

MOISTURE ADSORPTION ISOTHERMS OF ACACIA MANGIUM AND ENDOSPERMUM MALACCENSE USING DYNAMIC VAPOUR SORPTION

J Zaihan^{1,2,*}, CAS Hill¹, S Curling¹, WS Hashim² & H Hamdan²

¹Centre for Timber Engineering, School of Engineering and the Built Environment, Edinburgh Napier University, Edinburgh, UK

²Forest Research Institute Malaysia, 52109 Kepong, Selangor Darul Ehsan, Malaysia

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ZAIHAN J, HILL CAS, CURLING S, HASHIM WS & HAMDAN H. 2009. Moisture adsorption isotherms of *Acacia mangium* and *Endospermum malaccense* using dynamic vapour sorption. Two Malaysian hardwoods, namely, acacia (*Acacia mangium*) and sesenduk (*Endospermum malaccense*) were studied to determine their moisture sorption behaviour using a dynamic vapour sorption (DVS) apparatus. For comparison, two temperate softwoods, Sitka spruce (*Picea sitchensis*) and radiata pine (*Pinus radiata*), and one commercially modified wood, Accoya (radiata pine that is chemically modified with acetic anhydride), were tested with the same DVS. The sigmoid isotherm plot for each of the wood tested showed differences in the adsorption and desorption plots. At 90% relative humidity (RH), acacia and sesenduk had lower hygroscopicity (16.2 and 17.9% respectively) compared with radiata pine (18%) and Sitka spruce (20.1%). The modified Accoya had the lowest hygroscopicity (7.5%) due to bulking of the cell wall with acetyl. Hysteresis of Accoya also exhibited the lowest value between the adsorption and desorption isotherms. Data were analysed using the Hailwood–Horrobin (HH) model for isotherm fitting and determination of monolayer and polylayer moisture content. The OH group concentration in the HH monolayer did not correspond to the total accessible OH group derived by calculation based on the chemical composition of each of the wood species.

Keywords: Desorption, hygroscopicity hysteresis, water vapour, Hailwood–Horrobin model, DVS

ZAIHAN J, HILL CAS, CURLING S, HASHIM WS & HAMDAN H. 2009 Isoterma penyerapan lembapan oleh kayu *Acacia mangium* dan *Endospermum malaccense* dengan menggunakan alat erapan wap dinamik. Dua kayu keras Malaysia iaitu akasia (*Acacia mangium*) dan sesenduk (*Endospermum malaccense*) dikaji sifat erapan lembapan menggunakan alat erapan wap dinamik (DVS). Sebagai perbandingan dua kayu lembut iklim sederhana, sprus Sitka (*Picea sitchensis*) dan pain radiata (*Pinus radiata*), dan satu kayu yang diubah suai secara komersial yang dinamai Accoya (pain radiata yang diubah suai secara kimia menggunakan asetik anhidrida) telah diuji dengan alat DVS yang sama. Plot isoterma berbentuk sigmoid bagi setiap spesies kayu yang diuji menunjukkan perbezaan lengkungan penyerapan dan penyaherapan. Pada kelembapan relatif 90%, akasia dan sesenduk mempunyai kehidroskopikan yang lebih rendah (masing-masing 16.2% dan 17.9%) berbanding pain radiata (18%) dan sprus Sitka (20.1%). Kayu yang telah diubah suai iaitu Accoya menunjukkan sifat kehidroskopikan yang paling rendah iaitu 7.5% disebabkan dinding selnya telah dipenuhi oleh bahan asetil. Bagi sifat histeresis pula Accoya jelas menunjukkan perbezaan terendah antara isoterma penyerapan dengan penyaherapan. Data DVS ini juga dianalisis menggunakan model Hailwood–Horrobin untuk penetapan isoterma dan penentuan kandungan air lapisan mono dan lapisan poli. Kumpulan OH yang dihasilkan daripada lapisan mono model ini tidak sama dengan jumlah kumpulan OH yang dihasilkan daripada pengiraan berdasarkan komposisi kimia setiap spesies kayu.

INTRODUCTION

The physical and mechanical behaviour of wood as a hygroscopic material needs to be fully understood in order to improve its utilization. Wood adsorbs moisture in high humidity environments and desorbs moisture in low humidity. Most studies on the sorption characteristics of wood use saturated salt solutions

which is time consuming and only allows for collection of discrete moisture-time data. The use of a dynamic vapour sorption apparatus (DVS) is a relatively new technique developed to collect continuous weight change over time at any desired relative humidity (RH) between 0 and 95% within a short period of time.

*E-mail: z.jalaludin@napier.ac.uk

When the equilibrium moisture content (EMC) is plotted against RH at a constant temperature the resulting curve is a sorption isotherm (Skaar 1988). The adsorption/desorption properties of cellulosic and lignocellulosic materials are characterized by the sigmoidal (IUPAC type II) shape of the isotherm and hysteresis developed between the sorption and desorption loops. The sorption value extrapolated at 100% RH is often interpreted as the fibre saturation point (FSP) of the material. Tiemann (1906) defined the FSP as the EMC of the sample when the cell wall is at saturation, but there is no water in the macrovoids (such as cell wall lumina) of the material.

The sigmoidal nature of the isotherms has been described by numerous models (Skaar 1972, Simpson 1980, Okoh & Skaar 1980, Siau 1984) and is considered to consist of three components. At low RH values (0–15%), monolayer adsorption onto the internal surface of the cell wall is the dominant process; between 15 and 70% RH, polylayer water formation in the transient cell wall microcapillaries occurs and above 70% RH, capillary condensation becomes increasingly dominant. It is well known that sorption isotherm behaviour vary from one kind of wood to another (Siau 1984). There are many theories explaining the sorption of water by hygroscopic polymers such as wood and textile materials. The Hailwood–Horrobin (HH) model has been regularly used to explain the sigmoid sorption isotherm observed with the sorption of water vapour on polymers, specifically for wood (Hailwood & Horrobin 1946). It has proved to be useful although it has been criticized for some of the assumptions used in its derivation. Good descriptions of this model can be found in Spalt (1958), Skaar (1972, 1988), Okoh and Skaar (1980), Simpson (1980) and Hill *et al.* (2009).

The HH model considers that water sorbed by wood can exist in two forms, namely, water of hydration corresponding to water molecules that are H-bonded to the cell wall polymeric OH groups (monolayer water) and solid solution or dissolved water corresponding to water molecules that are less constrained, but nonetheless are located within the cell wall microvoids (polylayer water) (Hailwood & Horrobin 1946). In the HH model, the monolayer can change over the whole range of RH and is thus a much more appropriate description for an evolving surface associated with a material that changes

dimension as sorption proceeds compared with other models such as the BET isotherm. However, the HH model gave excellent agreement with experimental observations over a wide range of RH values, although it does not explain hysteresis and is only suited to describing the adsorption part of the isotherm.

Hysteresis is the phenomenon where the wood sigmoid sorption isotherm differs depending upon whether moisture is being adsorbed or desorbed. Stamm (1952) reported a higher moisture content value during desorption than adsorption at the same equilibrium relative vapour pressure. A number of explanatory theories have been proposed: a reduction in the number of surface sites due to lateral hydroxyl-to-hydroxyl bonding upon desorption to dryness; differences in the degree of aggregation or dispersion attained by the gel respectively in dry and saturated conditions; and differences between advancing and receding contact angle of water against capillary walls producing higher wettability under desorption than under adsorption conditions (Hill *et al.* 2009).

Adsorption of water onto a hygroscopic natural polymer such as wood or cellulose is thought to depend entirely on OH groups on the surface. The mechanism of water adsorption in wood has, therefore, been studied by evaluating the quantity and the state of the OH groups which act as the adsorption sites. Until now in order to measure the number of adsorption sites of water in wood, it has been usual to draw a sorption isotherm from which the adsorbed area can be calculated by assuming each water molecule in the monolayer water is associated with a primary sorption site (OH groups) according to the HH model. However theoretically, the number of the OH groups per unit mass of dry wood can also be calculated on the basis of an estimate of the number of the OH groups associated with each of the cell wall polymeric constituents (Rowell 1980). Despite various studies on these methods, little evidence about the relationship between the HH theory and Rowell's method has been discussed.

The present study was undertaken in order to determine the sorption behaviour of two tropical Malaysian hardwoods which are currently being promoted as potential plantation species in Malaysia (Rushdan *et al.* 2007), two temperate softwoods which are widely planted in the UK and New Zealand, and Accoya, a chemically

modified radiata pine. This study included a relationship between the water molecule in the monolayer (HH model) and the primary sorption site. A dynamic vapour sorption intrinsic (DVS) apparatus was used for this purpose. The DVS intrinsic is able to provide highly accurate isotherms over a RH range that can be preset while the temperature at which isotherms are recorded can be varied from 20 to 40 °C. The adsorption isotherms were then analysed using the HH sorption theory.

MATERIALS AND METHODS

Sample preparations

Four wood species, namely, acacia (*Acacia mangium*), sesenduk (*Endospermum malaccense*), Sitka spruce (*Picea sitchensis*), radiata pine (*Pinus radiata*) and one commercially treated wood, Accoya (radiata pine chemically modified by acetic anhydride), were used in this study. Kiln-dried timber of acacia and sesenduk were obtained from the Forest Research Institute Malaysia while Sitka spruce wood was sourced locally. Radiata pine and Accoya were supplied by Titan Wood, a company based in the Netherlands. The wood samples were ground to fine particles which passed through a BS410-1:2000 mesh sieve (850 micron).

Determination of isotherm using a dynamic vapour sorption intrinsic apparatus

Isotherm analyses were performed using a DVS (Surface Measurement Systems Ltd.). The DVS is designed to accurately measure the change in mass of a sample as it sorbs precisely controlled concentrations of water vapours in an air (or nitrogen) carrier gas. The sample material was placed onto a pre-cleaned sample pan and carefully placed on the hang down wire connected to a microbalance. Then the sample chamber was closed and clamped. Air with a known percentage of relative humidity (RH) was then passed over the sample at a flow rate of 200 cm³ s⁻¹ and temperature, 25 °C. Mass readings from the microbalance showed the vapour adsorption/desorption behaviour of samples.

The schedule for the DVS was set to 10 different RH values, namely, 0, 10, 20, 30, 40, 50, 60, 70, 80 and 90%. Data on mass change was acquired every 20 seconds. At each RH an in-

built algorithm was set to ensure that equilibrium had been reached when the slope of an adjusted tangent line to the curve of mass change with respect to time for the last 10 min of data was < 0.002% min⁻¹. Humidity and temperature probes were located close to the sample and reference holders. It was found that the temperature and humidity values were very stable during the tests, although they were seldom exactly at the pre-set values and it was necessary to take the actual readings at each adsorption and desorption stage from the output data spreadsheets.

Reproducibility of data

In this study the DVS only measured one sample of one species at a time. In order to determine the reproducibility of the sorption data, a sample of acacia was subjected to four consecutive sorption/desorption cycles instead of the one cycle used in normal testing. The result is shown in Figure 1. The sorption curves of the four cycles showed that it was reproducible over most of the RH range studied. Therefore, the DVS is assumed to be able to give reproducible sorption data for every sample tested.

RESULTS AND DISCUSSION

Isotherm plot of different wood species

Individual moisture adsorption/desorption isotherms are shown in Figure 2. All samples exhibited the classic Type II sigmoidal adsorption and desorption isotherms. However, there were considerable differences in the total amount of moisture present in the samples at a given RH as well as in the degree of hysteresis exhibited. The differences in adsorption and desorption behaviour were clearly illustrated when the curves were overlaid (Figure 3). It should be noted that because samples were desorbing from an EMC below FSP, the desorption curve was a 'scanning curve' rather than a 'boundary curve'. However, the desorption curve rapidly approached the boundary. The unmodified acacia, sesenduk, Sitka spruce and radiata pine exhibited higher EMCs on adsorption and desorption compared with the modified wood Accoya. Many factors influenced the moisture sorption properties of the treated wood, of which OH accessibility is a significant component. At a target RH of 90%, acacia had lower hygroscopicity than sesenduk,

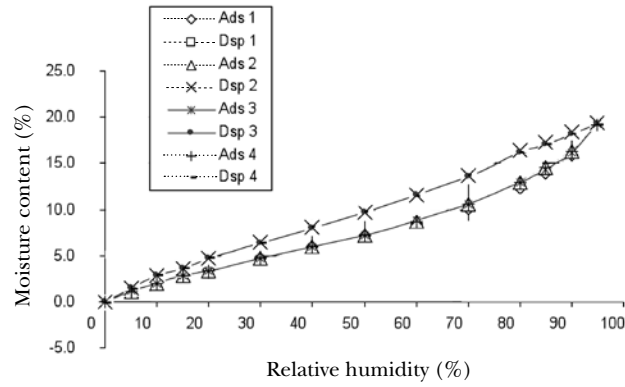


Figure 1 Reproducibility of moisture adsorption (Ads)/desorption (Dsp) of acacia (*Acacia mangium*) in four cycles

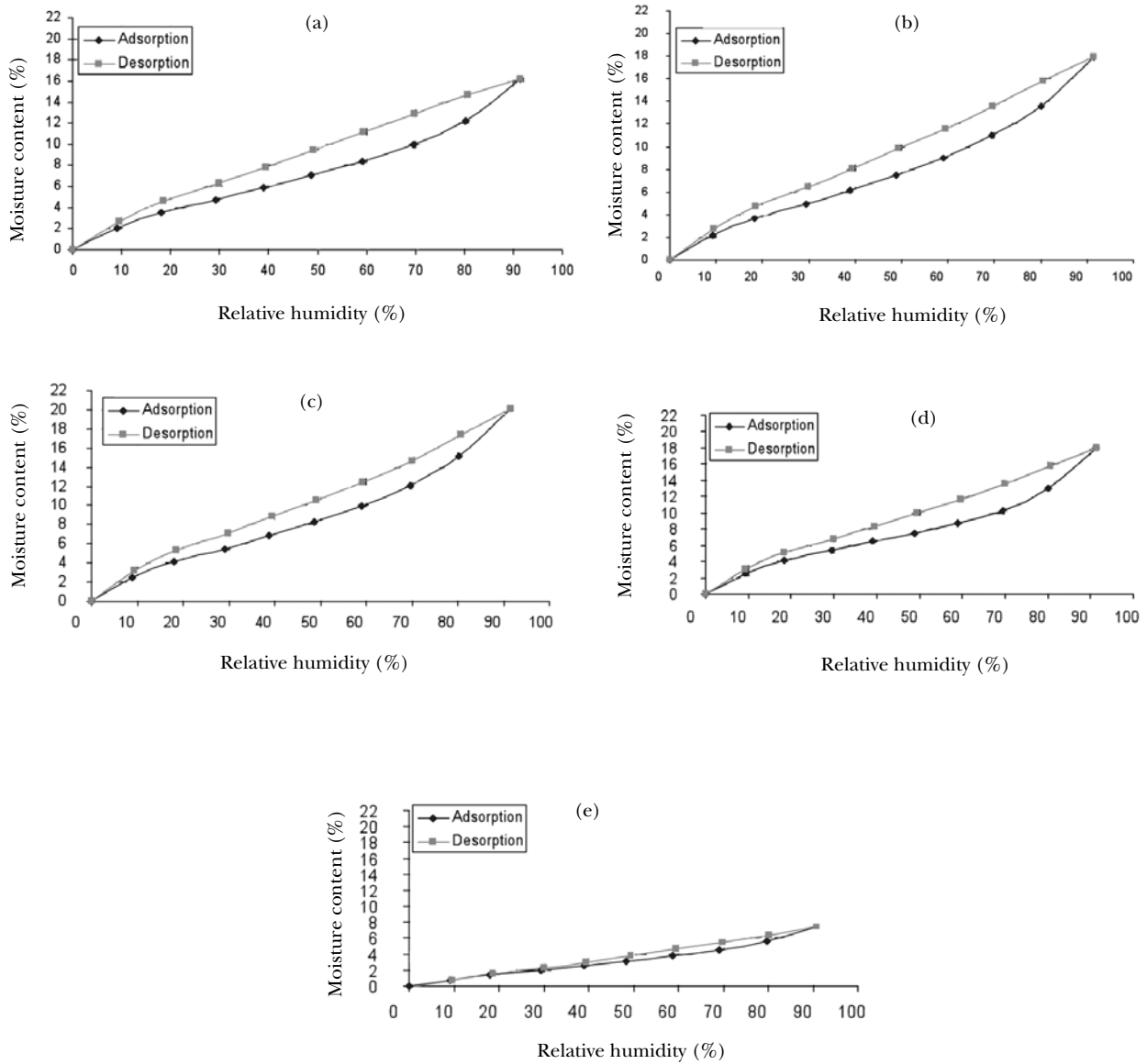


Figure 2 Moisture adsorption and desorption behaviour for (a) acacia, (b) sesenduk, (c) Sitka spruce, (d) radiata pine and (e) Accoya at 25 °C

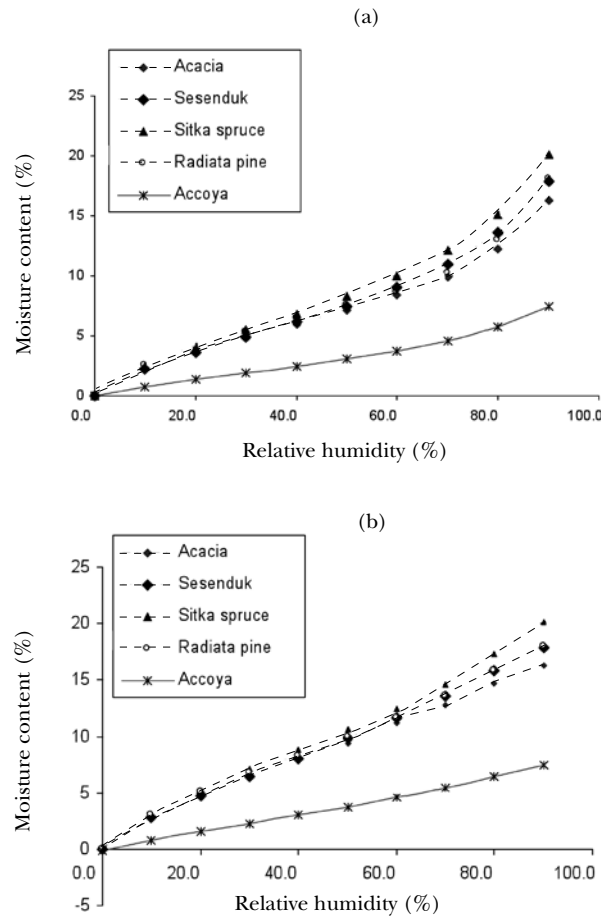


Figure 3 Graph comparing the moisture content adsorption (a) and desorption (b) behaviour of the five woods investigated in this study

Sitka spruce and radiata pine with values of 16.2, 17.9, 20.1 and 18.0% respectively (Figure 3a). These could be due to several factors such as differences in the proportion of major wood constituents (i.e. cellulose, hemicelluloses and lignin) present in the woods or differences in extractive contents (Siau 1984). Accoya had the lowest hygroscopicity at 7.5% (Figure 3a). It has been reported that the reduction in EMC of chemically modified wood was primarily determined by the volume adduct deposited in the cell wall (bulking) rather than by the number of hydroxyl groups that had been substituted (Papadopoulos & Hill 2003).

The unmodified woods also had larger hysteresis loops compared with Accoya (Figure 4). At 70% RH, radiata pine had the highest hysteresis followed by acacia, sesenduk, Sitka spruce and Accoya with values of 3.3, 2.9, 2.6, 2.6 and 0.9% respectively. Hysteresis over the entire range of relative humidity is predominantly influenced by polylayer water sorption (Spalt 1958). In the

upper range of relative humidities (above 60%), polylayer and total sorption hysteresis, under isotherm conditions, are positively correlated with wettability hysteresis. Consequently Chen and Wangaard (1968) suggest that sorption hysteresis is, at least in part, a consequence of difference in the wettability of cell wall capillaries when undergoing adsorption or desorption. The contribution of monolayer hysteresis to total hysteresis is of minor importance in the range of relative humidities higher than 60% (Spalt 1958, Wangaard & Granados 1967). However, hysteresis occurs throughout the sorption range and not just in capillary condensation region only.

Hailwood–Horrobin fitting to data

A full description of the HH theory is given in Skaar (1988) and Siau (1984). The HH model considers the state of equilibrium that exists between a vapour phase, liquid phase (in cell lumina) and a solid solution (in the cell wall).

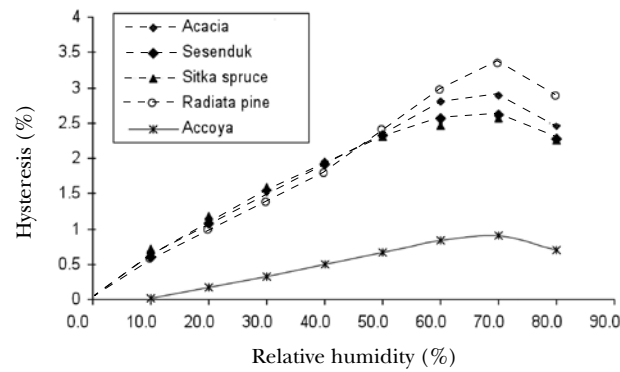


Figure 4 Hysteresis between adsorption and desorption curves (obtained by subtraction of equilibrium moisture contents) at different values of relative humidity

The classic sigmoidal shape of a sorption isotherm is deconvoluted by the HH model into a monolayer (Mh) and a polylayer (Ms) component in equation 1.

$$M = Mh + Ms = \frac{1800}{W} \left(\frac{K_1 K_2 H}{100 + K_1 K_2 H} \right) + \frac{1800}{W} \left(\frac{K_2 H}{100 - K_2 H} \right) \quad (1)$$

where

M = percentage moisture content at a given percentage relative humidity (H)

W = molecular weight of cell wall polymer per sorption site

K₁ and K₂ = constants.

The values of K₁ and K₂ were determined by plotting H/M against H, which was predicted by the HH theory to give a parabolic relationship of the form shown in equation 2.

$$\frac{H}{M} = A + BH - CH^2 \quad (2)$$

where A, B and C are constants obtained from the fitting parameters of the second order polynomial of equation and K₁, K₂, W, A, B and C are linked by the following relationships:

$$A = \frac{W}{18} \left[\frac{1}{K_2(K_1 + 1)} \right] \quad (3)$$

$$B = \frac{W}{1800} \left[\frac{K_1 - 1}{K_1 + 1} \right] \quad (4)$$

$$C = \left(\frac{W}{180000} \right) \left[\frac{K_1 K_2}{K_1 + 1} \right] \quad (5)$$

From the above relationships, the values of K₁, K₂ and W can be derived using equations 6 to 8.

$$K_1 = 1 + \frac{B^2 + B\sqrt{B^2 + 4AC}}{2AC} \quad (6)$$

$$K_2 = \frac{50(-B + \sqrt{B^2 + 4AC})}{A} \quad (7)$$

$$W = 1800\sqrt{B^2 + 4AC} \quad (8)$$

The values of A, B, C and coefficient of determination (R²) for the second order polynomial fit to the data points for the plot of H/M versus H, K₁, K₂ and W of the woods studied are shown in Table 1. In the HH model, W or molecular weight per sorption site for different species showed differences, with Accoya having the highest value at 984.9. This showed that the sorption site accessibility for hydroxyl groups in Accoya was less than the four wood species. Acacia had the highest W value and the lowest hygroscopicity compared with sesenduk, Sitka spruce and radiata pine. Results from the HH model fit to the experimental data for the five woods are shown in Figure 5. There was only a slight difference between the species. Relatively, wood species and wood modification also affected values of Mh and Ms obtained. With a value of 22.1%, acacia had a slightly lower FSP than the three wood species. This may be due to

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

Wood species	A	B	C	R ²	K ₁	K ₂	W
Acacia	1.313	0.212	0.0018	0.897	20.994	0.808	419.97
Sesenduk	1.301	0.202	0.0018	0.882	19.358	0.845	403.01
Sitka spruce	1.102	0.184	0.0016	0.899	21.217	0.827	364.57
Radiata pine	0.897	0.208	0.0018	0.942	28.8015	0.835	401.53
Accoya	4.214	0.475	0.0044	0.784	14.077	0.861	984.93

A, B and C = constants obtained from the fitting parameters of the second order polynomial of equation 2; W = molecular weight of dry wood per mole of water sorption sites; R² = coefficient of determination; K₁ = equilibrium constant where the hydrate is formed from dissolved water and dry wood; K₂ = equilibrium constant between dissolved water and water vapour

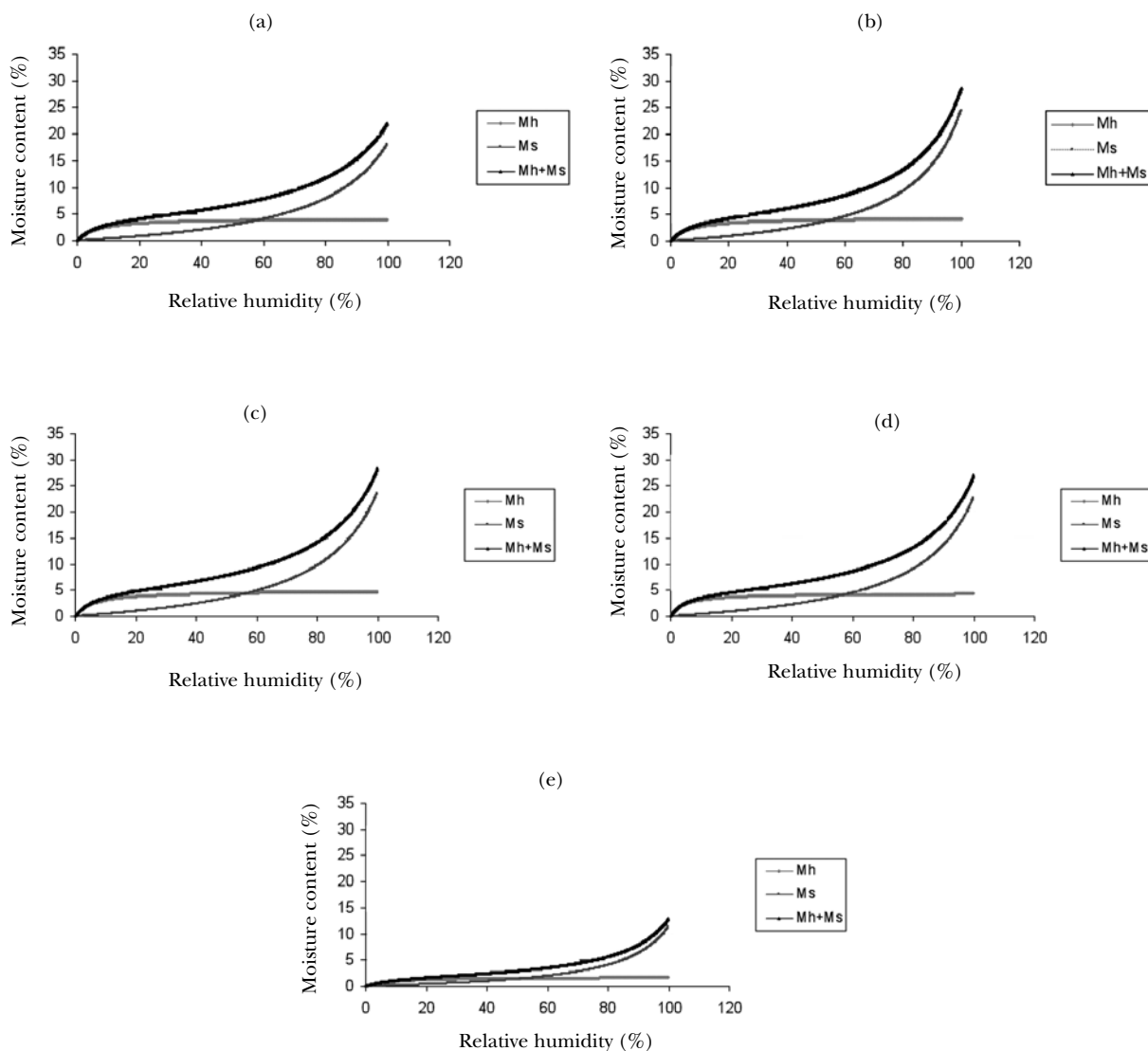


Figure 5 Comparison of monolayer, polylayer calculated using the Hailwood–Horrobin model and sum of polylayer and monolayer (total) adsorption isotherm fits with a best fit line through the adsorption isotherm data for (a) acacia (b) sesenduk, (c) Sitka spruce, (d) radiata pine and (e) Accoya; Mh = monolayer; Ms = polylayer; Mh + Ms = crude estimate of fibre saturation point (FSP)

differences in accessibility of sorption site and the chemical composition of each wood species (Table 2). It is known that FSP for wood is in the range of 25 to 30% (Skaar 1988). However, it should be noted that the use of sorption isotherm projections to give FSP is an erroneous method and, therefore, such data should be treated with caution (Stamm 1971, Hill 2006, 2008).

The reduced sorptive capacity of *Acocya* is a consequence of the conversion of OH groups to less hydrophilic groups and of the bulking resulting from the introduction of these groups into the cell wall (Papadopolus & Hill 2003). If it is assumed that each water molecule in the monolayer is associated with a primary sorption site (OH group), then it is possible to determine accessible OH content of the wood.

The number of OH groups per unit mass of dry wood can be calculated on the basis of an estimate of the number of OH groups associated with each of the cell wall polymeric constituents (Rowell 1980). For the cellulose component only 40% of the OH content was considered in the calculation as 60% of the groups were assumed to be in the crystalline region and thereby inaccessible. The values of the total OH concentration for each species are shown in

Table 3. Comparison on the number of accessible OH groups in the monolayer as determined by the HH theory did not correspond with that determined using Rowell's method for any of the samples. Also the W values did not correspond to the accessibility of the OH groups. Based on the HH model, the monolayer water sorption at 100% RH for all four species after dividing with water molecular weight showed OH concentrations between 2.26 to 2.59 mmoles g⁻¹. On the other hand, with Rowell's method the OH concentrations were 8.7 to 9.7 mmoles g⁻¹. This ratio of 3 or 4 to 1 implies that each water molecule in the monolayer is associated with three to four primary sorption sites. However, the results suggest that OH concentration cannot be reliably determined from the monolayer water sorption only. Previous studies have used wood exposed to deuterated water in order to estimate the accessible OH content. Results show that various species have different OH contents, e.g 7.97 and 6.9 mmoles g⁻¹ for black spruce and white birch respectively (Sumi *et al.* 1964) and 3.3 mmoles g⁻¹ for Japanese cypress (Taniguchi *et al.* 1978). Further investigations using deuterium exchange may be a useful method to determine the accessibility of OH content.

Table 2 Chemical composition (wt %) of acacia, sesenduk, Sitka spruce and radiata pine

Wood species	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Acacia ¹	50.3	27.2	23.5
Sesenduk ²	42.2	29.4	26.3
Sitka spruce ³	40.0	31.0	28.0
Radiata pine ⁴	45.3	23	26.8

References: ¹Rushdan *et al.* (2007), ²Mohd. Nor (1991), ³Fengel and Wegener (1989), ⁴Araque *et al.* (2008)

Table 3 Estimation of OH concentration (in mmoles per gram) of dry cell wall substance from monolayer water content at 100% RH obtained from the Hailwood–Horrobin fits

Wood species	HH monolayer OH concentration	Totally accessible calculated OH concentration	OH concentration (60% crystalline cellulose)
Acacia	2.26	15.2	9.6
Sesenduk	2.34	14.2	9.5
Sitka spruce	2.59	14.2	9.7
Radiata pine	2.39	13.7	8.7

CONCLUSIONS

This study has shown that there are differences in the adsorption/desorption behaviour between the two tropical Malaysian hardwoods, two temperate softwoods and Accoya. Between the unmodified woods, acacia had the lowest hygroscopicity. Accoya exhibited the lowest level of hysteresis compared with the rest of the samples tested. Estimating FSP using the HH model through extrapolation sorption isotherm is questionable. Based upon the present study, it is concluded that each water molecule in the monolayer is associated with three to four primary sorption sites.

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