Effect of Extractives on the Equilibrium Moisture Content and Shrinkage of Selected Tropical Wood Species

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The main objective of this research was to investigate tropical wood sorptive properties. For selected tropical wood species (courbaril, ipe, light red meranti, merbau, tatajuba, and teak), the equilibrium moisture content was determined at 20 °C and 9, 30, 55, 70, and 97% relative humidity. The experimentally determined values were analysed using the Hailwood-Horrobin sorption model to compute the fibre saturation point and mono- and multi-layer sorption. There were significant differences in the sorption behaviour of different wood species. Generally, the fibre saturation point of tropical wood species is lower than in wood species from moderate climate zones. The lowest values of fibre saturation point were found for ipe (18.7%), courbaril (20.4%), and tatajuba (20.5%). Furthermore, chloroform-ethanol extractives content was correlated with multilayer sorption and the fibre saturation point, such that a higher content of chloroform-ethanol extractives was associated with a lower equilibrium moisture content. Therefore, chemisorption was not influenced by chloroform-ethanol extractives. Ethanol extracts showed an influence on monomolecular-bound water.

Keywords: Tropical wood; EMC; Fibre saturation point; Wood extractives

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INTRODUCTION

Wood is a hygroscopic material that gains or loses moisture by variation in hygrothermic conditions. The equilibrium moisture content (EMC) of the wood is more important when it is left in conditions of relative humidity and constant temperature (Martins 1988; Popper *et al.* 2006, 2007, 2009; Hashemi and Latibari 2011). Combined with the fact that the wood has a high content of water, these properties hamper one of the main activities of wood processing, which is drying, especially for the sector that uses solid wood as a raw material (Martins 1988; Oliveira *et al.* 2005). Determination of the physical and mechanical behaviour of wood as a hygroscopic material is very useful in timber drying, conversion, and timber utilization. This knowledge is extremely important and prevents unpredictable and unexpected consequences during the exploitation of wooden products, such as floors, furniture, *etc.* (Sahri *et al.* 1998).

While the sorptive properties of wood have been explored in detail, knowledge in this field is incomplete, mainly because a number of new wood species have been introduced into the markets in recent years. Tropical wood and wood composites are being machined in increasing quantities in many countries for building construction and decorative purposes. As tropical woods and wood composites are used extensively in

many products, their properties are becoming more important (Darmawan *et al.* 2012). To comparatively evaluate the sorption behavior of many wood species, the sorption isotherm of fir wood is usually used (Popper *et al.* 2006). However, Keylwerth (1969) found significant differences between the sorption behaviors of different wood species. Also, the equilibrium moisture content of wood varies within a relatively broad range. This finding has been confirmed many times (Hernández 1993, 2007; Sahri *et al.* 1998; Popper *et al.* 2006, 2007).

Some wood constituents can be extracted with organic liquids such as ethanol, acetone, *etc*., and the extractive components include resin acids, fats, terpenes, lignans, flavonoids, and tannins. (Mantanis *et al.* 1995; Nzokou and Kamdem 2004; Hernández 2007). The content and composition of extractives vary greatly among different wood species and also within different parts of the same tree. Extractives are not structural components of wood. In general, the overall percentage of extractives in wood varies from 2 to 10%, with the exception of some tropical wood species (20 to 25%).

Extractives have profound effects on many wood properties with technological interest, including wood-water relations. Generally, wood extractives affect the sorption properties of wood (Nzokou and Kamdem 2004; Popper *et al.* 2006, Hernández 2007; Hashemi and Latibari 2011). Given their variable physical and chemical characteristics, these substances exhibit hygroscopic, hydrophobic, or neutral behaviors. The removal of extractives increases the dimensional changes in wood (Choong and Achmadi 1991; Militz and Homan 1993; Mantanis *et al.* 1994, 1995; Adamopoulos and Voulgaridis 2012). Mantanis *et al.* (1994) studied the effect of extractives on the swelling-shrinkage of spruce, maple, and aspen and determined activation energies for the swelling of extracted wood in water. The rate of swelling in water considerably increased after the removal of extractives, and the maximum tangential swelling also generally increased.

Because most studies either concentrate on examining wood types from moderate climate zones or investigate surface discoloration in a small number of tropical wood species, these studies exhibit little variation (Themelin 1998; Popper *et al.* 2006, 2007, 2009; Hashemi and Latibari 2011). Some tropical woods have high extractives content, but little is known about how these extractives affect the hygroscopicity of wood. According to Hernández (2007), extraction with organic solvents is the most suitable method for evaluating the effect of extractives on the sorption behavior of tropical hardwoods. In these studies, the cyclohexane-extracted fraction exerted little effect on the moisture content, but the acetone fraction of wood extractives greatly affected moisture content and the hygroscopic stability of tropical hardwoods.

Extractions are usually conducted using various combinations of solvents, temperatures, and exposure times (Choong and Achmadi 1991; Militz and Homan 1993; Mantanis *et al.* 1994, 1995; Popper *et al.* 2006, 2007; Hashemi and Latibari 2011; Adamopoulos and Voulgaridis 2012). Thus, limited specific information exists regarding the different functions of wood extractives in controlling the moisture content of wood. Similar studies using more species and focusing on the location and distribution of these substances within wood are required. This information would elucidate the effects of these substances on moisture sorption and other wood properties.

Although there have been studies on the physical and mechanical properties of some tropical woods, very limited data have been obtained for chemical composition (Kilic and Niemz 2012). Furthermore, the effect of extraction content on the water sorption of tropical wood species has not been investigated in depth.

In the present work, the sorptive properties of tropical wood species from Asia and South America were examined. The equilibrium moisture contents at 20 °C and variable relative humidity were determined, and the constants were computed according to the Hailwood and Horrobin model (1946). The main objective of the study was to determine the influence of extractives on the equilibrium moisture content and dimensional stability. The importance of wood density was also examined.

EXPERIMENTAL

Materials

The wood species used in this study are described in Table 1. These species were selected in order to have a representative sample of hardwood, with a wide range of density, types of amounts of extraneous substances, and different anatomical structures. European beech wood was used as a reference. All test materials were heartwood because it is more important commercially than sapwood. Wood from each wood species was acquired from DLH Poland, Warsaw, Poland. Material was identified in the laboratory using macroscopic techniques.

Table 1. Wood Species Used

Wood Name	Latin Name	Plant Family	Origin	Special Features
Tatajuba	*Bagassa guianensis* Aubl.	Moraceae	South America (Brazil)	Irregular fibres arrangement
Jatoba	*Hymenea courbaril* L.	Fabaceae	South America (Brazil)	Irregular fibres arrangement, axial parenchyma in narrow bands
Merbau	*Intsia* sp.	Fabaceae	Asia (Burma)	Irregular fibres arrangement
European beech	*Fagus sylvatica* L.	Fagaceae	Europe (Poland)	Wide wooden rays
Light red meranti	*Shorea* sp.	Dipterocarpaceae	Asia (Indonesia)	Irregular fibres arrangement, axial parenchyma in narrow bands
Ipe	*Tabebuia* sp.	Bignoniaceae	South America (Brazil)	Irregular fibres arrangement
Teak	*Tectona grandis* L.	Lamiaceae	Asia (Burma)	Axial parenchyma in narrow bands

Wood samples were prepared for investigation and analyses by using standard methods. Identical samples of each wood species were collected from one log; thus, differences in tested properties caused by differences in wood anatomy were avoided. Each part was quarter-sawn to produce planks of 4 cm thickness. Then they were conditioned to air-dry in a room with relative humidity of 60 % and temperature of 21 °C for nine months prior to testing. The defect-free planks were sawn and sized to samples

for moisture sorption and dimensional stability testing. Each sample had dimensions of 30 mm (tangential) × 30 mm (radial) × 5 mm (longitudinal).

For chemical investigation wood samples were ground in a laboratory mill and sieved. The fraction which passed through a 0.63 mm sieve was used. Ten samples were used per each test.

Methods

Sorption analysis

The specimens were exposed to a moisture sorption test (adsorption) consisting of oven-drying, conditioning at five different relative humidities ranging from 9 to 97%. As soon as each point of sorption was completed, the mass of the specimens was measured to the nearest 0.001 g, and their dimensions were taken to the nearest 0.01 mm. The conditioning of specimens to appropriate moisture content was possible with the use of sealed enclosures in which prescribed saturated salt solutions were placed at a temperature close to 20 °C (Table 2). The relative humidity was monitored and recorded using a hygrometer (AZ type 9871, AZ Instrument Corp., Taichung City, Taiwan). A criterion for equilibrium was established as three successive identical mass readings at 24-h intervals. The test results were used to obtain isotherm curves. For each wood species, 100 samples were used.

Table 2. Relative Humidity of Air at a Constant Temperature 20 ± 2 °C Obtained in Sealed Enclosures with the Use of Saturated Salt Solutions

Saturated Salt Solution	Relative Humidity (%) at 20 ± 2 °C
Potassium hydroxide, KOH	9
Magnesium chloride, MgCl	30
Sodium bromide, NaBr	55
Sodium chloride, NaCl	70
Potassium sulfate, K_2SO_4	97

Sorption experimental data were fitted with the Hailwood-Horrobin (H-H) single hydrate model (1946). The H-H model divides total water sorbed (U_{FS}) into its monomolecular (U_m) and multi-layer (poly-molecular) (U_p) components. These relationships are shown in Eq. 1,

$$U_{FS} = U_m + U_p$$

$$U_{FS} = \frac{1800}{M_p} \cdot \left(\frac{\alpha \cdot \beta \cdot h}{1+\alpha \cdot \beta \cdot h}\right) + \frac{1800}{M_p} \cdot \left(\frac{\alpha \cdot h}{1-\alpha \cdot h}\right) \quad (1)$$

where U_{FS} is the total water adsorbed (%), U_m is the monomolecular water adsorbed (%), U_p is the multi-layer water adsorbed (%), h is the relative humidity (%), M_p is the hypothetical molecular weight of the dry wood polymer, α is the equilibrium constant of the hydrated wood, and β is the equilibrium constant of the non-hydrated wood.

Swelling and density determination

Prior to swelling tests, wood samples were oven-dried for 24 h and then allowed to cool to ambient temperature in a desiccator. The wood block was quickly transferred from the desiccator into a flat-bottom plastic container, which was placed in an indicator

with an accuracy of 0.001 mm. The extension rod with a flat contact point had close contact with the sample, which applied a relatively small force on its surface in the measuring direction and prevented the samples from floating in the water. The indicator was set to zero, and the distilled water was poured into the plastic container until the wood samples were immersed. The data of 10 replicates were collected separately in both radial and tangential directions. Swelling (*S*) was calculated as the percentage of oven-dry dimension,

$$S = \frac{L_w - L_0}{L_0} \cdot 100 \quad (2)$$

where L_w is the swollen dimension (mm), and L_0 is the oven-dry dimension (mm).

Samples for swelling measurements were used for basic density determination, which was done by the ASTM D2395 (2002).

Extraction

Wood powder (flour) was extracted according to ASTM D1110-84 (2013), and ASTM 1107-96 (2013). Extraction was done with Soxhlet apparatus using a mixture of chloroform and ethanol (93:7 v/v) and two solvents, ethanol and hot water. The mixture of chloroform-ethanol was used as an ethanol-toluene substitution based on Antczak *et al.* (2006). All chemicals were analytical reagent grade products of Chempur, Poland.

Wood powder was placed in the extraction apparatus and successively extracted for 10 h. The solvents were evaporated to a viscous solution in a rotary evaporator and then dried to produce powder.

Statistical procedure

Statistical analysis of the test results was carried out using Statistica v. 10 software (StatSoft, Inc., Tulsa, OK, USA). Data were analyzed and provided as the mean ± standard deviation, the median, scatter plot of results around the median, and minimum and maximum values. Regression analysis was used to evaluate relationships between the measured properties. The effects of different concentrations of extractives on moisture content and dimensional stability were determined.

RESULTS AND DISCUSSION

Sorption Analysis

The average values of the measured equilibrium moistures for adsorption at 20 °C are shown in Table 3.

Table 3. Equilibrium Moisture Content

Wood Species	Equilibrium Moisture Content (%) at Relative Humidity				
	9%	30%	55%	70%	97%
Bagassa guianensis Aubl.	3.3	7.0	9.8	11.5	17.3
Fagus sylvatica L.	3.0	7.5	11.8	12.9	24.0
Hymenea courbaril L.	3.3	6.5	9.6	11.4	18.6
Intsia sp.	4.2	8.0	11.8	13.5	17.9
Shorea sp.	3.5	6.8	11.1	13.5	23.8
Tabebuia sp.	3.0	7.7	10.5	11.6	17.1

Tectona grandis L.	3.0	6.1	9.2	11.0	15.8

There were very large differences in the sorption behavior of different species. The highest differences in moisture content (MC) occurred in higher humidity (approximately 97%). The very low fiber saturation (U_{FS}) points of courbaril (20.4%), ipe (18.7%), and tatajuba (20.5%) (Table 4) were clearly below the values of wood from a temperate climate zone (European beech) and tropical wood species used in the tests.

Table 4. HH-Model Sorption Analysis of Selected Tropical Wood Species and European Beech at the Fiber Saturation Point

Wood Species	Sorption (%)		
	U_m	U_p	U_{FS}
Bagassa guianensis Aubl.	4.6	15.9	20.5
Fagus sylvatica L.	4.8	26.2	31.0
Hymenea courbaril L.	4.5	15.9	20.4
Intsia sp.	5.4	24.2	29.6
Shorea sp.	4.2	24.0	28.2
Tabebuia sp.	4.1	14.6	18.7
Tectona grandis L.	4.3	18.2	22.5

Table 5. Extractives in Selected Tropical Wood Species and European Beech

Wood Species	Extract (%)		
	Chloroform-Ethanol	Ethanol	Hot Water
Bagassa guianensis Aubl.	3.0	6.4	11.2
Fagus sylvatica L.	2.5	3.1	2.1
Hymenea courbaril L.	6.0	8.0	12.0
Intsia sp.	3.0	20.5	17.3
Shorea sp.	1.8	3.1	2.9
Tabebuia sp.	7.5	9.8	12.6
Tectona grandis L.	8.7	5.8	4.3

Note: relation w/w is given

The difference between the fiber saturation point of European beech wood and tropical wood was apparently caused by the amount of chloroform-ethanol extractives in the wood structure, which exhibited hydrophobic behaviour. The extractives contents in the tested wood species are summarized in Table 5. These results are consistent with those in the literature. According to Popper *et al.* (2006), the ethanol-toluene mixture yields the same relationship in African wood species – the higher the ethanol-toluene extract content, the lower the equilibrium moisture content. Results showed that wood extractives lowered the equilibrium moisture content of wood at high relative humidity.

Based on these results, the absolutely highest monomolecular bound water was with merbau. This wood species showed the strongest affinity for the water (Table 4). Generally, the quantities of ethanol extractives were substantially greater than those obtained with a chloroform-ethanol mixture. These results can be explained by considering the polar nature of the compounds present in extractives (Reichardt 1979).

Figure 1 shows the influence of extractives on the equilibrium moisture contents U_{FS}, U_p, and U_m. A linear regression model was used to describe the relationship between U_{FS}, U_p, U_m, and extractive content. The *P*-value of the test was less than 0.01 (for U_{FS} and U_p at the 99% confidence level), indicating a statistically significant relationship between U_{FS}, U_p, and chloroform-ethanol extract at the fibre saturation point (Fig. 1a).

No relationship between chloroform-ethanol extract and U_m was found (Fig. 1a). Therefore, chemisorption was not influenced by chloroform-ethanol extractives. This finding confirmed previous results with other solvents such as ethanol-toluene (Popper *et al.* 2006). The lowest sorption values were seen in the species with the highest amount of extractives. There was also a significant relationship between ethanol extract and U_m in contrast to hot water extract (fig. 1b). However, according to Hernández (2007), extractives content has little effect on the monolayer sorption of water; however, it did have an appreciable effect on polylayer sorption. According to Adamopoulos and Voulgaridis (2012) and based on the presented results, hot water extraction had no influence on sorptive properties. This conclusion was drawn from the black locust (*Robinia pseudoacacia* L.) results, and there was almost identical sorption behaviour in unextracted and extracted specimens. Based on results, the monolayer sorption depended on ethanol extractives. Regression analysis indicated a statistically significant relationship at the 95% confidence level (the *P*-value of the linear regression analysis was less than 0.05). For this reason, information about the sorptive properties of wood cannot be generalized but must instead be determined for each species.

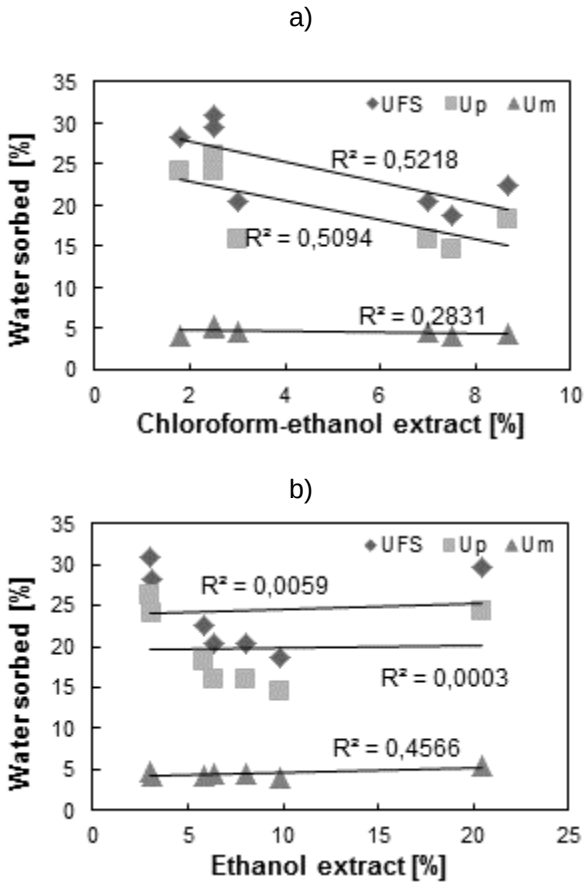

Fig. 1. Relationship between U_{FS}, U_p, U_m, and extractive content in a) a chloroform-ethanol mixture, b) ethanol

Density and Swelling

The results of regression analysis results showed that the basic density of tropical wood had no significant influence on the sorption properties of the tested wood species

(R^2 value of 0.26), but when merbau was not included within this analysis, the relationship between the fiber saturation point and density was significant. Regression analysis indicated a statistically significant relationship at the 95% confidence level (the *P*-value of the linear regression analysis was less than 0.05). This result reflected the high content of ethanol and hot water extracts with high affinity to water because of their polar character. According to Mantanis *et al.* (1994) and Adamopoulos and Voulgaridis (2012), extractives deposited in the cell wall structure have a definite influence on the swelling and shrinking of the cell wall, and therefore, would affect the swelling and shrinkage of wood as evidenced by the changes in external dimensions.

The results of the density and swelling of the tested wood species are summarized in Table 6. Wood density had an influence on the fiber saturation point during the testing of nine tropical hardwoods from Peru and sugar maple wood from Quebec (Hernández 2007). According to Babiak and Kúdela (1995), the wood density and its structure play important roles. The species tested here revealed a similar structure of diffuse-porous wood. In some (courbaril, light red meranti) but not all cases, an expanded axial parenchyma was observed.

Table 6. Density and Dimensional Changes of Selected Tropical Wood Species and European Beech

Wood Species	Density (kg/m^3)	Dimensional Changes (%)	
		Radial	Tangential
Bagassa guianensis Aubl.	900 (18.3)	4.23 (0.24)	6.24 (0.42)
Fagus sylvatica L.	640 (53.7)	6.00 (0.25)	14.09 (0.65)
Hymenea courbaril L.	950 (37.1)	4.40 (0.45)	8.61 (0.62)
Intsia sp.	871 (34.7)	4.11 (0.60)	6.12 (0.58)
Shorea sp.	515 (13.4)	3.88 (0.20)	8.87 (0.19)
Tabebuia sp.	900 (13.1)	5.31 (0.38)	6.72 (0.59)
Tectona grandis L.	600 (12.8)	2.54 (0.14)	5.66 (0.63)

Note: parentheses show the standard deviation

The observed radial shrinkage varied from 2.54% to 5.31%, and the tangential shrinkage varied from 5.66% to 8.87%. The lowest shrinkage values were observed in teak wood, which had higher chloroform-ethanol extractives contents and a relatively low content of hot water extractives (tab. 5). According to former studies it is typical for teak wood to have less polar compounds (Premrasmi and Dietrchichs 1967, Miranda *et al.* 2011).

There is a definite relationship between volumetric shrinkage (sum of radial and tangential shrinkage) and specific gravity (Choong and Achmadi 1991). In our study, the relationship was not significant ($P < 0.1$), and the correlation ($R^2 = 0.40$) was also rather low, indicating that only 40% of the variation in volumetric shrinkage was accounted for by wood density. Despite the reported relationship between density and shrinkage (Kord *et al.* 2010; Farsi and Kiaei 2014), there was no significant correlation observed in this study. In this case, the probable relationship was strongly affected by the high variability of extractives content, especially for teak. When teak was not included in this analysis, the relationship between the density and shrinkage of wood was significant (R^2 value of 0.56). But according to former studies (Mantanis *et al.* 1994, Adamopoulos and Voulgaridis 2012), it could be predicted that merbau would show relatively low

shrinking, because, as those researchers claimed, wood high in water soluble extractives is found to invariably shrink less. This is due to bulking effect of the extractives which are left deposited in the cell walls. The lowest shrinkage values were observed in teak wood, which had more chloroform-ethanol extractives and relatively low contents of hot water extractives.

CONCLUSIONS

1. The chloroform-ethanol extractives correlated with multilayer sorption and the fibre saturation point. Higher contents of chloroform-ethanol extractives were associated with lower contents of equilibrium moisture. Therefore, chemisorption was not affected by chloroform-ethanol extractives. The ethanol extracts showed an influence on monomolecular-bound water.
2. There was high variation in wood extractives and in hygroscopic stability within and among the tested wood species. Generally, the fibre saturation point of tropical wood species is lower than in the case of wood species from a moderate climate zone. Ipe wood had the lowest fibre saturation point (18.7%).
3. The lowest shrinkage values were observed in teak wood, which had more chloroform-ethanol extractives and relatively low contents of hot water extractives.
4. The high variability of extractives content strongly affected the relationship between wood density and its sorptive properties, such as the fibre saturation point and dimensional changes.

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