capillary pore-size distribution (CPSD), equilibrium moisture content (EMC), fiber saturation point (FSP), high relative humidity (RH), pressure plate technique (PPT), thermally modified wood

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Introduction

Wood is a porous, anisotropic, inhomogeneous, and hygroscopic biopolymer. All mass transport processes in wood – such as diffusion, capillary fluid movements, and transport of dissolved or colloidal substances – depend

not only on the sorption behavior but also on the porosity and pore-size distribution (PSD). The pores are categorized and termed corresponding to their dimensions. Griffin (1977) and Thygesen et al. (2010) classified the voids of wood as (1) macrovoids (lumina between 10 and 400 μm or greater), (2) microvoids (pointed ends of lumens, pit apertures, pit-membrane voids, and other small voids with diameters between 10 and 5 μm), and (3) nanovoids (pores in the cell wall with diameters <10 nm).

There are few methods for the quantitative determination of the PSD. Mercury intrusion porosimetry is one of them (Pfriem et al. 2009; Plötze and Niemz 2011; Zauer et al. 2014b). Differential scanning calorimetry allows statements about the PSD in the “nanodiameter range” between 4 and 400 nm and the fiber saturation point (FSP) (Fahlén and Salmén 2005; Repellin and Guyonnet 2005; Zauer et al. 2014a). The pressure plate technique (PPT), also known as the suction technique, is useful for studying the desorption isotherms at very high levels of relative humidity (RH) and PSD (Thygesen et al. 2010) in the wet state. Figure 1 presents an overview of the pore-size classification according to IUPAC and Griffin (1977) or Thygesen et al. (2010). Here, the diameter ranges of the individual wooden cells were taken from the works of Bosshard (1974) and Wagenführ (1999) as well as the three quoted methods for the determination of the PSD. It is obvious that the classification according to Griffin (1977) and Thygesen et al. (2010) complied better with the wood anatomy than the IUPAC definition of pore classes.

The relationship between the equilibrium moisture content (EMC) and RH in the hygroscopic range is usually investigated by equilibrating wood samples in a climate chamber or in a desiccator over saturated salt solutions. However, the components exposed to rain (e.g., decking boards and claddings) reach moisture contents above FSP. Hence, it is of high interest to study the relationship between the EMC and RH of wood in the range of high RH levels close to 100%, which is possible by means of PPT as mentioned above.

PPT works as follows: Water-saturated samples are exposed to a certain pressure in a sealed pressure vessel (extractor) while they are in hydraulic contact

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Figure 1: Overview of the different defined pore classifications.

(1) IUPAC and (2) Griffin (1977) and Thygesen et al. (2010), diameter ranges of the individual wooden cells (Bosshard 1974; Wagenführ 1999), and measurement methods for the determination of the PSD in different scale levels. HW, hardwood; SW, softwood.

with atmospheric pressure outside of the vessel trough water-saturated porous ceramic disc having small pores (Figure 2). Depending on the applied pressure inside of the vessel, water will flow out of the samples until equilibrium is reached. RH and pore diameter correspond to the applied pressure by combining the Kelvin and Laplace equations (Dullien 1992) and can thus be calculated. However, it must be noted that this formula is only applicable for stiff, partial water-filled, porous materials (Thygesen et al. 2010), in which the PSD is independent of the moisture content. A detailed description of this technique is given by Cloutier and Fortin (1991), Tremblay et al. (1996), and DIN ISO 11274 (2014).

Stone and Scallan (1967) were the first to study wood by means of PPT. The investigations were conducted on black spruce (*Picea mariana*) from the water-saturated state up to 99% RH in four equilibrium steps. At 99.75% RH, the authors observed an upward bend of the sorption

isotherm. The corresponding EMC was 40%, which was taken as FSP. Cloutier and Fortin (1991) investigated the relationship between the EMC and matrix potential (MP) of the sapwood of aspen (*Populus tremula*) as a function of the temperature by means of PPT in combination of pressure membrane technique (PMT) and different saturated salt solutions. Tremblay et al. (1996) did the same with sapwood of red pine (*Pinus resinosa* Ait.). In each study, three steps were applied for the PPT experiments. The results show that the MP increases with temperature at the given moisture content. Cloutier and Fortin (1991) reported that desorptions in tangential direction were slightly higher than those in radial direction. Tremblay et al. (1996) presented the effective cumulative and log-differential PSD inferred from the relationship between the EMC and MP, and for red pine, a maximum of a diameter of 0.4 μm was found and assigned to the pit-membrane openings. Almeida and Hernández (2006a,b) applied PMT combined with dimensional measurements for the investigation of desorption behavior of yellow birch (*Betula alleghaniensis*), American beech (*Fagus grandifolia*), congona (*Brosimum alicastrum*), and cachimbo (*Cariniana domestica*) between 99.99% and 96.43% RH. The results showed that a radial, tangential, and volumetric shrinkage occurred at the EMC between 40% for congona and 56% for cachimbo. Thygesen et al. (2010) determined the desorption isotherms of untreated, acetylated, and furfurylated Norway spruce [*Picea abies* (L.) Karst.] by means of

Figure 2: Scheme the principle of the PPT.

PPT between 95.9% and 99.9% RH and saturated salt solutions (91.9%, 94.1%, and 97.7%) always at 20°C. Here, the isotherms of the untreated and acetylated samples did not show an increasing trend with increasing RH. The conclusion was that capillary condensation does not play a significant role for water sorption in wood below FSP. Wang et al. (2014) determined the EMC for red pine (*P. resinosa* Ait.) by means of PPT between 96.9% and 99.9% RH and compared their results with reported EMC data of other studies. A high variability of the test results was found under high RH conditions, although the same method (PPT) was applied. Their results showed an extremely upward bend of the desorption curve at 99.6% RH, which corresponded to an EMC of 29.5%.

All quoted investigations demonstrate the applicability of PPT for the investigation of the EMC, water potential, or MP at high values of RH. It is also shown that, under certain assumptions, the pore diameter corresponds with the atmospheric pressure or RH. However, the focus of the mentioned studies was not the determination of the capillary PSD (CPSD), except Tremblay et al. (1996). Mostly, only a few pressure steps were applied (Stone and Scallan 1967) or the start pressure steps were small (Thygesen et al. 2010; Wang et al. 2014) and the measurement steps were too large (Almeida and Hernández 2006a,b). Under these conditions, a complete or realistic PSD and desorption curves are difficult to obtain.

In focus of the present paper was thermally modified (TM) wood because of its practical and commercial importance. The porosity, PSD, and sorption behavior of TM wood is essentially modified (Junghans et al. 2005; Pfriem et al. 2009, 2010; Zauer et al. 2014a,b) due to the degradation and modification of the cell-wall components (Fengel and Wegener 2003; Windeisen et al. 2007; Borrega et al. 2013; Olek and Bonarski 2014). Although this subject was extensively studied, there are still gaps in our knowledge concerning the PSD of TM wood. This gap should be closed by gathering further insights into the CPSD of untreated and TM wood by means of PPT under optimal conditions. Additional attention was given to the relationship between the EMC and RH at very high values of RH between 99.9% and ≈100%.

Materials and methods

The heartwood of untreated and TM Norway spruce [*P. abies* (L.) Karst.] were investigated. All wood samples were collected from the same annual rings (matched). Thermal modification was performed under N₂ atmosphere at 200°C for 4 h at a lab scale. Dimension of the samples: 2×30×30 mm³ (L×R×T). The listed geometries were

obtained after climatization at 20°C and 55% RH. All specimens were dried at 80°C to determine the oven-dried mass. Subsequently, the samples were stored over silica gel and then successively conditioned (as indicated) before being stored in demineralized water until the water saturation was reached. The water saturation was finished when the samples sunk to the bottom of the storing vessel.

For the PPT, two extractors from Soilmoisture (Santa Barbara, CA, USA) were available and designed for pressures up to 15 bar. Different ceramic plates (0.5, 1.0, 3.0, 5.0, and 15.0 bar) were used for the individual applied pressure steps. Wet cellulose-filter paper (Whatman grade 1, D=5 mm) ensured a good hydraulic contact between the specimens and the porous ceramic plates, contrary to the conventional wet clay (kaolin) layers. A wet cellulose cloth over the samples ensured an evaporation protection at each measurement period.

Both the mass (precision: 0.1 mg) and the geometry (precision: 0.01 mm) of the sample were determined after each pressure step. The pressure steps were chosen to create RH in very small intervals between 99.2% and ≈100%. The duration of each pressure step was 6 weeks, taking care that no water outflow occurred. The RH conditions were established in a desiccator over concentrated salt solutions [97.4% RH (K₂SO₄) and 94.3% RH (KNO₃)] according to ISO 12571 (2013) during 6 months at 25°C.

The capillary pore diameter d_p was determined by Equation (1):

$$d_p = \frac{-4\gamma \cos\theta}{p} \quad (1)$$

where γ is the surface tension, θ is the contact angle (CA), and p is the applied pressure at each step. The surface tension (γ) at 25°C was 72.0 mN m⁻¹ (Thygesen and Hansen 2007) and the CA (θ) was 0° (Scheffler 2008), assuming a complete wetting. The cumulative and log-differential CPSDs were calculated as a function of the mass loss of the samples based on their oven-dried mass using a density of water of 1000 kg m⁻³.

Table 1 summarizes the essential parameters and results.

Table 1: Comparison of the used pressure steps or used saturated salt solutions, the corresponding or resulting RH and capillary diameter, and the determined average EMC of untreated and TM spruce.

Pressure step (hPa)	Salt solution	RH (%)	Capillary diameter (μm)	EMC (%)	
				Untreated spruce	TM spruce
30		99.998	96.00	114.22	98.20
50		99.996	57.60	109.72	95.09
145		99.989	19.86	79.83	77.63
285		99.979	10.11	55.87	53.45
385		99.972	7.48	38.09	33.79
665		99.952	4.33	36.13	31.20
1000		99.927	2.88	33.09	26.83
1400		99.898	2.06	32.63	26.67
2800		99.797	1.03	32.50	26.54
3800		99.724	0.76	32.00	26.19
6600		99.521	0.44	31.89	26.08
11 000		99.203	0.26	31.62	25.71
	K ₂ SO ₄	97.4		27.90	21.72
	KNO ₃	94.3		22.54	19.47

Results and discussion

The coefficients of variation of all following data are for the reference (maximum of 2.5%) and TM wood (maximum of 4.5%) satisfactory because matched samples were tested. The average oven-dried densities were 0.42 and 0.43 g cm⁻³ for the reference and TM wood, respectively. In Figure 3a, the average cumulative (on the left y-axis) and log-differential CPSDs are presented. The latter data are unimodal, which is based on the gradual transition from early-wood (EW) to latewood (LW) tracheids in softwood contrary to hardwoods, which are diffuse or ring porous and show always a bimodal characteristic owing to vessels with a large lumen and the fiber tracheids and libriform fibers with small lumina. The capillary pores in spruce (Figure 3a) with a diameter of approximately 16 µm represent primarily the EW tracheids and those below 16 µm represent the LW tracheids (Figure 1) and further the pointed ends of the tracheids and pit chambers (Thygesen et al. 2010). The log-differential CPSD curves of spruce and TM spruce have different gradients, although their

pore diameter maxima are very similar (approximately 7–8 µm). The proportion of capillary pores above 10 µm is significantly lower in TM spruce as a consequence of cell-wall compression during thermal modification procedure (Zauer et al. 2013). This narrows the lumen of the tracheids. Furthermore, cracks emerged particularly in the LW cell wall (Fengel 1966) or in the intercellular spaces in the middle lamella (ML), leading to a reduced proportion of higher pore diameters. Especially, the higher proportion of intercellular spaces in the ML of TM spruce results in an increased capillary water uptake in longitudinal direction via the end grain compared with the reference as a consequence of the higher capillary forces in tubes with lower diameter. Pfriem (2011) found in longitudinal direction a 30% higher capillary water absorption coefficient of TM spruce compared to the untreated one.

Figure 3b shows the average cumulative and log-differential CPSD of red pine and aspen sapwood based on the desorption data of Cloutier and Fortin (1991) as well as Tremblay et al. (1996), measured by means of PPT and PMT at 18°C. The log-differential distribution curves show a unimodal characteristic for red pine and a bimodal one for aspen sapwood, probably because aspen has diffuse-porous-containing vessels and fiber tracheids or libriform fibers. The pore diameter between 40 and 140 µm is equivalent to the amount of the vessels, and the diameters below 10 µm reflect the fibers (Figure 1). The highest peak is at approximately 0.7–0.8 µm and reflects primarily the pointed ends of the fibers and the pit chambers. The latter is the most significant in the vessel walls (Wagenführ 2007). The CPSD curves (Figure 3b, cumulative and log-differential) of red pine are indicative of pores between 20 and 140 µm (EW tracheids) and between 6 and 20 µm (LW tracheids or the pit openings). The pore diameters below 6 µm represent the pointed ends of the tracheids or pit chambers and the pit-membrane openings in the margo (Figure 1). The highest peak at approximately 0.7–0.8 µm reflects the pointed end of the tracheids and the pit chambers.

As expected, the EMCs of untreated spruce are higher than the EMCs of TM spruce at a certain RH (Table 1). Furthermore, it is obvious that these are much higher at the first two pressure steps compared to the following steps. This is the consequence of the reduced pore volume of TM spruce owing to cell wall shrinking, which leads to less water absorption compared to the untreated spruce. The log-differential CPSD of untreated spruce displays a significantly higher proportion of cells with diameters between 20 and 100 µm. Accordingly, in the first two PPT steps, the EMC of spruce is considerably higher than that of TM spruce. At the following measurement steps, the

Figure 3: Average capillary cumulative and log-differential PSD of (a) untreated and TM spruce to be studied as well as (b) red pine and aspen sapwood based on the desorption data of Cloutier and Fortin (1991) and Tremblay et al. (1996).

pore-size-related data between the two spruce groups are small.

Figure 4 shows for both series an extreme upward bend at 99.97% RH. The determined average EMC at this stage of desorption was 38.09% for spruce and 33.79% for TM spruce. The corresponding capillary pore diameter of the applied pressure is 748 μm , and it is a micropore according to Griffin (1977) and Thygesen et al. (2010). The pore diameters above 748 μm are equivalent to the amount of the EW tracheids and the main proportion of the LW tracheids (Figure 1). The pore diameter below 748 μm reflects especially the pointed ends of the EW or LW tracheids, the pit chambers, the pit-membrane voids, and the interfibrillar spaces (Figure 1). Thus, the extremely upward bend of the desorption curve reflects primarily capillary free water in the lumen of the EW and LW tracheids. It is obvious that, between 99.97% and 99.20% RH, the EMC decreases with decreasing RH (Table 1), but an extremely upward bend in this range cannot be observed and confirms the results of Thygesen et al. (2010). The water in these pores (diameter: 748–0.26 μm) is retained owing to the higher suction tension as a consequence of the capillary effects of these pore sizes in the pointed ends of all cell elements and pit-membrane voids. To remove this water, a higher atmospheric pressure is necessary, that is, the determined EMC includes water vapor and capillary water at these stages of desorption.

Stone and Scallan (1967) determined a point of inflection of desorption isotherm of black spruce (*P. mariana*) at 99.75% RH, equivalent of a pore diameter of 0.8 μm . The corresponding EMC of 40% was taken as being FSP. However, the inflection point is difficult to identify with high precision due to the low number of pressure steps.

Figure 4: EMC as a function of RH between 99.9% and $\approx 100\%$ of untreated and TM spruce.

Griffin (1977) tried to correct the inflection point of Stone and Scallan (1967) by plotting the EMC against MP. Finally, the inflection point was at $\approx 99.99\%$ RH, equivalent of a pore diameter of 3.0 μm . In this case, the lumen of all EW and LW tracheids is empty and the remaining capillary water is in the pointed ends of the tracheids and pit chambers as well as in the pit-membrane voids in margo and interfibrillar spaces (Figure 1). The corrected EMC (water vapor and capillary water) was 43%. Wang et al. (2014) found an extremely upward bend for red pine already at 99.63% RH and determined an EMC of 29.5%. However, the determined EMC for the next three RHs seems to be too large. For the next point of the desorption curve of 99.80% RH, they determined an EMC of 51.2%, whereas, for the last point of 99.93% RH, the EMC was 96.4%. In our study on spruce, the EMC was 32.50% at 99.8% RH and 33.09% at 99.93% RH. The time taken for the samples to reach equilibrium in the PPT apparatus varied in the study of Wang et al. (2014) from 6 to 75 days. A detailed information of equilibrium time of each individual pressure step is not given. The dimensions of the applied samples were $40 \times 40 \times 8 \text{ mm}^3$ (L). We believe that, during the 6 days' storage time in the first pressure step (from 100% to 99.93% RH), the EMC cannot be achieved for the whole sample.

At all PPT measurement steps in the present study from $\approx 100\%$ down to 99.2% RH, no tangential and radial shrinkage of the samples could be found. It can be assumed that no internal changes of the pore structure occurred, that is, no falsifying influences occurred for pore diameter calculation by Equation (1). The determined EMC at 99.20% RH was 31.62% for spruce and 25.71% for TM spruce. The corresponding pore diameter of the applied pressure is 0.26 μm and reflects mainly the pointed ends of the cells and also the pit-membrane openings (Figure 1). Figure 5 shows the scanning electron microscopy (SEM) images of a bordered pit of spruce (Figure 5a) and a higher resolution of its membrane closed to the pointed end of the pit chamber (Figure 5b). The pit membrane (margo) of a bordered pit in softwood consists of radially directed and intersecting cellulose fibrils, which has a porous structure. The thicknesses of the membranes are between 0.1 and 0.5 μm (Wagenführ 1999; Rosenthal et al. 2010) and the diameters of the capillary openings in the membranes are between 0.01 and 0.7 μm (Liese and Fahrenbrock 1952; Stamm 1967; Wagenführ 1999), where even pore diameters up to 4.0 μm are possible (Siau 1995). The pit membranes are degraded owing to thermal modification, so that there are essentially less small voids and the TM pit membrane gets larger pores compared to the reference (Zauer et al. 2014a). That means that, during thermal modification, not

Figure 5: (a) SEM image of a bordered pit of spruce and (b) a higher resolution of its membrane closed to the pointed end of the pit chamber.

only the water vapor is diminished (Pfriem et al. 2010) but also the capillary water. This contributes essentially to the improved biological durability of TM spruce.

At 97.4% RH, there is some shrinkage perceptible (EMCs 27.90% and 21.72% for spruce and TM spruce, respectively). The average shrinking in the tangential direction was 0.56% (spruce) and 0.63% (TM spruce), whereas the corresponding average shrinkage in the radial direction was 0.16% and 0.23%, respectively. That means that shrinkage occurred between the EMCs of 31.62% to 27.90% (spruce) and 25.71% to 21.72% (TM spruce). Almeida and Hernández (2006a,b) found no shrinkage up to 99.49% RH by means of PMT for American beech (*F. grandifolia*), but a first shrinkage was at the next step at 98.56% RH, equivalent to a pore diameter of 0.144 µm. This pore diameter reflects primarily voids in the pit membrane (margo) of the hardwood pits (Figure 1) and the pointed ends of the hardwood fibers and pit chambers. The determined EMC at this stage of desorption was 40.2% and thus significantly higher than the EMC of our study.

The average desorption curve shows at 99.97% RH an extremely upward bend. On the contrary, the samples display the first radial and tangential shrinkage between 99.2% and 97.4% RH. This raises the question where the FSP is, which is critically discussed by Engelund et al. (2013). The point of upward bend determined in our study does not reflect the FSP, because the lumens are not empty. This also applies for the investigation of Stone and Scallan (1967). The fiber saturation range between 32% and 28% for spruce and 26% and 22% for TM spruce is important, when the samples begin to shrink. Tiemann (1906) defined FSP as the moisture content when strength begins to increase. In our opinion, both phenomena occur virtually at the same time. That means that the strength increase is the point where the cell wall is shrinking,

because the fibrils approach each other and the shear forces between the individual microfibrils can be better transferred. Skaar (1988) presented the idealized behavior of several mechanical properties of untreated wood as a function of the EMC and demonstrated that the mechanical properties are independent from the EMC above 25%, which is termed as FSP.

Conclusions

The desorption experiments at 25°C of water-saturated untreated and TM Norway spruce were conducted in the 94.3% to ≈100% RH range by means of PPT and saturated salt solutions for the determination of the CPSD and the relationship between RH at very high values and the EMC. TM spruce under nitrogen atmosphere at 200°C for 4 h changes the CPSD and lowers the EMC. During thermal modification, the proportion of capillary pores decreases above a diameter of 10 µm. The cell wall is compressed and the lumens are narrowed and intercellular cracks occur in the ML. The desorption curves show an extremely upward bend at 99.97% RH, equivalent of an EMC of 38.1% for spruce and 33.8% for TM spruce, where the average pore diameter is 748 µm. That means that the upward bend includes the capillary free water in the lumen. Accordingly, the EMCs between 38.1% and 33.8% do not represent the FSP. Based on our study, the FSP is between 32% and 28% (spruce) and 26% and 22% (TM spruce), where the samples show the first shrinkage.

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