Original article

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Water vapour sorption behaviour and physico-mechanical properties of methyl methacrylate (MMA)- and MMA–styrene-modified batai (*Paraserianthes falcataria*) wood

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Abstract: The purpose of this study was to determine the applicability of methyl methacrylate (MMA) and MMA–styrene in treating batai (*Paraserianthes falcataria*) wood. The effectiveness of the treatment was evaluated based on Fourier transform infrared spectroscopy (FTIR) analysis, physico-mechanical properties of the treated wood, and moisture sorption isotherm using dynamic vapour sorption (DVS) apparatus. Physico-mechanical properties of the modified batai wood were improved. The MMA-treated batai wood showed better improvement in terms of physical and mechanical properties compared to the MMA–styrene-treated batai wood. The equilibrium moisture content (EMC) for untreated batai wood was higher than that of treated samples. At 95% RH, the EMC for untreated batai wood was 20.7%, whereas the EMC for MMA- and MMA–styrene-treated samples was 7.9 and 8.8%, respectively. The findings indicate that the modified batai wood absorbed less moisture compared to the untreated batai wood. Moreover, the untreated batai wood had larger hysteresis loop than the treated batai wood. The highest hysteresis value was observed at 80% RH for untreated batai wood (3.8%), followed by MMA–styrene-treated batai wood (1.7%) and MMA-treated batai wood (1.2%). Both MMA and MMA–styrene proved to be effective treatments for batai wood because they reduced its hygroscopicity.

Keywords: chemical modification; equilibrium moisture content; Hailwood–Horrobin; relative humidity; vapour sorption.

1 Introduction

Paraserianthes falcataria or locally known as batai, belongs to the family Leguminosae. It is a very fast-growing species and light-density wood. Both the heartwood and sapwood of batai are white to light brown in colour with a pink tinge. Its texture is coarse, and it is easy to resaw and crosscut. In addition, planing is easy for this wood and the surface produced is smooth as well as having rather good nailing property (Lim and Chung 2002). In recent years, batai has been gaining popularity in Malaysia owing to its fast growth rate and easy natural regeneration as well as its suitability to be used in the production of pulp and plywood. Nevertheless, from the aspect of durability and treatability, batai is non-durable, easily attacked by fungi, and not amenable to preservative treatment, especially the heartwood (Lim and Chung 2002). Fungal attack often occurs when the wood is moist, and therefore, efforts to reduce the moisture absorption of wood should be emphasised. An effective way to reduce the amount of moisture absorbed by wood is to modify the wood by either thermal treatment (Lee et al. 2018) or impregnating it with chemicals or resin (Leemon et al. 2015). Various polymers and resins have been used for chemically modifying wood; one of them is methyl methacrylate (MMA) (Zhang et al. 2006a,b).

The hygroscopicity changes of wood due to chemical modification are often assessed by wood moisture sorption isotherm analysis (Hill 2009). To fit the experimental

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sorption data, sorption isotherm models are always employed. For example, Xie et al. (2010a) modified Scots pine sapwood with glutaraldehyde and reported reduced EMC in the modified wood, owing to less moisture absorption in the polylayer water. The authors concluded that the modification has filled the nanopores of the wood cell wall due to the deposition of glutaraldehyde. In addition, the expansion of the nanopores was restricted by the crosslinking of glutaraldehyde and cell walls. Papadopoulos (2011) esterified elm wood using acetic and maleic anhydride, and the sorption behaviour of the modified wood was investigated. Both acetic and maleic anhydride reduced the hygroscopicity of the esterified elm wood, but acetic anhydride proved to be a more effective option owing to its smaller anhydride molecules. In another study, Papadopoulos et al. (2020) treated pine wood with chitosan polymer, and the sorption behaviour of the treated wood was investigated. The treatment with chitosan polymer-based compounds successfully reduced the hygroscopicity of the treated pine wood, and a 24% reduction of total sorption at saturation was recorded.

As mentioned earlier, MMA and styrene have been used in the modification of wood to improve its dimensional stability and biological durability (Gorbani et al. 2018; Li et al. 2013). A combination of styrene, MMA, and nanoclay with benzoyl peroxide as initiator was impregnated into batai wood, and this resulted in enhanced compression strength and thermal stability. In addition, smoother surface morphology was also observed (Rahman et al. 2013). However, to the best of the authors' knowledge, the sorption properties of wood treated with MMA and MMA–styrene have not yet been studied. Therefore, the purpose of this paper was to determine the effect of MMA and MMA–styrene treatment on the physical, mechanical, and sorption properties of batai wood. In the current study, the hygroscopicity changes of the MMA- and MMA–styrene-modified batai wood was evaluated based on the HH model to find out the applicability of MMA treatment in reducing the moisture absorption of batai wood.

2 Materials and methods

2.1 Preparation of materials

Five-year-old batai wood was used in this study. The kiln-dried wood samples were supplied by Sarawak Forestry Corporation (SFC), Kuching, Sarawak. Meanwhile, MMA and styrene were obtained from a commercial chemical company in Kuala Lumpur, Malaysia. Batai wood was cut and planed into specimens with dimension of 20 mm (R) × 20 mm (T) × 300 mm (L) using a bandsaw and a single face-planing machine before they were conditioned in a conditioning room. Later, the specimens were coated with epoxy paint on both ends to prevent penetration in the radial direction to control solution uptake. Fifty control (untreated) samples were stored in a climate room with a temperature of 20 ± 2 °C and a relative humidity (RH) of 65 ± 3% until further use. Meanwhile, 100 samples were used for impregnation modification with monomers.

2.2 Treatment of batai wood

Impregnation procedures for the batai wood with monomers were based on Norul Hisham and Anwar (2005). Two different mixtures were prepared, namely MMA and MMA–styrene. MMA–styrene was prepared with the ratio of 50 MMA:50 styrene (w/w). Benzoyl peroxide (1%) was added as initiator. Fifty specimens were submerged in the mixture of MMA and MMA–styrene in a container and put inside a vacuum chamber. The samples were impregnated using vacuum and pressure process of 700 mmHg and 34 psi, respectively. The vacuum pressure was maintained for 1 h before the air in the vacuum chamber was slowly released within 1.5 h. Then, the treated samples were cured in an oven for a certain duration and temperature. Once cured, all the samples were stacked in a conditioning chamber with a temperature of 20 ± 2 °C and a RH of 65 ± 3% until 12% MC was attained. The mass of the samples was checked daily to make sure the desired MC had been achieved prior to property evaluation.

2.3 Fourier transform infrared spectroscopy (FTIR) analysis

FTIR was conducted to identify the functional groups existing in the treated wood due to the MMA and MMA–styrene treatment. The surfaces of the treated and untreated batai wood were scraped off and ground into powder form. Then, the wood powders were scanned with Perkin Elmer FTIR instrument (1 cm^{-1} resolution, 32 scans, KBr method) in transmittance mode over a spectral range of 450–4000 cm^{-1}. The FTIR spectra of untreated, MMA-treated, and MMA–styrene-treated batai wood was plotted using absorbance value derived from transmittance value. Using OriginPro 8.5 software (OriginLab Corporation, USA), baseline correction was conducted on the spectra by subtracting the baseline data. After baseline correction, the spectra were normalised into the range [0, 1] using OriginPro 8.5 software.

2.4 Determination of physical properties

The tests for density, water absorption (WA), and thickness swelling (TS) were conducted based on the procedures specified by Anwar et al. (2009). Test specimens having dimensions of 20 × 20 × 20 mm were prepared. The density of the specimens was calculated by dividing the mass over volume. Six specimens from every treatment were immersed horizontally (30 mm below the water surface) in the water for 24 h. The weight of soaked samples was measured immediately after removing excess water with a dry cloth. Thickness of the same specimens was also recorded to determine the TS after being soaked in water. Measurements were taken by using digital Vernier callipers with a precision of 0.01 mm. The WA and TS were calculated based on the difference of weight or thickness before and after water immersion, over the initial weight or thickness of the specimens.

2.5 Determination of mechanical properties

Static bending, namely modulus of rupture (MOR) and modulus of elasticity (MOE), of the treated and untreated batai wood were evaluated according to the central loading procedures specified in BS EN 375:1957 – methods of testing small clear specimens of timber. A total of 120 specimens (40 replicates for each treatment) having dimensions of 20 × 20 × 300 mm were tested. The test specimens were placed on an Instron Universal Testing Machine over a span of 280 mm. The loads were applied on the specimens with a constant loading speed of 11×10^{-5} m/s. The maximum loads that caused the failure of the specimens were recorded and used to determine the MOR value. Meanwhile, the loads at proportional limit and the resultant deflection at midspan were recorded and used to determine the MOE of the specimens. The limit of proportionality was taken as the point in the stress–strain diagram at which the curve deviates from the straight line.

2.6 Determination of sorption isotherms

Dynamic vapour sorption (DVS) intrinsic instrument (Surface Measurement Systems Ltd., London, UK) was used for analysis. The material was placed onto a cleaned sample pan which was carefully hooked onto the hanging wire connected to the microbalance. Nitrogen with a preset percentage RH was passed over the sample at a flowrate of 200 cm^3 min^{-1} at 25 °C. The schedule for the DVS was set to 10 different RH values (10, 20, 30, 40, 50, 60, 70, 80, 90, and 95%). The sample mass readings from the microbalance (every 20 s) revealed the vapour adsorption/desorption behaviour of the material. At each RH percentage, an inbuilt algorithm was set to ensure that pseudo-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}. Equilibrium moisture content (EMC) was determined using the following equation: EMC (%) = 100 [(W_c – W_o)/W_o] (Eq. 1), where W_o is the oven-dried weight (g) and W_c is the constant weight after reconditioning (g). To eliminate the effect of mass gain due to the deposition of modification agent, reduced EMC (EMC_R) was adopted. Then, EMC values obtained were adjusted using EMC_R (%) = EMC (1 + WPG) (Eq. 2) as specified by Thybring (2013), where WPG is the weight percent gain (%) of the samples after treatment. The EMC reported in this study was reduced EMC, which is denoted by EMC.

2.7 Statistical analysis

The changes in the physical and mechanical properties of untreated and treated samples were analysed using Statistical Analysis Software (SAS). Least significant difference (LSD) method was used to evaluate the significance level of the mean values at $p \leq 0.05$.

3 Results and discussion

3.1 FTIR analysis

The FTIR spectra of both treated and untreated wood are displayed in Figure 1. The absorption peaks at 3388, 1736, and 1238 cm^{-1} respectively correspond to the –OH stretching, C=O stretching of acetylated xylem, and C–O stretching of acetyl groups of the untreated wood (Yap et al. 1991). Two new peaks appeared at around 2995 and 2951 cm^{-1}, indicating the presence of ester functional group and those related to the methyl group of MMA-treated wood. These two peaks correspond to the C–H stretching of the methyl group, with the latter peak receiving some contribution from the C–H stretching of the methylene group (Yap et al. 1991). Similar to the findings reported by Yap et al. (1991), three peaks were observed in the region of 1390–1490 cm^{-1} for the MMA-treated wood, which could be attributed to the deformation of C–H bonds in the methyl group. C–O–C stretching of the ester group of MMA was also observed at the peak around 1148 cm^{-1}.

The peak observed at around 1736 cm^{-1} indicates the characteristic C=O stretching of the ester carbonyl in MMA. The intensity of the peak increased after the MMA treatment. Another peak that increased in intensity was around 1238 cm^{-1}, which corresponds to the ester bond (C–O) stretching vibration as the hydroxyl groups of the wood successfully reacted with MMA (Huang et al. 2019). As for

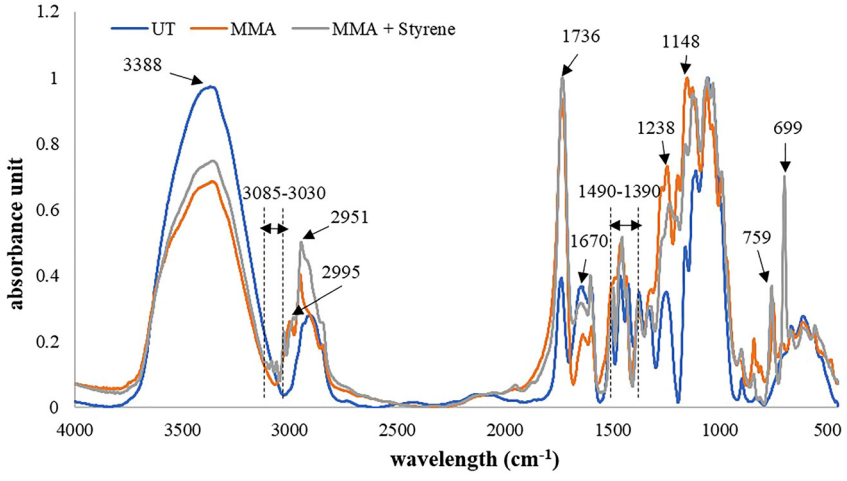

Figure 1: FTIR spectra of untreated wood (UT), MMA-treated wood and MMA–styrene-treated wood (3388 cm^{-1} = –OH stretching; 3085–3030 cm^{-1} = aromatic ring of styrene; 2995 and 2951 cm^{-1} = C–H stretching of the methyl and methylene group; 1736 cm^{-1} = C=O stretching of the ester carbonyl in MMA; 1670 cm^{-1} = stretching of conjugated aryl carbonyl groups; 1390–1490 cm^{-1} = deformation of C–H bonds in the methyl group; 1238 cm^{-1} = ester bond (C–O) stretching vibration; 1148 cm^{-1} = C–O–C stretching of the ester group of MMA; 759 cm^{-1} and 699 cm^{-1} = aromatic ring of styrene).

wood treated with MMA–styrene, new peaks in the region of 3030–3085 cm^{-1} were observed, which are attributed to the aromatic ring of styrene, as well as the peaks observed at 759 and 699 cm^{-1} (Yap et al. 1991). After the treatment with MMA and MMA–styrene, the OH groups of the untreated wood were reduced, as indicted by the reduced peak intensity at around 3338 cm^{-1}, which corresponds to the carbohydrates of polysaccharides. The finding is in agreement with Rahman et al. (2013), who suggested that new chemical bonds between styrene and MMA with cellulose are formed. Another peak that showed decrement after treatment is 1670 cm^{-1}, which corresponds to the stretching of conjugated aryl carbonyl groups.

3.2 Physical properties of treated wood

The average MC, WA, and TS values of both untreated and treated samples are shown in Table 1. The average MC of untreated samples was 9.7%, while the MC for MMA-treated and MMA–styrene-treated wood was 3.7 and 3.6%, respectively. The WA of batai wood was significantly reduced after the MMA and MMA–styrene treatment. A similar trend was observed for TS too. The MMA-treated batai wood exhibited lower WA and TS compared to those of the MMA–styrene-treated batai wood. The addition of styrene did not significantly improve the TS and WA of batai wood, but it could reduce the overall cost as it reduced the usage of MMA. The improvement in WA and TS may be due to the penetration of monomer into batai wood, where the resin occupied the lumen either as granules or in patches once the monomer has been cured. The deposition of polymer within the wood cell walls (bulking effect) leads to better dimensional stability as well as higher decay resistance (Anwar et al. 2009; Nabil et al. 2016; Xie et al. 2013). Apart from that, the reduction in WA is also caused by the increase in dry mass after the treatment, as increased dry mass automatically resulted in lower WA value. As the weight percent gain (WPG) of MMA- and MMA–styrene-treated batai wood in this study was 66.9 and 53.5%, respectively, the WA value in this study was adjusted by multiplying it with (1 + WPG) (Thybring 2013).

3.3 Mechanical properties of treated wood

Table 2 shows the mean values of density and mechanical properties of batai wood after impregnation with MMA and MMA–styrene. The density of treated batai wood increased almost twofold compared to the untreated samples. The untreated batai wood has a density of 272 kg/m^3. After the treatment, the density increased to 537 kg/m^3 for MMA-treated wood and 454 kg/m^3 for MMA–styrene-treated wood, indicating that the monomer was able to penetrate into batai wood easily. As for the mechanical properties, MOR and MOE increased significantly after treatment. The MOR of batai wood increased from 40.5 to 68.7 MPa when treated with MMA and to 58.7 MPa when treated with MMA–styrene. The same results were observed for the MOE values of treated and untreated batai wood. However, no significant difference was observed between MMA- and MMA–styrene-treated batai wood in terms of MOR and MOE. The higher MOR and MOE of treated wood than those of untreated wood might be attributed to the presence of resin in the wood (Leemon et al. 2015). The findings are in agreement with Rahman et al. (2013), who reported that the mixture of MMA/styrene/nanoclay filled the voids in the wood and subsequently improved the stiffness of the wood. Wood filled with polymers normally exhibits higher mechanical properties due to co-polymerisation of the wood cell wall (Hamdan et al. 2010).

Table 1: Reduced EMC, water absorption and thickness swelling of untreated and treated batai wood after 24 h soaking.

Treatment	Reduced EMC (%)	Water absorption (%)	Thickness swelling (%)
Untreated	9.7^{a} (0.2)	37.8^{a} (4.2)	1.98^{a} (0.67)
MMA	3.7^{b} (0.3)	15.8^{c} (3.9)	0.98^{b} (0.20)
MMA–styrene	3.6^{b} (0.5)	20.2^{b} (7.8)	0.96^{b} (0.31)

Values in parentheses are standard deviations; within the same properties row, mean values followed by different letters a, b, and c are significantly different at $p \leq 0.05$.

3.4 Isotherm plot of untreated and treated batai wood

The EMC for treated wood was corrected for the mass gain due to the modification as specified in Thybring (2013). Table 3 displays the experimental reduced EMC values

Table 2: Density and mechanical properties of untreated and treated batai.

Treatment	Density (kg/m^3)	MOR (MPa)	MOE (GPa)
Untreated	272^{a} (31)	40.5^{a} (4.2)	5.7^{a} (0.7)
MMA	537^{b} (145)	68.7^{b} (17.2)	6.6^{b} (1.0)
MMA–styrene	454^{c} (59)	58.7^{b} (14.6)	6.3^{b} (0.7)

Values in parentheses are standard deviation; within the same properties row, mean values followed by different letters a, b, and c are significantly different at $p \leq 0.05$.

Table 3: Experimental reduced EMC values obtained at various levels of RH for untreated and treated batai.

	Adsorption			Desorption		
RH (%)	Untreated	MMA	MMA–styrene	Untreated	MMA	MMA–styrene
10.0	1.72	0.76	0.76	2.46	0.89	0.83
20.0	3.18	1.36	1.38	4.38	1.71	1.71
30.0	4.44	1.907	1.93	6.13	2.47	2.47
40.0	5.64	2.46	2.44	7.80	3.18	3.21
50.0	6.86	3.03	2.92	9.53	3.92	3.93
60.0	8.26	3.68	3.43	11.45	4.71	4.73
70.0	10.06	4.43	4.05	13.69	5.60	5.66
80.0	12.72	5.35	5.01	16.48	6.57	6.67
90.0	16.94	6.66	6.64	19.40	7.48	7.65
95.0	20.73	7.91	8.80	20.73	7.91	8.08

obtained at various levels of RH for untreated and treated batai. The EMC for untreated batai wood was generally higher than that of treated samples in both isotherms. Isotherms of wood are generally divided into adsorption and desorption isotherms. A sorption isotherm for wood describes the relation between the EMC of wood and the ambient RH. Individual relationships between moisture content and RH for all samples are shown in Figure 2(a–c). The samples exhibited type II sigmoidal adsorption and desorption isotherms. The type II sigmoidal isotherm is well known for its association with many natural materials (Popescua et al. 2016). The EMC for desorption is always higher than absorption at a defined RH, and the phenomenon is known as sorption hysteresis.

Figure 3(a, b) present the differences in adsorption and desorption behaviour between untreated and treated samples when the curves were overlaid. As the wood samples were desorbed from an EMC below FSP, the desorption curve was called scanning curve, instead of boundary curve (Zaihan et al. 2009). Untreated batai wood exhibited higher EMCs on both adsorption and desorption compared with treated batai wood. At 95% RH, the EMC of untreated batai wood was 20.7% compared to that of the MMA- and MMA–styrene-treated batai wood, which recorded EMC values of 7.9 and 8.8%, respectively. However, between MMA- and MMA–styrene-treated batai wood, both patterns were plotted almost within the same line. This may be because the treatment reduced the sorption of batai due to lower accessible OH groups. However, the main reason would be the bulking effect caused by the treatment. MMA and MMA–styrene deposited and bulked the cell wall of batai wood and led to reduction in water uptake. It is well known that the modification of wood results in lower EMC compared to untreated wood (Dieste et al. 2010).

Hysteresis model describes the response of the cell wall matrix to the ingress or egress of water molecules under conditions of adsorption or desorption, respectively (Hill et al. 2009). Figure 4 presents the hysteresis between adsorption and desorption curves of untreated and treated

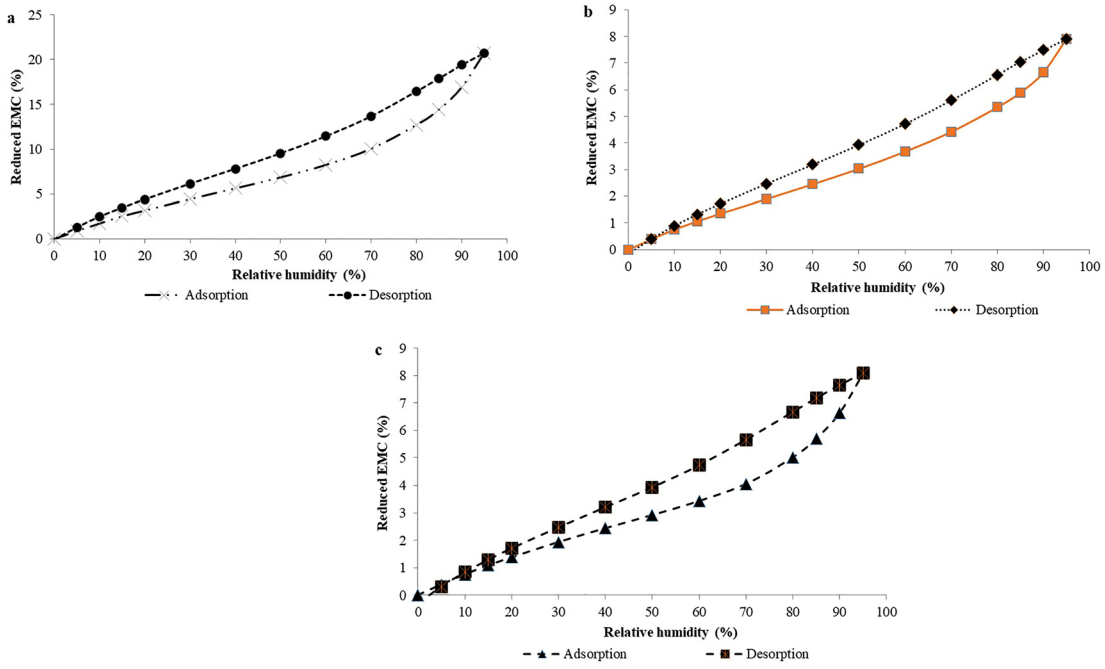

Figure 2: Experimental isotherms for (a) untreated (UT), (b) MMA-treated, and (c) MMA–styrene-treated batai wood.

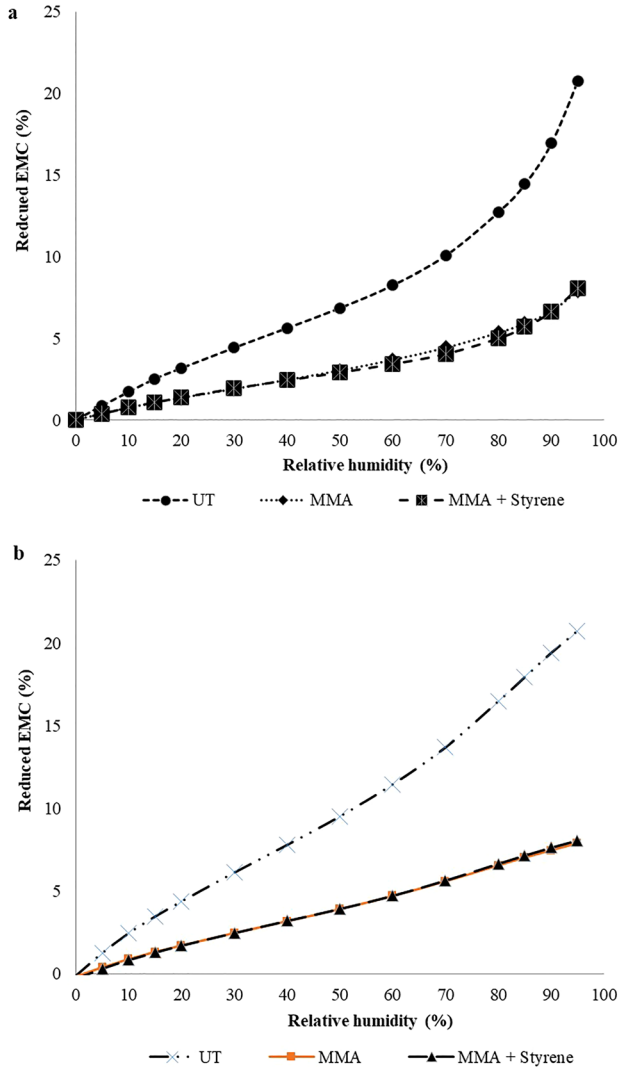

Figure 3: Comparing the moisture content behaviour during (a) adsorption and (b) desorption of the untreated (UT), MMA-treated and MMA–styrene-treated batai wood.

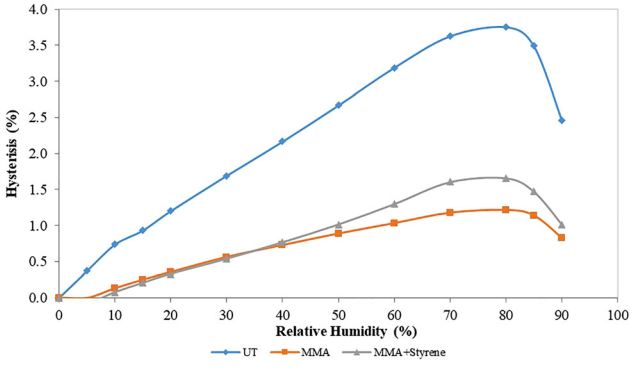

Figure 4: Hysteresis plotted for the untreated (UT), MMA-treated and MMA–styrene-treated batai wood at different relative humidity.

batai wood, which was obtained by subtraction of EMCs at different values of RH. Untreated batai wood had larger hysteresis loop compared to treated batai wood. The peak in absolute hysteresis was observed at 80% RH for untreated batai wood (3.8% EMC), followed by MMA–styrene-treated (1.7% EMC) and MMA-treated (1.2% EMC) batai wood. In this study, the treatment exhibited a reduction in hysteresis by means of low hysteresis values in MMA- and MMA–styrene-treated batai wood. A similar finding was also observed by Zaihan et al. (2010) and Hosseinpourpia et al. (2017). Altered cell wall structure as a result of MMA and MMA–styrene treatment might be a probable reason for the reduced hysteresis of batai wood. Cell wall and crosslinking of cell walls caused by the incorporation of MMA and MMA–styrene led to an increment in wood stiffness after treatment. Consequently, lower deformation occurred during the sorption process and resulted in lower hysteresis (Jakes et al. 2019; Xie et al. 2010b).

However, it should be noted that the assessed hysteresis is not between desorption and absorption isotherms. Instead, hysteresis between scanning isotherm and the absorption isotherm was assessed in this study. The results might have been complicated by the application of scanning isotherms instead of desorption isotherms and may lead to misinterpretations of the mechanisms behind sorption hysteresis (Fredriksson and Thybring 2018). Fredriksson and Thybring (2018) noted an obvious difference between the sorption hysteresis calculated from desorption isotherms and scanning isotherms, where the former resulted in linear absolute sorption hysteresis but nonlinear curves for the latter. Apart from that, a peak in absolute hysteresis was observed, and it would be absent if desorption isotherms were used. The peak in absolute hysteresis when using scanning isotherms was recorded at around 75% RH when desorption was initiated from 95% RH, which is very close to the findings in this study (80% RH). Fredriksson and Thybring (2018) also pointed out in their work that the use of scanning isotherms in interpreting the sorption hysteresis is not necessarily less correct, depending on the theory that one employs.

4 Conclusions

Batai wood was treated with MMA and MMA–styrene, and the improvement in the physical and mechanical properties of the treated wood over untreated wood indicates that the treatment is applicable in treating batai wood. Apart from reduced WA and TS, MOR and MOE of the treated wood were also enhanced. MMA was a slightly better treatment than MMA–styrene based on the evaluated

properties. Moreover, MMA and MMA–styrene proved to be effective treatments for batai wood as they reduced its hygroscopicity. Different adsorption/desorption behaviours of untreated and treated batai wood were observed in this study. The MMA-treated batai wood showed lower level of hysteresis followed by MMA–styrene-treated batai wood and untreated batai wood.

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