

ANALYSIS OF THE WATER VAPOUR SORPTION ISOTHERMS OF OIL PALM TRUNK AND RUBBERWOOD

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ZAIHAN J, HILL CAS, HASHIM WS, MOHD DAHLAN J & SUN DY. 2011. Analysis of the water vapour sorption isotherms of oil palm trunk and rubberwood. Oil palm (*Elaeis guineensis*) trunk and rubberwood (*Hevea brasiliensis*) were studied to determine their sorption isotherm behaviours using a dynamic vapour sorption (DVS) apparatus. Oil palm trunk (OPT) was separated into six parts: two cross-sectional zones (inner and outer), vertical zones (top and bottom) and three types of tissues (parenchyma + vascular bundle, parenchyma and vascular bundle). The different parts of OPT and rubberwood showed differences in equilibrium moisture content (EMC) at each target relative humidity (RH) except for those from the top and bottom. The adsorption isotherms were analysed using the Hailwood–Horrobin (H–H) model and it was found to fit well with the experimental data. The dissolution water (Ms) and the projected fibre saturation point (p-FSP) in all parts of OPT were higher than in rubberwood. However, the values of hydration water (Mh) of all parts of OPT and rubberwood were similar. In the sorption hysteresis, rubberwood was higher than OPT. This could be related to differences in stiffness of the material. The sorption behaviour of the OPT material was affected by the presence of starch.

Keywords: Equilibrium moisture content, dynamic vapour sorption, Hailwood–Horrobin, hysteresis, fibre saturation point, parenchyma, vascular bundle

ZAIHAN J, HILL CAS, HASHIM WS, MOHD DAHLAN J & SUN DY. 2011. Analisis isoterma erapan wap air bagi batang kelapa sawit dan kayu getah. Batang kelapa sawit (*Elaeis guineensis*) dan kayu getah (*Hevea brasiliensis*) dikaji untuk menentukan sifat erapan isoterma dengan menggunakan alat erapan wap dinamik (DVS). Batang kelapa sawit (OPT) dibahagi kepada enam bahagian: dua zon keratan rentas (dalam dan luar), zon tegak (atas dan bawah) dan tiga jenis tisu (parenkima + berkas vaskular, parenkima dan berkas vaskular). Setiap bahagian OPT dan kayu getah menunjukkan keseimbangan kandungan lembapan yang berbeza pada setiap kelembapan relatif kecuali di bahagian atas dan bawah. Erapan isoterma dianalisis menggunakan model Hailwood–Horrobin (H–H) dan didapati padan dengan data eksperimen. Air lapisan terlarut (Ms) dan titik tepu gentian yang diramal (p-FSP) adalah lebih tinggi pada semua bahagian OPT berbanding kayu getah. Namun, air lapisan terhidrat (Mh) pada semua bahagian OPT adalah sama dengan kayu getah. Dalam histeresis erapan, kandungan lembapan kayu getah lebih tinggi daripada OPT. Ini mungkin berkaitan dengan perbezaan sifat kekenyalan dua bahan tersebut. Sifat erapan OPT dipengaruhi oleh kandungan kanji.

INTRODUCTION

Oil palms are planted for oil production and have a life span of between 25 and 30 years. Currently, oil palm trunks (OPTs) are left to rot or are burned. However, both practices are not acceptable nowadays because the time taken for rotting is too long and open burning will affect the environment. The availability of large amounts of lignocellulosic material creates a need for research on the utilisation of OPT. Research on OPT is now gaining importance because of the shortage of raw material for the wood-based industry in Malaysia.

Oil palm is a monocotyledon plant, which comprises vascular bundle and parenchyma. It is well known that the sawn timber of OPT is weaker than normal wood. However, if OPT is peeled into veneers, it can be used for making composite panels. Laminated veneer lumber (LVL) panels from OPT veneer had been reported (Razak et al. 2008) to be comparable with rubberwood veneers and they passed the JAS standard (Anonymous 2003). Some of the studies on OPT reported on the properties of medium density fibreboard (MDF) (Abdul Khalil et al. 2007), plywood

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(Othman et al. 2008, Abdul Khalil et al. 2010) and LVL (Hashim et al. 2004, Kamaruzaman et al. 2004, Razak et al. 2008, Othman et al. 2009). Oil palm trunk as a lignocellulosic material behaves like wood. When the material is exposed to water vapour in the hygroscopic range, it adsorbs and desorbs water molecules in response to the moisture conditions of the surrounding environment. If a lignocellulosic material is placed in an environment where the relative humidity (RH) is stable at constant temperature, it will eventually attain a constant moisture content (MC), known as the equilibrium moisture content (EMC). When the EMC is plotted against RH at constant temperature, the resulting curve is a sorption isotherm (Skaar 1988). The adsorption/desorption properties of cellulosic and lignocellulosic materials are characterised by the sigmoidal (IUPAC Type II) shape of the isotherm and hysteresis between the adsorption and desorption loops.

The Hailwood–Horrobin (H–H) model has been used regularly to explain the sigmoid sorption isotherm observed with the sorption of water vapour on natural polymers and specifically for wood (Hailwood & Horrobin 1946). The H–H model, in the so-called two hydrate form, considers that water sorbed by wood can exist in two forms. The forms are water of hydration corresponding to water molecules that are H-bonded to the cell wall polymeric OH groups (hydration water) and solid solution or dissolved water corresponding to water molecules that are less constrained but are located within the cell wall nanopores (dissolution water) (Hailwood & Horrobin 1946). The H–H solid–solution model has also been used comprehensively to investigate the sorption behaviour of wood, chemically modified wood and non-wood such as bamboo (Siau 1984, Skaar 1988, Yasuda et al. 1994, Yamamoto et al. 2005, Dieste et al. 2008, Hill 2008, Ohmae & Nakano 2009, Zaihan et al. 2009, Xie et al. 2010). There are no studies on water vapour sorption using H–H model for OPT and its components such as parenchymatic and vascular bundle tissues. Most studies on the sorption isotherm characteristics of wood have used saturated salt solutions which can be time consuming and less precise. Lately, researchers have been using the dynamic vapour sorption (DVS) apparatus (Hill et al. 2009, Zaihan et al. 2009, Hill et al. 2010, Zaihan et al. 2010a, b, c) in water vapour isotherm studies. The objective

of this study was to determine the water vapour isotherms of rubberwood and different parts of OPT.

MATERIALS AND METHODS

The OPTs used in this experiment were harvested from a plantation in Nibong Tebal, Penang, Malaysia. All trunks were peeled into veneer and dried at a mill in Butterworth, Penang. The specimens were separated into six parts: two cross-sectional zones (inner and outer), two vertical zones (top and bottom) and three types of tissues (parenchyma + vascular bundle, parenchyma and vascular bundle). Rubberwood was used as comparison. The specimens were ground to fine particles and passed through a BS410-1:2000 mesh sieve no. 20 (0.841 mm sieve opening).

Determination of morphology characteristic of samples

The microstructure of samples were characterised using a scanning electron microscope (SEM). The specimens were affixed on stubs and carbon-coated. The accelerating voltage used was 3kV.

Determination of sorption isotherm data

A DVS apparatus was used for the sorption analysis. The material was placed on a clean sample pan which was carefully hooked onto the wire connected to the microbalance. Nitrogen with a pre-set percentage RH was passed over the sample at $200 \text{ cm}^3 \text{ min}^{-1}$ flow rate at $25 \pm 0.1 \text{ }^\circ\text{C}$. The schedule for the DVS was set at 14 different RHs (0, 5, 10, 15, 20, 30, 40, 50, 60, 70, 80, 85, 90 and 95%). The sample mass readings from the microbalance (measured every 20 s) show the vapour adsorption/desorption behaviour of the material. The instrument maintained a sample at a constant RH until the weight change per minute (dm/dt) reached a value less than $0.002\% \text{ min}^{-1}$ over a 10 min period; a criterion that from previous experiments had been shown to give a sample MC within less than 0.1% of the equilibrium value (Hill et al. 2010). During the operation, it was found that the temperature and humidity values were very stable ($\text{RH} \pm 0.1\%$, $\text{temperature} \pm 0.1 \text{ }^\circ\text{C}$), although both RH and temperature did not necessarily stabilise precisely at the pre-set values. A full description of the

apparatus and methodology is reported in Hill et al. (2009).

Evaluation of sorption isotherms using Hailwood–Horrobin model

The adsorption behaviour of wood and different parts of oil palm at each RH were analysed by fitting the experimental data using the Hailwood–Horrobin model (H–H) (Hill 2008, Zaihan et al. 2009) as shown below.

$$M = M_h + M_s = \frac{1800}{W} \left(\frac{K_1 K_2 H}{100 + K_1 K_2 H} \right) + \frac{1800}{W} \left(\frac{K_2 H}{100 - K_2 H} \right)$$

where M is the EMC at a given relative humidity (H), M_h is the moisture content associated with water of hydration, M_s is the moisture content associated with solution water, K_1 is the equilibrium constant between water of hydration and dissolved water with dry wood, K_2 is the equilibrium constant between atmospheric water vapour and dissolved water, and W is the molecular weight of cell wall polymer per mole of water sorption sites. The detailed theory of the H–H model can be found in Siau (1984) and Skaar (1988).

RESULTS AND DISCUSSION

Morphological characterisation of OPT and rubberwood

The morphology of OPT and rubberwood was characterised using SEM (Figure 1). Although the materials were in powder form, the cell wall of OPT and rubberwood was still intact.

Comparison of sorption isotherm of different parts of OPT and rubberwood

The moisture adsorption isotherms showed the characteristic IUPAC Type II (sigmoid shape) and IUPAC Type III (concave upwards) curves with sorption hysteresis as seen in Figure 2 for rubberwood and all parts of OPT respectively. OPT comprises mainly parenchymatic tissue which contains substantial starch deposits (Tomimura 1992). All parts of OPT showed the IUPAC Type III curves of the isotherm which were similar to that observed for starch (Bertuzzi et al. 2003). At a target RH of 95%, rubberwood had lower hygroscopicity than OPT-bottom-inner, OPT-bottom-outer, OPT-top-outer, OPT-bottom-outer (parenchyma), OPT-

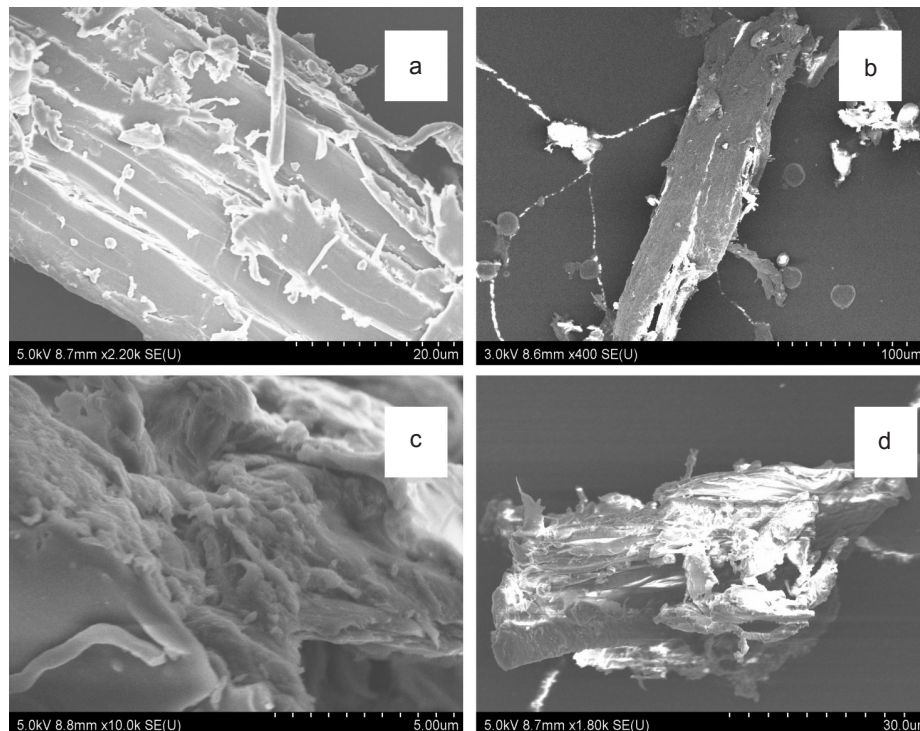


Figure 1 The morphological characteristics of OPT (a, b) and rubberwood (c, d)

bottom-outer (vascular bundle) and OPT-bottom-outer (parenchyma + vascular bundle) with values of 20.9 (Figure 2d), 51.3, 41.4 (Figure 2a), 39.9, 41.4 (Figure 2b), 52.3, 28.7 and 41.4% (Figure 2c) respectively. Oil palm trunk (monocotyledon) has structural differences compared with normal wood (dicotyledon) which mostly consists of secondary xylem. The OPT is composed of a very hard peripheral ring surrounding the soft central region. The hard peripheral zone (outer layer) is composed of a small number of parenchyma cells and a large number of vascular bundles (Lim & Gan 2005). Conversely, the soft central region (inner layer) has a small number of vascular bundles embedded within a large amount of parenchymatic tissue.

Figure 2a reveals that different parts of OPT show different EMCs, with the inner part having higher EMC than the outer. The variation in the composition of parenchyma and vascular bundle in the different regions of OPT affected the sorption properties. The outer region is

rich in vascular bundles and the inner region, parenchymatic tissue with high starch content (Tomimura 1992). Similar observation was reported for bamboo, where the inner region exhibited higher EMC than the outer (Yamamoto et al. 2005).

Figure 2b shows that the bottom and top parts of OPT have equivalent EMCs at various RHs. Both portions were taken from the outer part, so the compositions of parenchymatic and vascular bundle tissues are quite similar.

Figure 2c shows that the parenchymatic tissue and vascular bundle tissue of OPT show the highest and lowest levels of water sorption respectively. From this observation, it is confirmed that the inner part of OPT with higher proportion of parenchyma cells exhibits higher EMC. Ohmae and Nakano (2009) also noted that the vascular bundle tissue from bamboo was less hygroscopic than the parenchymatic tissue.

Figure 2d shows that rubberwood has the lowest EMC compared with all parts of OPT.

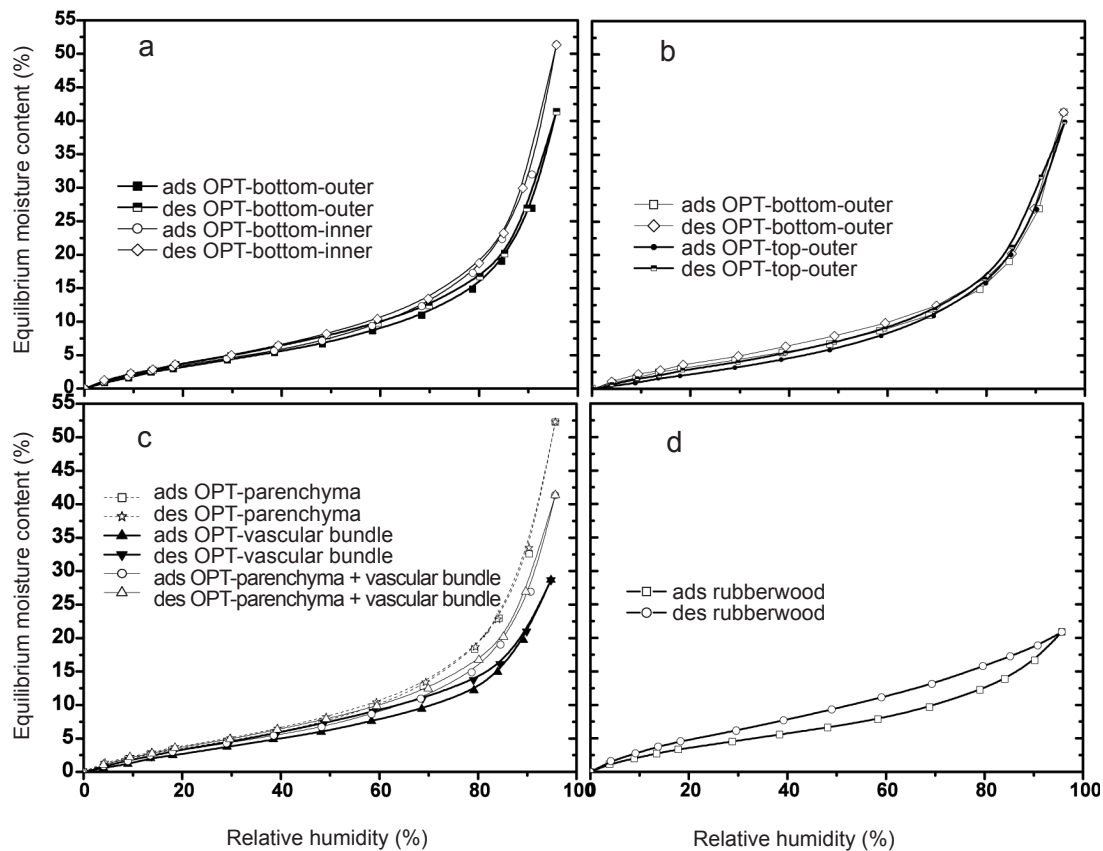


Figure 2 Comparison of moisture adsorption (ads) and desorption (des) behaviours for (a) OPT bottom-inner and bottom-outer parts, (b) OPT bottom-outer and top-outer parts, (c) OPT parenchyma, vascular bundle and parenchyma + vascular bundle, and (d) rubberwood

Analysis using Hailwood–Horrobin (H–H) fitting to the data

The H–H model considers that water sorbed by wood exists partly as water of hydration and partly in solid solution. The three chemical species are polymer (dry cellulose or wood), hydrated polymer and dissolved water. The values of A, B, C, K_1 , K_2 and W of the specimens are shown in Table 1. All parts of OPT had lower K_1 values compared with rubberwood. Low K_1 implies the decrease in the activity of hydrated wood with respect to both the activities of dry wood and dissolved water. The K_2 value was a bit higher in OPT compared with rubberwood which showed increased activity of dissolved water in nanopores. However, the W values were comparable. From the H–H fitting model using the equation above, the adsorbed water was calculated as hydration water (Mh) and dissolution water (Ms). The results of the H–H fits to the data for the different parts of OPT and rubberwood are shown in Figure 3. The sum of these two components can be extrapolated to 100% RH to give the projected fibre saturation point (p-FSP) of the material. The use of the term p-FSP is to distinguish this parameter from FSP values obtained by other methods (such as solute exclusion) which do not give the same results (Hill 2008). It should be noted that the projection method can produce spurious data, especially when the isotherm is rising sharply towards the higher end of the hygroscopic range. Furthermore, the use of the term FSP can be misleading in situations (as here) where not all of the sorbed water is located in the cell wall.

The values for Mh, Ms and p-FSP obtained from the H–H fits to the data of different types of tissue of OPT and rubberwood are presented in Figure 4. The Mh values projected to 100% RH for all the specimens were similar, ranging from 3.3 to 3.9% MC. The Ms values varied between the different parts of OPT and were very much higher than rubberwood. The parenchymatic tissue of OPT had the highest projected Ms at 99.6% and the vascular bundle tissue had the lowest at 37.6% MC. The Ms for rubberwood was 17% and was the lowest. The p-FSP showed significant differences between the various OPT samples and rubberwood. The inner and outer OPT parts were totally different in both Ms and p-FSP values. However, between the top and bottom parts of OPT, there were no differences. The Ms value of parenchymatic tissue was higher than vascular bundle. Starch content was reported to be higher in parenchyma cells (55.5%) than in vascular bundles (2.4%) of OPT (Tomimura 1992). Studies have shown that starch has a high EMC at the top end of the hygroscopic range (Pascual & Clara 1999, Bertuzzi et al. 2003, Al-Muhtaseb et al. 2004, Perdomo et al. 2009). Consequently, the presence of large amounts of starch in the OPT dominates the sorption properties of the material. Much of the adsorbed water is not located in the cell wall of the material but rather within starch deposits. This results in the derivation of the IUPAC Type III isotherm, rather than the Type II which is associated with cellulosic and lignocellulosic materials. Furthermore, it is incorrect to interpret the projected isotherm to 100% RH as fibre

Table 1 Fitted and physical constants calculated from the Hailwood–Horrobin adsorption isotherm

Material	A	B	C	K_1	K_2	W
Rubberwood	2.859	0.167	0.0015	8.210	0.81	383.94
OPT-bottom-inner	3.456	0.153	0.0018	5.675	0.94	394.49
OPT-bottom-outer	3.991	0.149	0.0017	5.036	0.92	400.96
OPT-top-outer	3.974	0.149	0.0017	5.069	0.92	400.02
OPT- bottom-outer (vascular bundle)	4.979	0.150	0.0017	4.375	0.89	429.32
OPT- bottom-outer (parenchyma)	3.278	0.152	0.0017	5.869	0.95	386.61

A, B and C are the constants obtained from the fitting parameters of the second order polynomial, W is the molecular weight of dry wood per mole of water sorption sites, K_1 is the equilibrium constant where the hydrate is formed from dissolved water and dry wood, K_2 is the equilibrium constant between dissolved water and water vapour.

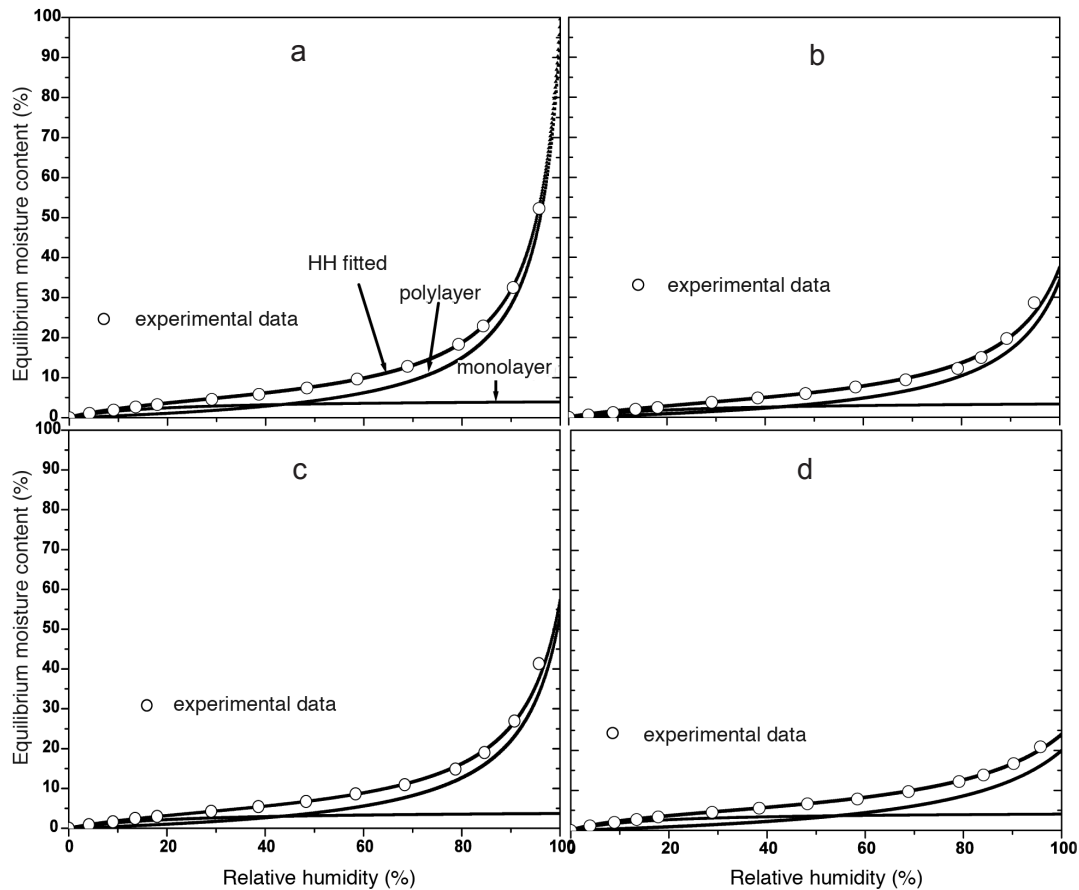


Figure 3 Comparison of hydration (Mh) water and dissolution (Ms) water calculated using the Hailwood-Horrobin model, and sum of hydration and dissolution water (total) adsorption isotherms fits compared with a best fit line through the adsorption isotherm data for the (a) parenchyma, (b) vascular bundle, (c) vascular bundle + parenchyma OPT and (d) rubberwood

saturation point. The partitioning of the water between the cell wall and starch is not known but it is clear that much of the adsorbed water is located within the starch component.

Sorption hysteresis

The rubberwood was found to have much larger hysteresis loops compared with the different parts of OPT (Figure 5). Only at 90% RH did the top part of OPT have higher hysteresis and this may be due to capillary condensation phenomenon. Recently, a model has been introduced to explain hysteresis in wood and plant fibres (Hill et al. 2009). This model describes the response of the cell wall matrix to the ingress or egress of water molecules under conditions of adsorption or desorption respectively. Water molecules entering the cell wall cause the formation or expansion of nanopores within the matrix between the microfibrillar reinforcing elements,

similarly as water molecules exit the cell wall, the nanopores collapse behind them. However, the rate of expansion or collapse of the nanopores is controlled by the local stiffness or cross-link density of the enveloping matrix. In a glassy solid below the glass transition temperature (T_g), the opening and closing of nanopores are not instantaneous but kinetically hindered. As a result, adsorption and desorption occur in a material which is in different states, giving rise to the property of hysteresis. A more detailed description is given by Xia and Pignatello (2001), Lu and Pignatello (2002) and Sander et al. (2005). In humic soils, the magnitude of hysteresis is greater where the cross-linking density of the matrix has been increased due to the addition of aluminium ions (Lu & Pignatello 2004). The relatively large values exhibited by the rubberwood may well be related to lignification of the cell wall and local stiffness of the cellular matrix (Table 2). The lack of hysteresis exhibited by the

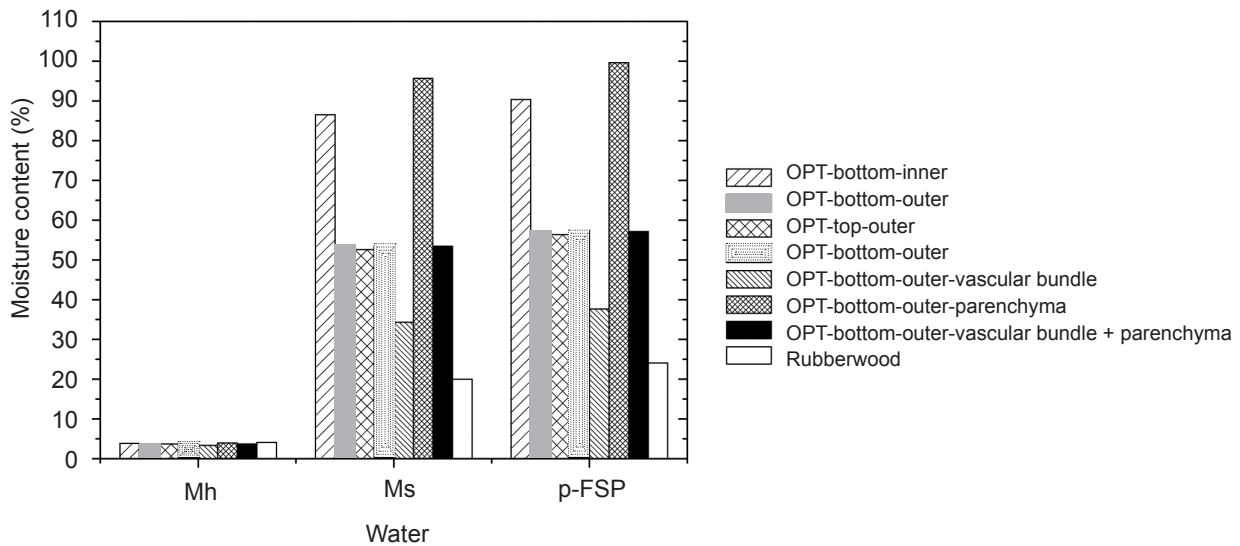


Figure 4 Values for Mh (hydration water), Ms (dissolution water) and p-FSP (projected fibre saturation point) water derived from Hailwood–Horrobin fits projected to 100% RH for all parts of OPT and rubberwood

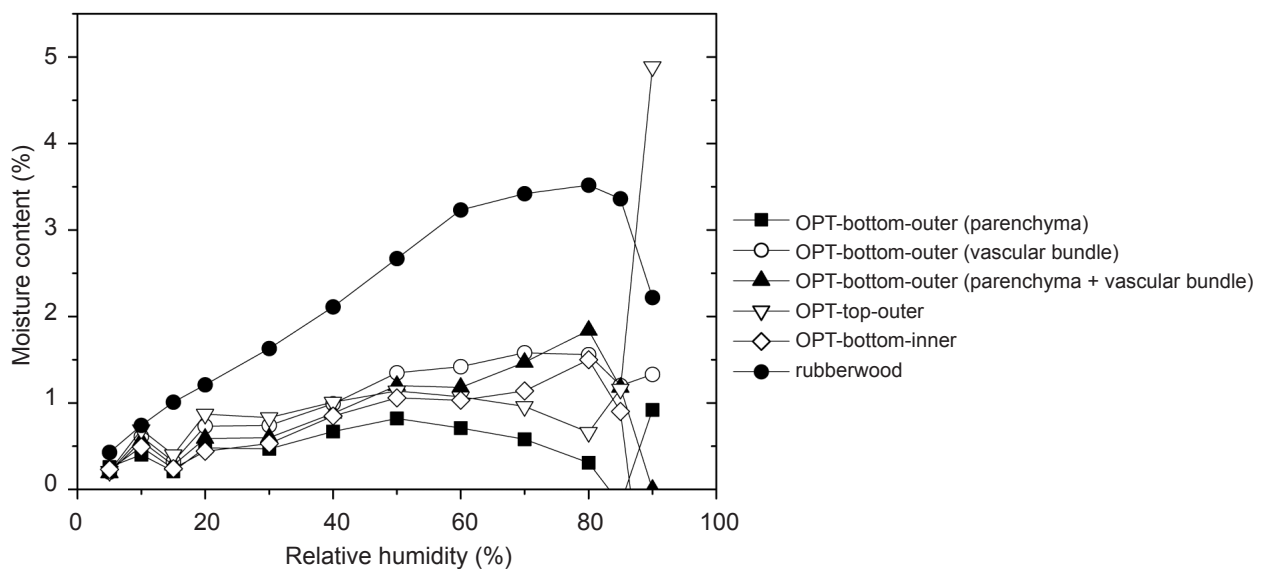


Figure 5 Sorption hysteresis between adsorption and desorption curves (obtained by subtraction of EMCs) for all parts of OPT and rubberwood at different values of RH

Table 2 Chemical compositions of oil palm trunk and rubberwood

Chemical composition (%)	Oil palm trunk ^a	Rubberwood ^b
Cellulose	41.2	41.5
Hemicellulose	34.4	25.5
Lignin	17.1	26.0
Extractives	0.5	1.5
Ash	3.4	1.5

^aSun and Tomkinson (2001), ^bHanafi (2001)

OPT material is likely to be related to the high starch content of the parenchymatic tissue. In addition, vascular bundle tissue (with less starch) has more hysteresis than the parenchymatic tissue (Figure 5). The starch derived from tapioca showed reducing Tg as the moisture content increased, ranging from 175 °C at 5% MC to 70 °C at 15% and dropping to 30 °C at 27% MC (Chang et al. 2000). Assuming similar values for the starch in OPT implies that hysteresis should be larger at the lower end of the hygroscopic range (Tavakolipour & Kalbasi-ashtari 2008).

CONCLUSIONS

This study showed that there were large differences in the adsorption/desorption behaviour between all parts of OPT and rubberwood. However, there were no differences between the top and bottom of OPT. All parts of OPT showed a high EMC at each target relative humidity, with the inner part having a higher EMC due to higher starch content in parenchyma cells. The two hydrate H–H model showed that the differences observed were almost entirely due to changes in the dissolved water content. The sorption hysteresis of rubberwood was found to be higher than all parts of OPT due to the presence of starch in the OPT tissue.

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