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ORIGINAL

Effect of ACQ treatment on surface quality and bonding performance of four Malaysian hardwoods and cross laminated timber (CLT)

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Abstract

The objective of this study is to determine the effects of alkaline copper quaternary (ACQ) treatment on the surface quality and bonding performance of four Malaysian hardwood species, namely batai, sesenduk, rubberwood and kedondong. The samples were impregnated with 2% ACQ preservatives and bonded with phenol-resorcinol–formaldehyde resin for cross laminated timber (CLT) fabrication. The changes in density and the retention of both ACQ and copper after the treatment were recorded. Surface roughness and wettability of both treated and untreated samples were measured. Block shear and delamination tests were performed to evaluate the bond-line strength of CLT. The study revealed that the average surface roughness (Ra) of each species increased significantly. Wettability of batai, sesenduk, rubberwood and kedondong was significantly higher than that of the untreated samples suggesting an improvement in surface wettability. For single-species CLT, treated rubberwood has the highest shear strength followed by kedondong, sesenduk and batai with values of 9.53 N/mm^2, 6.00 N/mm^2, 5.68 N/mm^2 and 4.19 N/mm^2, respectively. While for mixed-species CLT, the combination of ACQ-treated rubberwood-sesenduk-rubberwood has the highest block shear strength with a value of 8.05 N/mm^2. No delamination was observed from all samples. ACQ treatment was found to not affect the block shear strength significantly. Therefore, ACQ preservatives can be used to produce CLT with good bonding performance.

1 Introduction

A shift in interest from forest hardwoods towards fast-growing planted hardwood is getting much attention from researchers in Malaysia (Ong 2018). Batai, sesenduk, rubberwood and kedondong are Malaysia's hardwood species that are categorised as relatively fast-growing species. The availability of these species and its fast growth rates created the opportunity to explore the strength and potential uses (Zaidon 2017). Cross-laminated timber, also known as CLT, is a prefabricated multilayer engineered wood product that consists of structural panels made up of several layers of crosswise stacked boards and glued together on their faces (Dugmore et al. 2019). The development of CLT from tropical hardwoods in Malaysia may be an option to give added value towards underutilised hardwoods in Malaysia, to reduce the construction costs and to increase the profit margins of local plantation owners. A previous study by Hamdan et al. (2016) on CLT using underutilised hardwood sesenduk timber shows that lesser-known species can provide adequate strength for construction. Another study by Yusof et al. (2019) on CLT manufactured using *Acacia mangium* revealed that the mechanical properties of such species are more or less the same as with other works.

CLT is considered as a sustainable and renewable material having low carbon footprint due to the ability of the panels to store impressive carbon amounts (Scarlet 2015). CLT is a solid wood construction product having positive eco-balance accompanied by long-lasting value, flexible and creative design without a grid pattern as well as excellent static properties. Apart from superior fire resistance, it also exhibits good thermal and sound insulation characteristics.

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Laminated timbers are usually untreated due to interference in adhesion and bonding concerns. However, in tropical regions, wood is easily degraded either by the environment or biological attack. Preservative treatment is the solution to protect the wood. Bonding quality evaluation tests proposed by CLT standards have been developed for glulam, but problems arose as the experts found that the testing requirements are too severe for CLT, especially for treated CLT. Further analysis and evaluation have to be considered and will be beneficial for further use.

A rise in low mammalian toxicity and environmentally friendly water-based preservatives has caused other chemicals such as creosote oil, pentachlorophenol and coal tar to be abandoned in many countries. Chromated copper arsenate (CCA), a most popular exterior-grade wood preservative, also faced a decline in usage due to toxicity concerns, even though it is low in cost and has high efficiency (Lorenz and Frihart 2006). ACQ has comparable effectiveness as CCA and is now used as an alternative to replace CCA in providing long term protection against decay and rot, as well as insect. Besides they are also biodegradable in the soil after some time. This preservative has good permeability in wood, resistance to leaching in water, long term efficiency and low toxicity (Goodell et al. 2007; Qin et al. 2019).

Upon treatment, however, the surface of the wood normally becomes rougher and lower in wettability due to the raised fibres and chemicals being deposited into the wood. Such effects could, to some extent, affect the bonding ability of the surface (Qin et al. 2019). Ozdemir et al. (2015) reported that surface roughness and adhesion strength are dependent on wood species and the chemical composition of preservatives. They found that waterborne wood preservatives increased the surface roughness of wood due to increased surface porosity and raised fibres on the surface of wood cells. However, organic borne wood preservatives decreased surface roughness of wood due to the filled surface cavity and decreased surface porosity. Their results also revealed that while treatment with immersol has significantly reduced the adhesion strength, the effect was opposite for boric acid.

Aside from surface roughness, another prominent effect of preservative treatment is surface wettability. Ozdemir and Hiziroglu (2007) reported an increased adhesion of bleached, stained and preservative-treated beech and spruce samples, which can be attributed to their increased surface roughness after treatment. They concluded that contact angle is well correlated to surface wettability of wood and consequently exerts direct influence on the adhesion of different adhesives. Therefore, it is of utmost importance to understand the relation between surface roughness and wetting.

To date, some of the literature reported on the bonding performance of tropical hardwoods (Martins et al. 2019; Malek et al. 2019; Mohamad et al. 2019; Yusof et al. 2019; Srivaro et al. 2019) but very little was reported on treated tropical hardwoods (Shukla et al. 2019; Lim et al. 2020; Qin et al. 2019). Limited studies have been found on determining the bonding of wood treated with ACQ or CA-B. A study of hardwood glulam made up of beech, hard maple and red oak by Yang et al. (2012) found that high retention of ACQ reduced the modulus of elasticity of the treated wood, which resulted in poor bonding properties, but no significant difference between treated and untreated wood was observed. Studies on Japanese larch concluded that the preservatives did not adversely affect wettability or curing of PRF as measured by infrared spectroscopy, but it accelerated the curing as measured by torsional braid dynamical mechanical testing (Miyazaki et al. 1999). However, the gel time of PRF between untreated and treated wood was similar (Lorenz and Frihart 2006).

In a related study, these preservatives did not affect bond strength and delamination (Miyazaki and Nakano 2003). However, industrial laboratories have reported that bonding ACQ-treated wood is more difficult than bonding CCA-treated wood (Frihart 2003). Thus, a study was initiated to examine the adhesion and bonding strength of ACQ-treated hardwood in comparison to untreated hardwood using a PRF adhesive.

Besides, there are fewer data found on the adhesion and bonding parameters using mixed tropical hardwood timber. The parameters that shall be addressed including press pressure, surface roughness, wettability, delamination and shear test, glue spread and assembly time are critical to avoid starve joint and delamination. By understanding the adhesion and bonding between adjacent layers of mixed species, this will minimize the possibilities of shear failure and delamination when wood products are in service. In this study, four tropical hardwood species were selected based on their availability and density. All the wood belongs to light hardwood with a density below 620 kg/m^3. The woods were vacuum treated with alkaline copper quaternary (ACQ) and the effects of treatment on the surface roughness and contact angle of the treated wood surfaces were investigated. Single and mixed-species CLT were manufactured to study the bonding performance of treated laminated timber.

2 Materials and methods

2.1 Preparation of raw material

Four selected timber species, namely batai (*Paraserianthes falcataria*), sesenduk (*Endospermum malaccensis*), rubberwood (*Hevea brasiliensis*) and kedondong (*Canarium sp.*) were obtained from a commercial wood sawmill. The density of the wood was 220 kg/m^3, 500 kg/m^3, 590 kg/m^3 and 620 kg/m^3, respectively. The wood was kiln dried until it reached about 12% moisture content. The samples were cut and planned before treatment. ACQ compound was supplied by a local company. Approximately 2% of ACQ solutions [contained 33.3% alkyldimethylbenzyl-ammonium chloride (ADBAC) and 66.7% copper oxide (CuO)] were prepared by diluting 1 kg of ACQ from stock solutions in distilled water.

2.2 Treatment process

Wood samples of 20 mm × 20 mm × 300 mm in dimension and moisture content of 12% were used. Both ends of the wood samples were coated with oil paint. The treatment was carried out by using a vacuum pressure cycle according to MS360:2006. The samples were immersed in 2% ACQ solutions and pre-vacuum for 30 min at 85 kPa. Then, 520 kPa pressure was applied for 120 min. Final vacuum at 85 kPa for 30 min was applied again.

The retention of ACQ preservatives was calculated according to a previous study by Ozdemir et al. (2015). Ten replicates for each treatment group were used. The wet wood samples were then wiped lightly to remove excess ACQ solution and weighed (to the nearest 0.01 g) for gross retention determination. Below equation was used to calculate the retention of preservatives in the wood.

$$\text{Retention} = \text{G} \times \text{CV}\left(\text{kg m}^3\right) \quad (1)$$

where G: uptake of preservative (kg), C: preservative concentration (%), V: Volume of wood specimen (m^3).

All samples were stacked and dried under ambient condition for 2 weeks. The drying process was important to allow fixation of copper inside the wood.

2.3 Copper content determination

A commercial atomic absorption spectrophotometer (AAS) was used to determine the amount of copper oxide in the treated samples. The procedures were conducted according to MS 821 (2011). Wood samples were ground into fine sawdust. 0.5 g of wood samples was transferred to a 250 ml conical flask. Next, 20 ml sulphuric acid solution and 4 ml hydrogen peroxide solution were added. The solutions were heated at 75 ± 3 °C in a water bath for 30 min with occasional swirling to mix the contents in the flask. 40 ml of distilled water was added. Then, the solution was boiled for another 30 min. Following this, the samples were allowed to cool to room temperature and the solution was filtered through a filter paper into a 100 ml volumetric flask. 10 ml of sodium sulphate solution was added and the mark was made up with distilled water. The samples were then analysed by AAS and copper content percentage was calculated.

2.4 Determination of wood density

Twenty pieces of wood samples from each species (ten for each untreated and treated) of size 20 × 20 × 20 mm were placed in the oven at 103 ± 3 °C until they reached a constant weight. The oven-dry density of each sample was then determined by using the water immersion method after 24 h.

2.5 Surface roughness

The Mitutoyo Surfest SJ-301 was employed for the surface roughness measurement. Average roughness (Ra) and mean peak-to-valley height (Rz) were standard roughness parameters that can be calculated from digital information. The Ra roughness parameter was measured to evaluate surface roughness of the surface of untreated and treated samples according to previous studies by Hiziroglu et al. (2008). At a constant speed of 1 mm/s, the stylus traverses over 15 mm tracing length. A total of three roughness measurements across the grain orientation with a span of 40 mm were taken from each sample. Ten replicates were used for each group to evaluate the surface roughness. The surfaces were then observed under scanning electron microscope (SEM).

2.6 Contact angle measurement

First Ten Angstroms FTA 1000 was employed for contact angle measurement. All samples were conditioned at 20 ± 2 °C and 65 ± 3% relative humidity for a week before testing. Twenty samples of 4 mm × 20 mm × 40 mm in size from each species were prepared for the measurement. Distilled water was dropped onto the surface of untreated and treated samples. When the water droplet dropped on a wood surface, an elliptical shape was formed with elongation in the direction of the wood fibres. The angle between droplet and wood surface was measured after 2 s. It was continuously measured until the contact angle value became zero. The contact angle was determined for each image by the digital image analysis software. The mean contact angle value and standard deviation for each sample were calculated.

2.7 Preparation of CLT

CLT panels with dimension 300 mm × 300 mm × 75 mm thickness were produced for both ACQ-treated single-species and mixed-species. Untreated single-species CLT was manufactured as a comparison. The configurations for mixed species CLT were based on the density of that particular species when glued together, where batai and sesenduk having low density were positioned at the centre of CLT. The layers were bonded into 3-ply with 90° perpendicular to the grain. Phenol-resorcinol–formaldehyde (PRF), a commonly used structural adhesive was used in this study. Each layer was bonded by PRF at a ratio of 3:1 (resin: hardener) according to the manufacturer`s recommendation. Hardener used was in powder form and mixed thoroughly with resin. Two different pressures (0.7 N/mm^2 and 1.4 N/mm^2) and three different glue spread rates (200 g/m^2, 250 g/m^2 and 300 g/m^2) were used to study the influence of different parameters on the bonding performance of untreated and ACQ-treated CLT. A cold press machine was used for edge bonding and face bonding of assemblies and the pressure was retained for 3 h before being conditioned in the conditioning room at 65 ± 5% RH and 20 ± 2 °C at least one day prior to cutting into specimens for delamination and shear tests. The samples were then stored in the conditioning room for several weeks before testing. Configurations for both single- and mixed-species are shown in Fig. 1, and the panel layup and assembly of CLT are shown in Fig. 2.

2.8 Block shear test

The shearing behaviour was determined in a block shear test according to BS EN 16351:2005. The specimens were tested parallel to the grain with the shear plane corresponding to the adhesive layer. The shear test was performed with a universal testing machine, INSTRON with a displacement rate

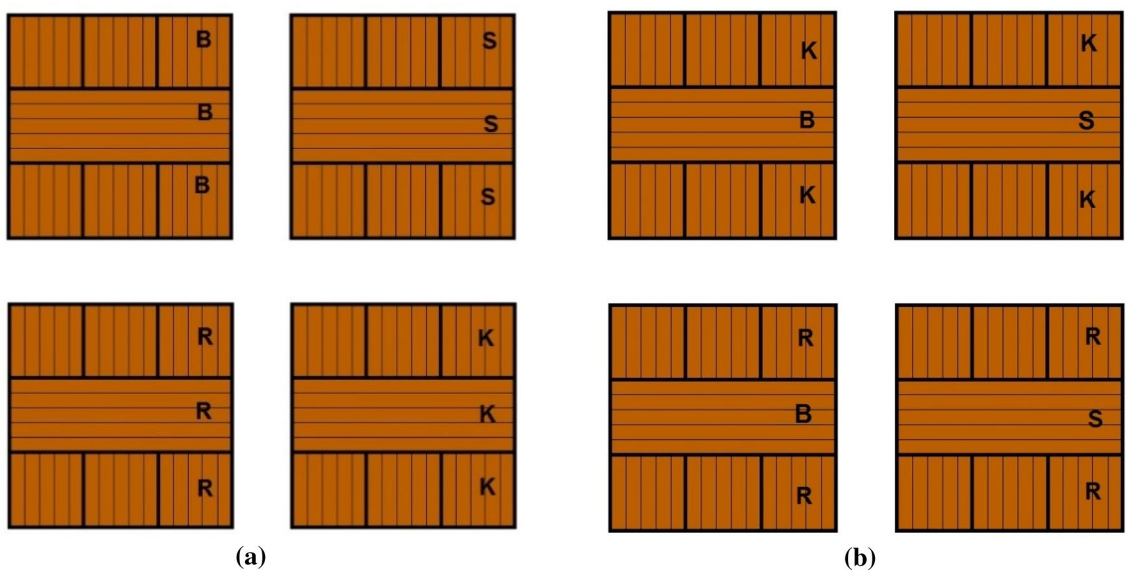


Fig. 1 Type of configuration for **a** single-species and **b** mixed-species CLT. *B* Batai, *S* Sesenduk, *R* Rubberwood, *K* Kedondong

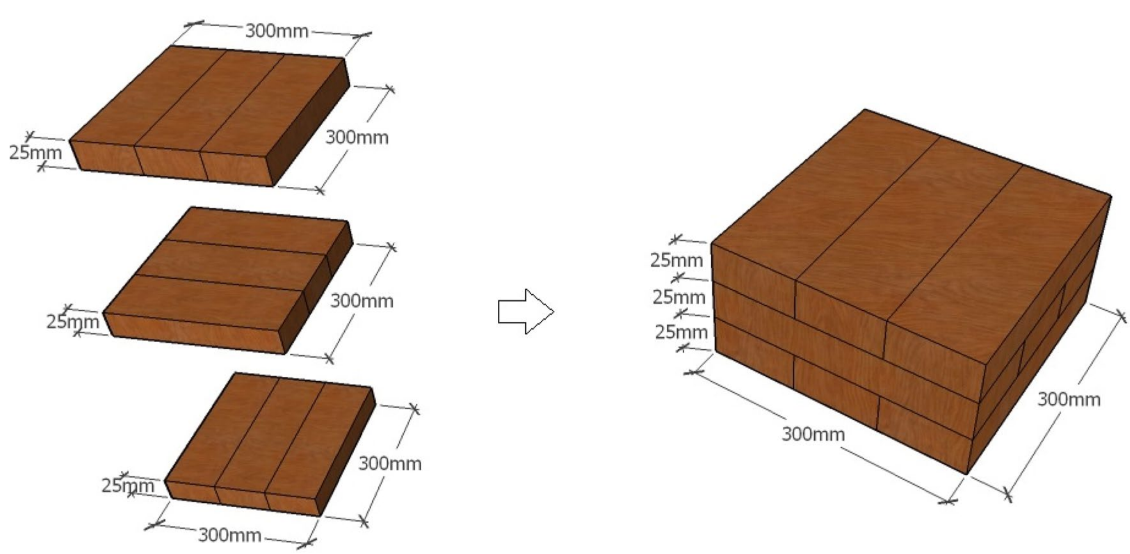


Fig. 2 Panel layup and assembly of CLT

of 1.8 mm/min. The shear strength, fv was determined for each glue line and calculated using the following formula.

$$fv = f\mu A \quad (2)$$

where fv is shear strength (N/mm^2), fμ is pure shear strength/ultimate shear strength (N), A is sheared area (mm^2).

All specimens were evaluated according to BS EN 16351 (2015) (Annex D) to assess their conformity to the requirements stipulated in the standard. The requirements for pass shear are as follows: The shear strength (fv) of each glue line must be at least 1 N/mm^2.

2.9 Delamination test

The samples were cut from both ends of each CLT board and the glue lines were measured. For soaking delamination test, the specimen were immersed in water at room temperature condition of 10–20 °C, with 30 min vacuum. Then, the pressure was applied for 2 h and then oven-dried in the oven at 70 °C until completely dried. After the test, the lengths of the glue line openings were determined. The length of the glue line opening was determined by inserting a thin metal probe between the two delaminated surfaces and only counted if the delamination depth was more than 2.5 mm. Total delamination ($Delam_{tot}$) and maximum delamination ($Delam_{max}$) of each test piece were calculated using Eq. (3).

$$Delam_{tot} = 100\frac{l\ tot,delam}{l\ tot,glue\ line}\ (\%)$$
$$Delam_{max} = 100\frac{l\ max,delam}{l\ glue\ line}\ (\%) \quad (3)$$

where $l_{tot,\ delam}$ is the total delamination in length (mm), $l_{tot,\ glue\ line}$ is the sum of the perimeters of all glue lines in a delamination specimen (mm), $l_{max,\ delam}$ is the maximum delamination length (mm), $l_{glueline}$ is the perimeter of one glue line in a delamination specimen (mm).

All specimens were evaluated according to BS EN 16351 (2015) (Annex C) to assess their conformity to the requirements stipulated in the standard. Bond strength was considered sufficient and "Pass Delam" if:

1. Maximum delamination (D_{max}) length did not exceed 40% of the total length of each glue line or,
2. Total delamination (D_{tot}) length did not exceed 10% of the sum of both glue lines.

2.10 Statistical analysis

Statistical analysis was carried out using SPSS programming software. Analysis of variance (ANOVA) was conducted to analyse any differences in surface properties of untreated and treated sample studied. If the differences were significant, least significant difference (LSD) test was used to determine which of the means were significantly different from one another. On the other hand, three-level analysis using General Linear Model (GLM) was performed with input factors species, glue spread and pressure to show the effect of the variables on the response variables shear strength (fv) and total delamination (D_{tot}) as well as their interactions with one another.

Table 1 Analysis of variance (ANOVA) for the effects of treatment and species on the physical properties of hardwood species

Source	df	Density	Surface roughness	Contact angle of wettability
Treatment	1	0.000**	0.000**	0.008**
Species	3	0.000**	0.000**	0.000**
Treatment × species	3	0.000**	0.000**	0.000**

df degrees of freedom

**Significantly different at $p \leq 0.05$

Table 2 Average density and density increment of four Malaysian hardwood species before and after treatment with ACQ

Wood	Density (kg/m^3)		Increase in density (%)
	Untreated	Treated	
Batai	218.87 (5.25)[g]	259.86 (3.25)[f]	15.76[a]
Sesenduk	501.46 (7.58)[e]	513.4 (2.15)[d]	2.4[b]
Rubber	589.52 (2.69)[c]	602.66 (3.12)[b]	2.2[b]
Kedondong	619.20 (6.61)[a]	624.69 (4.52)[a]	0.88[b]

Means followed by the same letters a, b, c, d, e, f and g are not significantly different at $p \leq 0.05$ according to Least Significant Difference (LSD) test. Numbers in parentheses are standard deviations

3 Results and discussion

Table 1 displays the effect of treatment and species used on the physical properties of Malaysian hardwood species. All the examined properties were found to be significantly affected by the treatment and species used ($p \leq 0.05$). These effects were further analysed by the least significant difference (LSD) method and the results are tabulated in Tables 2, 3 and 4.

3.1 Density, retention and copper content

Table 2 shows the density of untreated and treated hardwood samples. The density of untreated batai, sesenduk, rubberwood and kedondong ranged from 200 to 620 kg/m^3. The density was highly influenced by anatomical features. Batai has the lowest density among the species followed

Table 3 Average ACQ retention and copper content of four Malaysian hardwood species

Wood	Fibre lumen diameter[1]	Vessel diameter[1]	Retention (kg/m³)	Cu content (w/w %)
Batai	24.20	158.18	14.84 (0.35)[a]	0.30 (0.04)[a]
Sesenduk	33.46	161.14	11.85 (0.80)[b]	0.21 (0.03)[b]
Rubber	15.20	177.09	8.68 (0.56)[c]	0.11 (0.00)[c]
Kedondong	18.53	164.68	9.57 (1.19)[c]	0.13 (0.01)[c]

Means followed by the same letters a, b, c in the same column are not significantly different at $p \leq 0.05$ according to Least Significant Difference (LSD) test. Numbers in parentheses are standard deviations

[1]Syahirah et al. (2019)

by sesenduk, rubberwood and kedondong. This might be because batai has short fibre length and thin fibre wall thickness in comparison to the other species. According to Dean et al. (2002), fibre length and fibre wall thickness exert high influence on the density of the wood. This result was also supported by Ziemińska et al. (2013) and Syahirah et al. (2019) who found a high correlation between density and anatomical features of the wood.

Upon treatment with ACQ, the density of the samples increased in the range of 0.88–15.76% depending on the species. As shown in Table 2, batai experienced the highest increment in density followed by sesenduk, kedondong and rubberwood. Batai having the lowest density showed a density increment up to 15.8%, while kedondong having the highest density increased by only 0.88%. On the other hand, for sesenduk and rubberwood, the density increases were 2.4% and 2.2%, respectively. According to Lee and Ashaari (2015), the density of wood influences the preservative uptake, which could be related to the anatomical structure that influences the penetration of ACQ into the wood (Cooper 1998), where low-density wood has more voids leading to greater solution uptake (Halvenson and Lebow 2011).

Significant difference was found among the species at $p \leq 0.05$ and it appears that both species and density had a great influence on the amount of chemical retention and copper content inside the treated wood. Average retention and copper content are shown in Table 3. All samples met the minimum requirement of retention stipulated in MS 360 (2006), which is 4 kg/m³ for above-ground usage. With regards to density, batai has the highest retention (14.84 kg/m³) and copper content (0.30 w/w%) followed by the other species. Batai (density 218 kg/m³) shows high retention among the species probably due to the vessel of batai, which is diffuse-porous and has a high surface roughness. Moreover, wood species with lower density tends to obtain higher chemical uptakes and vice versa (Temiz et al. 2005). Thus, higher chemical uptake was observed in batai compared to other species.

Table 3 shows the fibre lumen diameter of all species. Syahirah et al. (2019) found that batai and sesenduk have larger lumen diameter compared to rubberwood and kedondong. Total numbers of vessels and their diameter were extremely important for impregnation because these characteristics influenced wood permeability (Wiedenhoeft and Miller 2005). Similar findings by Baraúna et al. (2004) and Martha et al. (2019) also found that the penetration of preservatives was fast due to the large diameter of vessel lumens and the penetrability was highly dependent on the close or open conditions of the vessels or pores. This explained the high retention obtained by batai and low retention obtained by rubberwood, as batai has a larger lumen diameter in comparison to rubberwood (Syahirah et al. 2019).

On top of that, both rubberwood and kedondong contain tyloses, which consequently limit the chemical uptake, as the tyloses tend to block the chemicals from penetrating the wood cells (Syahirah et al. 2019). According to Baraúna et al. (2004), denser wood contains less air space thus it holds fewer preservatives compared to batai and sesenduk. Interestingly, rubberwood had lesser chemical uptake than kedondong, even though it has a lower density. Rubberwood has the highest vessel diameter with a narrow lumen diameter (Lim et al. 2002), which explained the lesser chemical uptake compared to kedondong. Vessel diameter and lumen diameter play an important role. The larger the lumen diameter, the easier the chemical penetration and the higher the number of chemicals to be retained in the treated wood.

3.2 Surface roughness

The evaluation profile of each wood species between untreated and treated samples is shown in Fig. 3. The average roughness (Ra) of untreated samples ranges between 3.0 and 9.0 µm. Among the four hardwood species, batai has the roughest surface, while kedondong has the smoothest with an average Ra value of 8.26 µm and 3.78 µm, respectively. The surface roughness differences between the species can be related to their differences in density. These findings are supported by Hiziroglu et al. (2008), who found that low-density wood gave high roughness values compared to high-density wood. When comparying treated and untreated samples, the roughness profile of the untreated wood has

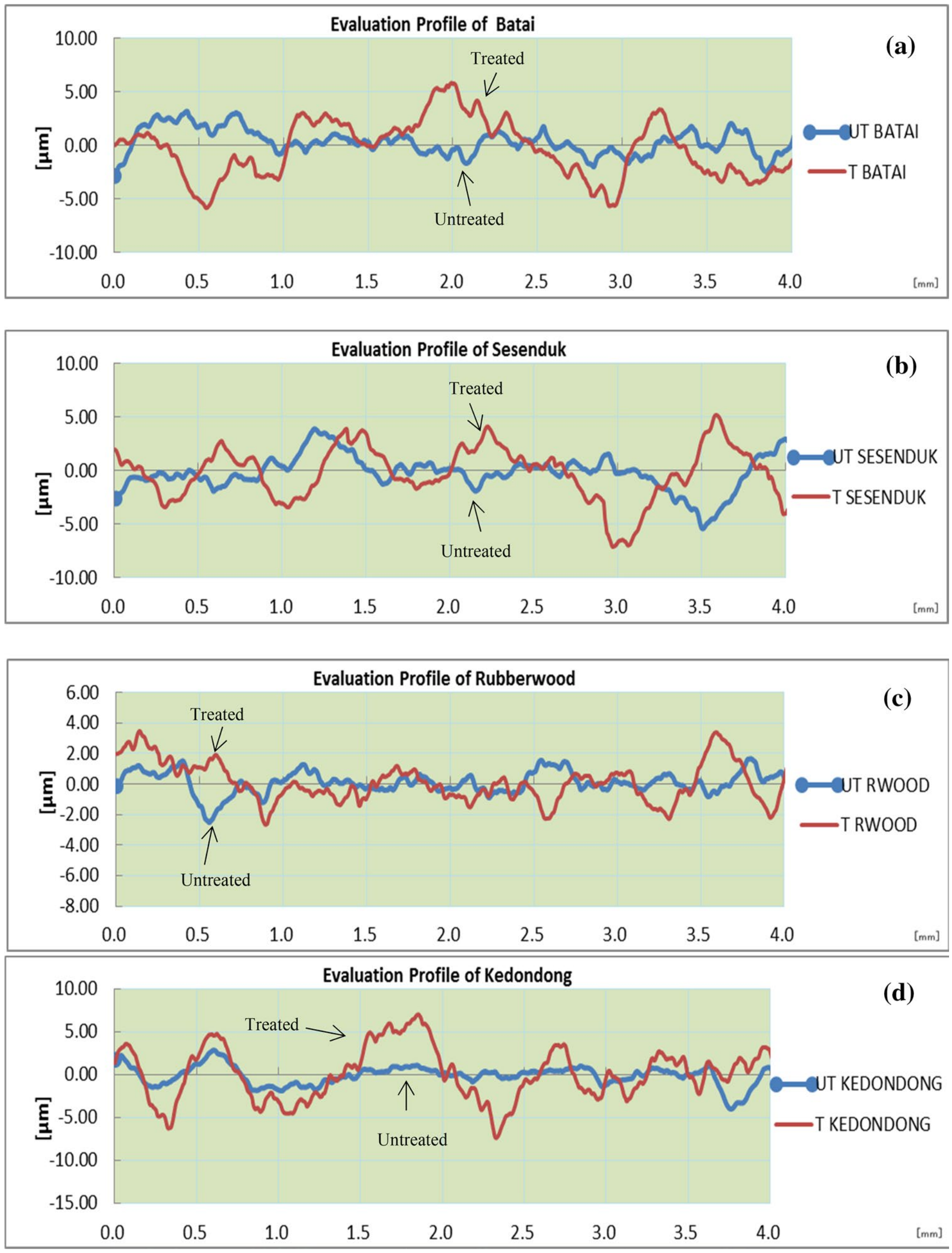


Fig. 3 Typical surface roughness profile of untreated and treated Malaysian hardwood species, **a** Batai, **b** Sesenduk, **c** Rubberwood, **d** Kedondong

lower waviness compared to that of the roughness profile of the treated samples.

Surface roughness varies between wood species due to the anatomical differences in hardwood (Turgay and Hiziroglu 2007; Alia Syahirah et al. 2019). Similar findings by Kilic et al. (2006), Kilic (2015) and Ozcan et al. (2012) reported that rougher surface was mainly due to porous anatomy, while the smoothest surface was mainly attributed to uniform grain orientation. Syahirah et al. (2019) found that the size of voids in low density wood such as batai and sesenduk influences the roughness of the wood surface. Moreover, the large lumen diameter of batai and sesenduk also contributed towards open grain after machining that makes the surface rougher (Kilic et al. 2006). Batai and sesenduk having density of 218.87 kg/m^3 and 501.46 kg/m^3, respectively with the absence of tyloses have larger pores that lead to their rougher surface. On the contrary, rubberwood and kedondong have large vessel diameter with narrow lumen diameter and consequently a smoother surface. It is reported that the roughness of wood is a complex phenomenon since wood is an anisotropic and heterogenous material. Factors such as machining properties, growing characteristics of species, anatomical differences and pretreatments applied to wood before machining must also be considered (Aydin and Colakoglu 2005).

The roughness of untreated and ACQ-treated samples was observed under SEM and is shown in Fig. 4. As observed from Fig. 4, the surface of ACQ-treated wood looks rougher than that of untreated samples. The increase in surface roughness for treated samples may be due to the presence of ACQ that tends to increase the surface porosity and raise the fibres on the wood surface after the drying process (Turgay et al. 2015). The increase in roughness could also be attributed to the second drying of wood (Knorz et al. 2015). Similar findings by Baysal et al. (2016) reported that copper-based preservatives increased the value of surface roughness of the treated wood samples compared to untreated wood samples. Besides, the copper in ACQ reacts with wood. According to Temiz et al. (2005), copper-wood interactions after treatment can be considered as a factor that contributes to the roughness value. These findings were in agreement with those reported by Jiang (2000) that ACQ treatment reduces the degradation of the wood surface, which contributes to a relatively important modification of wood surfaces' roughness when copper in ACQ forms some complexes with wood components (Temiz et al. 2005).

Table 4 shows the average surface roughness (Ra) between untreated and treated samples. A highly significant difference was found in the interaction between treatment and species at $p \leq 0.05$. LSD tests conducted show that the surface roughness of the samples was statistically significant between species. With regards to density, batai has the lowest increase in roughness followed by sesenduk, rubberwood and kedondong. Ra of ACQ-treated samples increased from 10 to 48% compared to the untreated samples, suggesting that the wood species (density) as well as preservative concentration influenced the surface roughness of wood (Turgay et al. 2015).

3.3 Contact angle

The initial contact angles of distilled water on the surface of untreated and ACQ treated samples are shown in Fig. 5. For untreated wood, batai has the lowest initial contact angle followed by sesenduk, rubberwood and kedondong with contact angle values of 46.76°, 58.26°, 59.64° and 69.05°, respectively. As mentioned earlier, batai with the lowest density and the roughest surface resulted in the lowest initial contact angle. The low contact angle of batai indicates that it has high wettability, thus water was absorbed in a short time. Wood is a porous and hygroscopic material, therefore when distilled water begins to contact the wood surface, wood starts to absorb the liquid (Cao et al. 2005). In the case of high-density wood with smooth surface, such as kedondong, high contact angle values were recorded. High contact angle indicates low wettability and therefore the liquid remains longer on the surface and spreads slower.

As shown in Fig. 5, the effect of ACQ treatment on the initial contact angle depends on the ACQ solution and density of the hardwood species used. Wood treated with ACQ shows lower initial contact angle compared to untreated wood. ACQ treatment exerted significant effect on the value of initial contact angle as the contact angle value decreased after treatment at 2% ACQ concentration.

It is interesting to note that there is a close relationship between surface roughness and wettability. Another study reported that a lower surface free energy generates a higher contact angle, and vice versa (Martha et al 2019). The higher the surface free energy of the woods, the higher the energy on the surfaces of the wood to be used to breakdown the liquid of the adhesive to spread and penetrate on the surfaces. The value of surface free energy tends to increase as the surface roughness of the wood increases (Qin et al. 2014). Yuningsih et al. (2019) reported that the decrease in the roughness of teak wood causes a decrease in the surface free energy value. Cao et al. (2005) also reported that the high surface free energy of ACQ treated Chinese fir causes low initial contact angle value of the wood.

According to Qin et al. (2014), the contact angle was formed and decreased with increasing time due to porosity and anisotropy of wood. Previous studies by Papp and Csiha (2017), Syahirah et al. (2019) and Qin et al. (2019) also stated that there is a relation between surface roughness and contact angle. Higher surface roughness normally resulted in good wettability, which was also observed by Gardner et al. (2016). The authors found that on differently rough wood surfaces, the wettability is highly influenced by polymer constituents of the cell wall. As the surface became rough, the contact angle value was reduced and the penetration of liquid became faster.

Figure 6 gives the contact angles of distilled water on the surfaces of untreated and ACQ treated wood versus equilibrium wetting time. By plotting all data, the declining slope of the contact angle with time was obtained. The slope can be defined as the rate of penetration. As shown in Fig. 6, the contact angles of untreated wood change very slowly with time especially for rubberwood and kedondong compared to the ACQ treated wood. It suggests a slow rate of penetration of distilled water into untreated wood surface. On the contrary, ACQ treated wood samples take less than 50 s to penetrate as batai, sesenduk, rubberwood and kedondong take only 28 s, 44 s, 14 s and 44 s, respectively to penetrate completely into the wood.

According to Qin et al. (2014), the interfacial tension becomes high after ACQ treatment as the wood surface becomes hydrophilic. Thus, the absorption of water droplet becomes faster and the contact angle value for treated samples decreases. Besides that, the presence of the copper compound in ACQ preservatives may also affect the wettability of the wood and in turn, increase the surface

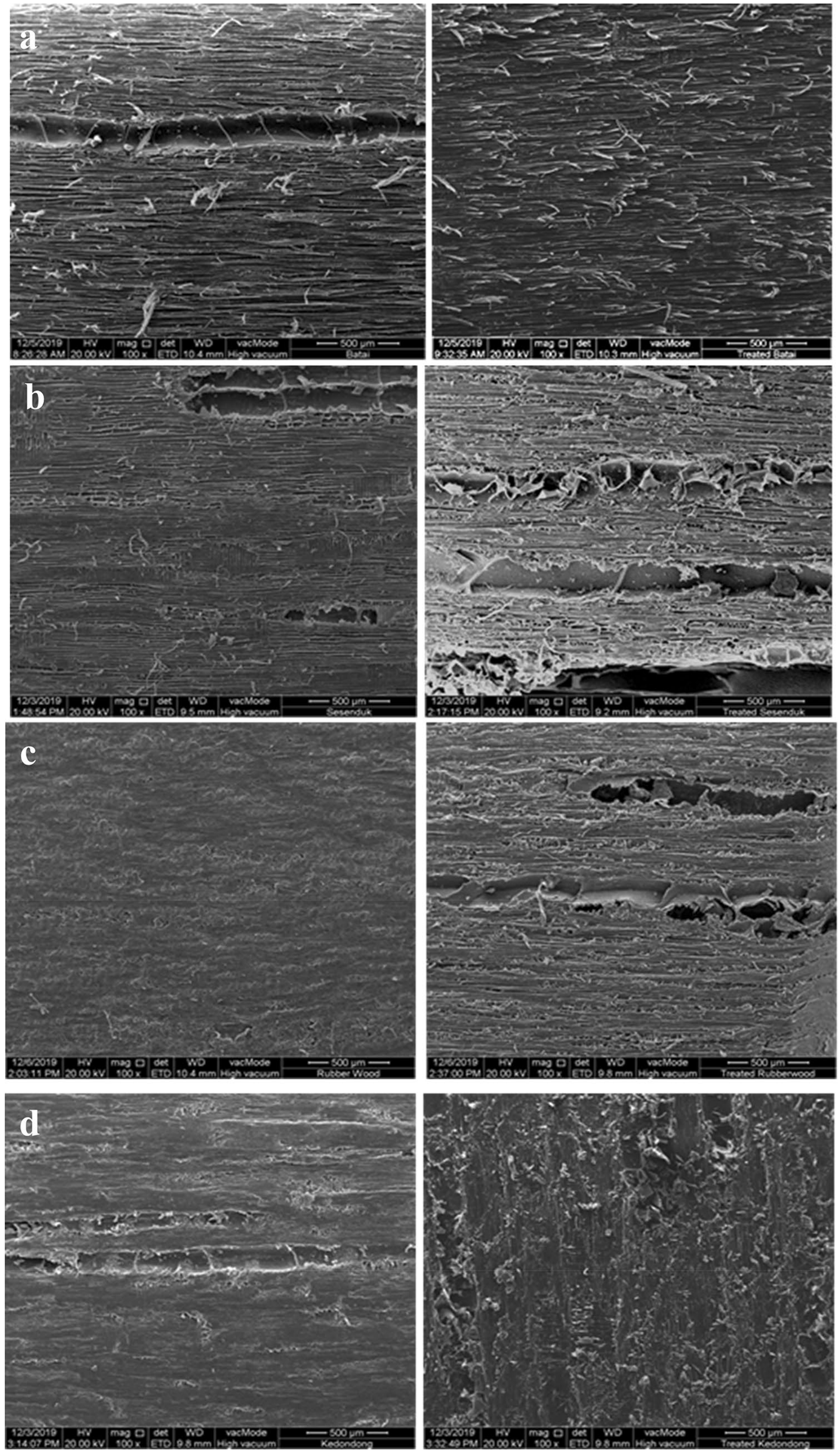


Fig. 4 Surface of untreated and ACQ-treated Malaysian hardwood species examined under SEM at ×100 magnification. **a** Batai, **b** Sesenduk, **c** Rubberwood, **d** Kedondong, (left): untreated, (right): treated

Table 4 Average value of the surface roughness (Ra) across the grain

Wood	Average surface roughness (µm)		Increase in roughness (%)
	Untreated	Treated	
Batai	8.41 (1.08)[b]	9.43 (1.58)[a]	10.81
Sesenduk	6.31 (0.55)[d]	8.03 (1.21)[bc]	21.42
Rubber	3.92 (0.66)[e]	6.45 (0.98)[d]	39.22
Kedondong	3.78 (0.30)[e]	7.31 (0.67)[c]	48.29

Means followed by the same letters a, b, c, d and e are not significantly different at $p \leq 0.05$ according to Least Significant Difference (LSD) test. Numbers in parentheses are standard deviations

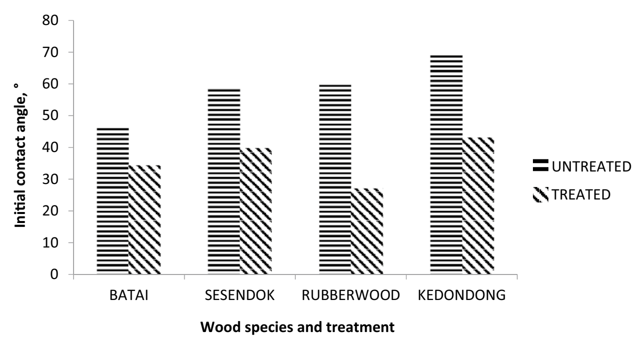


Fig. 5 Initial contact angle of untreated and ACQ treated samples of each hardwood species

energy. Wood treated with copper is usually more hygroscopic than untreated wood. Hashim et al. (1994) verified that there is a definite correlation between chemical treatments and water-uptake properties of wood. Variations in the chemical composition of wood may influence the sorption properties. Structural and chemical modification of wood cell wall constituents also may lead to the formation of additional hydrogen-bonding sites for water (Ayrilmis et al. 2009).

3.4 Bonding properties of CLT

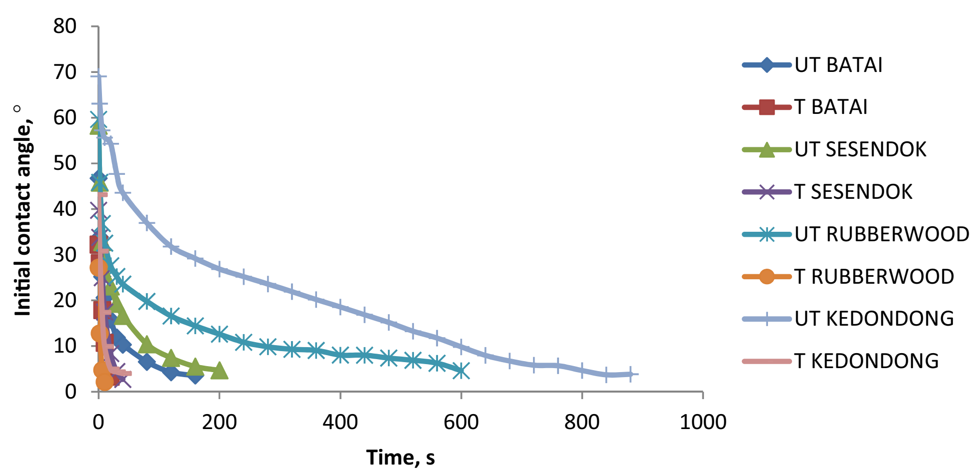
3.4.1 Block shear strength of single-species CLT

To understand the effect of ACQ preservative on the bonding performance of treated CLT, block shear and delamination tests were carried out with comparison to untreated CLT samples. Figure 7 presents the mean block shear strength of different CLT samples from different species manufactured at three different glue spread rates (200, 250 and 300 g/m^2) and two different pressures (0.7 and 1.4 N/mm^2).

According to Fig. 7, untreated batai and rubberwood recorded the highest block shear strength at 300 g/m^2, while sesenduk and kedondong at 250 g/m^2 glue spread rate. For ACQ-treated batai, sesenduk, rubberwood and kedondong, the highest block shear strength was recorded at 300 g/m^2. The highest block shear strength for untreated batai, sesenduk, rubberwood and kedondong were 3.82 N/mm^2, 4.82 N/mm^2, 7.48 N/mm^2 and 6.98 N/mm^2, respectively, while for ACQ-treated batai, sesenduk, rubberwood and kedondong, the highest block shear strength was 4.19 N/mm^2, 5.68 N/mm^2, 9.53 N/mm^2 and 6.00 N/mm^2, respectively. The changes in pressing pressure from 0.7 to 1.4 N/mm^2 did not significantly affect the block shear strength of all the samples.

Wood with high wettability, indicated by low contact angle, is often accompanied by increased block shear strength owing to the well-spreading of glue on the wood surface (Ahmad et al. 2017). Nevertheless, this is not the case in this study, as untreated batai with the lowest contact angle of 46.76° recorded the lowest block shear strength at every glue spread rate. What is more, ACQ-treated batai samples with lower contact angle of 34.43° displayed even lower block shear strength, except when fabricated at a glue spread rate of 300 g/m^2. This phenomenon could be explained by the high surface roughness of both treated and untreated batai samples. There is strong evidence that the

Fig. 6 Contact angle of untreated and treated Malaysian hardwood samples versus equilibrium wetting time



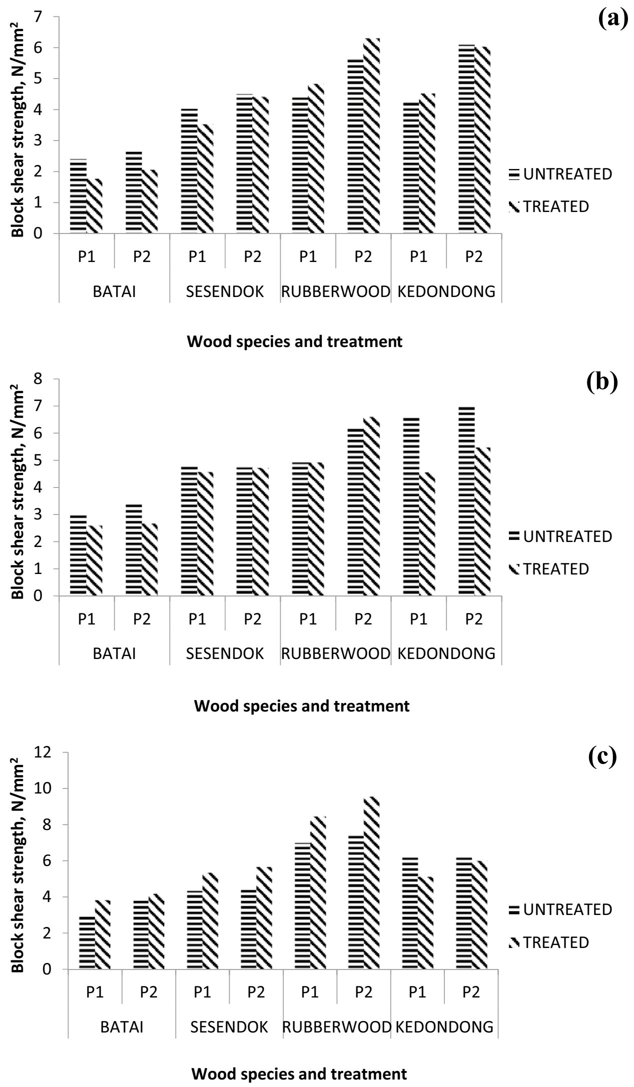


Fig. 7 Block shear strength of untreated and treated single-species CLT fabricated with **a** 200 g/m^2, **b** 250 g/m^2 and **c** 300 g/m^2 glue spread rate; P1 = 0.7 N/mm^2, P2 = 1.4 N/mm^2 pressure

roughness promotes adhesives dry out and over-penetration that resulted in poor quality and lack of intimate contact between wood and adhesive (Aydin and Colakoglu 2007). As the adhesives are less available on the bonding surface, it resulted in starve joints and reduced bond quality. However, CLT samples fabricated with ACQ-treated batai at 300 g/m^2 glue spread rate exhibited higher block shear strength than untreated samples. The low block shear strength at 200 and 250 g/m^2 glue spread rate could be attributed to the effect of insufficient adhesive spreading rate on the wood species because not enough adhesive material penetrates into wood at low level, and it formed a starved glue line, which has a negative effect on the shear strength. However, with the increasing adhesive spreading rate, more adhesives are able to penetrate the wood, which caused a continuous and stable glue line, and the shear strength increased. Nevertheless, it should be noted that too much adhesive may result in a thick glue line leading to a negative effect on the gluing performance, hence decreasing the shear strength (Sikora et al. 2016; Lorenz and Frihart 2006).

A similar trend was also observed for CLT samples fabricated with treated and untreated sesenduk. However, as for kedondong, ACQ-treated samples displayed lower block shear strength than untreated samples even at the highest glue spread rate of 300 g/m^2. This finding maybe due to the fact that kedondong has the highest increment in surface roughness value (48.29%) among all the species after ACQ treatment. In a similar way, the increment in surface roughness may cause the starved glue line due to over-penetration and eventually reduced the block shear strength of kedondong CLT. Interestingly, rubberwood CLT behaves completely different from the other wood species in this study. At every glue spread rate, ACQ-treated rubberwood samples possessed higher block shear strength compared to untreated samples. This might be due to the fact that treated rubberwood has the lowest surface roughness compared to the other treated samples. Such results could also probably be attributed to the anatomical characteristics of the rubberwood itself. A study by Syahirah et al. (2019) revealed that, among these four wood species, rubberwood has the narrowest lumen diameter of 15.20 µm. Stables (2017) stated that the specific penetration of resin is positively correlated to the lumen diameter. In comparison to untreated rubberwood, increment in surface roughness has facilitated the spreading of resin on the surface of treated rubberwood but not too rapidly to cause the starvation of glue on the surface. Furthermore, its narrow lumen diameter may prevent the absorbed resin to penetrate deeper into the cell wall too soon in a given specific time and hence prevent the formation of starved glue line. Therefore, a synergistic effect between these two factors might have led to such observations.

According to the results of block shear strength, the values differ based on species, glue spread rate and pressure. To determine the individual or/and combined effects of these factors, ANOVA analysis was performed and the results are listed in Table 5. The analysis shows that the interaction between glue spread rate and pressure was not significant since $p > 0.05$. However, interaction between species and pressure ($p = 0.003$) as well as interaction between species and glue spread rate ($p = 0.000$) was found significant, implying that wood species is the dominant factor in influencing the properties of the resultant CLT samples.

3.4.2 Block shear strength of mixed-species CLT

Mixed-species CLT was manufactured to study the effects of ACQ preservatives on mixed CLT bonding performance. By considering the production cost and economic

Table 5 ANOVA of the effects of species, pressure and glue spread on the shear strength of single-species CLT

Source	df	Mean Square	F	Sig
Species	7	59.677	131.661	0.000**
Pressure	1	30.058	66.315	0.000**
Glue spread	2	42.765	94.350	0.000**
Species × pressure	7	1.469	3.241	0.003**
Species × glue spread	14	5.029	11.095	0.000**
Pressure × glue spread	2	1.286	2.836	0.061
Species × pressure × glue spread	14	0.386	0.852	0.612

df degrees of freedom

**Significantly different at $p \leq 0.05$

benefit, the optimum process parameters were selected based on the analysis. The optimum process parameters selected were a pressure of 1.4 N/mm^2 and glue spread rate of 300 g/m^2. ANOVA analysis found highly significant differences between the types of configuration at $p \leq 0.05$.

Block shear strength for both ACQ-treated kedondong-sesenduk-kedondong and rubberwood-sesenduk-rubberwood shown in Fig. 8 was 7.32 N/mm^2 and 8.05 N/mm^2, respectively higher than combinations for both kedondong-batai-kedondong and rubberwood-batai-rubberwood. Block shear strength of ACQ-treated kedondong-batai-kedondong and rubberwood-batai-rubberwood were 4.92 N/mm^2 and 6.41 N/mm^2 respectively. The high density of kedondong and rubberwood contributed to the high block shear strength of ACQ-treated mixed species as they are able to offset the inferior strength of batai and sesenduk located at the core layer of the CLT panel. This indicates that the adhesive joints were able to transfer the stress from component to component through the interphase region sufficiently (Kamke and Lee, 2007). Besides, the high surface roughness of batai contributes to the low block shear strength for ACQ-treated kedondong-batai-kedondong and rubberwood-batai-rubberwood.

The results obtained suggest that ACQ treatment does not affect the bonding strength of glued timber as reported by Jin et al. (2016) and Qin et al. (2019). These results might be attributed to the fact that the bonding quality of treated wood does not rely on preservative characteristics and treatment method only, but also depends on a variety of factors and interactions, such as wood species, adhesive type, wettability, adhesive spread rate and processing variables used (Lee et al. 2006; Jin et al. 2016; Knorz et al. 2015). The structural difference in wood species and uneven distribution of ACQ in woods might also influence the variation in bonding properties (Qin et al. 2019), because the preservative treatment alone did not adversely affect the bonding properties of hardwood species (Tascioglu 2013).

3.4.3 Delamination on the glue line

Excellent quality and high resistance towards delamination were found in both single and mixed-species CLT. A reduction in bondline length was observed due to shrinking and swelling of wood but the glue line was found unaffected, indicating a good bonding and high resistance to delamination. All samples did not show any visible delamination after testing indicating that glue problems at the interface between wood species did not exist. The adhesive had the most influence on the durability of the bondline (Gong et al. 2016). Adequate glue spread and the optimum amount of penetration are generally considered important for good bond formation. Delamination was found in ACQ and copper treated SYP by Lorenz and Frihart (2006) reporting poor bonding quality of copper-based treated wood. However, no delamination was found in ACQ-treated poplar (Jin et al. 2016), ACQ-treated beech, ACQ-treated maple and ACQ-treated red oak (Yang et al. 2012), ACQ-treated Japanese larch (Miyazaki and Nakano 2003) and ACQ-treated southern pine (Shukla and

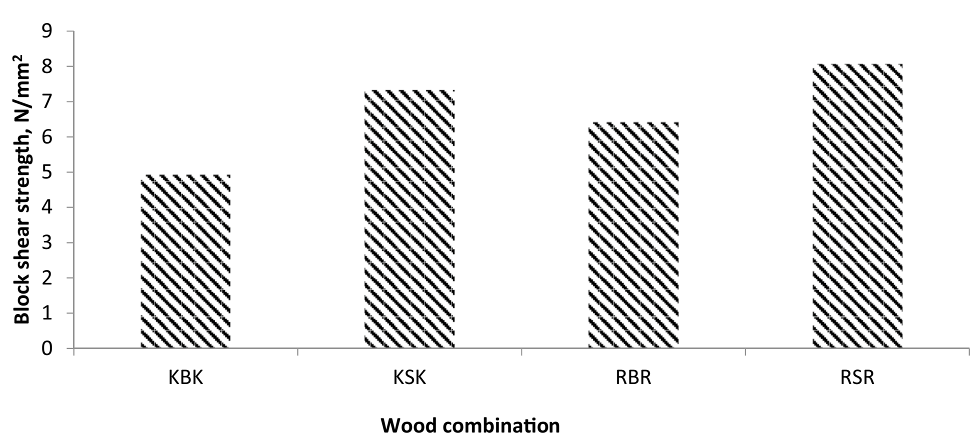


Fig. 8 Block shear strength of treated mixed-species CLT fabricated at a pressure of 1.4 N/mm^2 and glue spread rate of 300 g/m^2. *KBK* Kedondong-Batai-Kedondong, *KSK* Kedondong-Sesenduk-Kedondong, *RBR* Rubberwood-Batai-Rubberwood and *RSR* Rubberwood-Sesenduk-Rubberwood

Kamdem 2012), which was similar to the findings in this study. These findings also indicated that the ACQ retention levels in treated wood did not significantly affect the resistance to delamination.

4 Conclusion

Single-species and mixed-species CLT were successfully made from four Malaysian hardwood species. The conclusions according to the results of the present study are summarized as follows:

- ACQ treatment increased the density of each hardwood species significantly. Low density wood such as batai absorbs more preservatives and has higher copper content compared to kedondong with higher density.
- ACQ treatment increases the surface roughness value probably due to the change in surface chemistry and effects from second drying after pre-treatment.
- Wettability of each species was reduced significantly, allowing better spreading of adhesive on the wood for more effective bonding.
- ACQ treatment has no significant effect on block shear strength of manufactured CLT. Block shear strength of ACQ-treated single-species CLT was found higher than for untreated CLT except for ACQ-treated kedondong. 300 g/m^2 glue spread rate and 1.4 N/mm^2 pressure were the optimum parameters in this study and were used to manufacture ACQ-treated mixed-species CLT. The differences in block shear strength for each species were affected by the amount of glue spread rate, pressure, hardwood species, wettability and other processing parameters.
- No visible delamination was observed on all samples indicating good bonding and good resistance towards delamination.

This study shows that ACQ preservatives can be used to produce CLT with good bonding performance in terms of shear strength and delamination for laminated wood product purposes. The results obtained in this study give information on the possibility of manufacturing treated CLT and combining of two different timber species.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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