Properties of Laminated Composite Panels Made from Fast-Growing Species Glued with Mangium Tannin Adhesive

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Laminated composite is a wood panel constructed from timber pieces then are laminated together. Bio-adhesives such as tannin adhesive are a potential alternative to synthetic adhesives. The purposes of this study were to characterize the chemical makeup of tannin from mangium (*Acacia mangium*) bark extract and to determine the physical-mechanical properties of the panels made from jabon (*Anthocephalus cadamba*) and sengon (*Falcataria moluccana*), and adhesives based on either mangium tannin or methylene diphenyl diisocyanate (MDI). The panels made from five layers of lamina were 5 cm × 24 cm × 120 cm in thickness, width, and length, respectively. Based on results from gas chromatography–mass spectrometry, mangium tannin had 34.04% phenolic compounds. Both wood species were low density, 0.31 g/cm^3 for sengon and 0.44 g/cm^3 for jabon, with an average moisture content of 12.4%. The panels had better width shrinkage than solid wood, with an anti-shrink efficiency of 72.5%. With regard to mechanical properties, none of the panels met the standard for the MOE or shear strength; however, sengon panel with MDI met the standard for MOR. In the delamination test, sengon panel was resistant to cold water immersion. All panels had low formaldehyde emission and met the standard requirements.

Keywords: Laminated composite panels; Fast-growing species; Methylene diphenyl diisocyanate; Tannin adhesive

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INTRODUCTION

Wood has long served as a structural material and as a raw material in some industries. Over time, the source of wood has changed from natural forests to plantation forests. Plantation forests provide small-diameter trees owing to short cutting cycles of approximately 5 to 8 years (Hadi *et al.* 2015). These trees generally have a large amount of juvenile wood that has inferior physical–mechanical properties and lower biodeterioration resistance compared with mature wood from natural forests (Hadi *et al.* 2010).

Laminated panels are an engineered wood product created from small diameter logs processed into large, stiff panels. When they are designed and intended to meet the stringent requirements for structural uses, such panels are called cross-laminated timber (CLT) panels. Laminated panel products are normally made of several stacked wooden layers and glued together, with each layer's fibers being perpendicular to those of neighboring layers. The layers themselves consist of boards that are edge-glued together. An odd number of layers is typically used, which results in a product that has high dimensional stability and load-bearing capabilities in more directions than regular sawn timber or glulam (Gagnon and Pirvu 2011).

Adhesive layers weigh less than bolts and rivets, and since there are no holes to drill (which weaken the structure and may permit ingress of moisture) or fasteners to install, significant labor savings and more rapid production rates are possible. Therefore, adhesive plays an important role in the manufacture of laminated composite panels.

At present, laminated composite production uses adhesives made from non-renewable materials. These adhesives are expensive and contain high levels of formaldehyde, and their availability will steadily decrease with time (Santoso *et al.* 2014). Bio-adhesives that use renewable materials can substitute for synthetic adhesives in laminated composite manufacture. Mangium has become a favored type of tree in plantations because its fiber can be used as a raw material in the pulp and paper industry. Mangium can produce up to 200 m^3/ha at the age of 8 years, with annual growth of 25 m^3/ha (Malik *et al.* 2007). Processing of the wood yields a lot of bark waste, which contains tannins. These naturally occurring polyphenolic compounds can be used as raw material for a green wood-based adhesive. Use of such an adhesive, or bio-adhesive, could reduce the cost of laminated composite manufacturing.

The objectives of this study were to characterize the liquid extract of mangium wood and to determine the physical and mechanical properties of laminated composite panels made from two fast-growing wood species; two adhesives were used, namely mangium tannins and methylene diphenyl diisocyanate (MDI). The physical and mechanical properties, shear strength, and formaldehyde emission of the laminated composites were evaluated, and the results were compared with Japanese Agricultural Standard (JAS) 234 (2003) for glued laminated timber.

EXPERIMENTAL

Extraction and Modification of Mangium Tannin

Chipped mangium stem bark underwent hot-water extraction (70 °C to 80 °C), and the liquid tannin extract was then filtered. Tannin yield was calculated from the difference between the weight of tannin obtained and the amount of the extract solution used.

The Stiasny number is the reactivity of polyphenols in tannin towards formaldehyde and was ascertained as follows. Twenty grams of unmodified dehydrated extract was added to a beaker containing 40 mL of water. The solution was adjusted to pH 7 by adding sodium hydroxide pellets. One hundred milliliters of formalin and 15 mL of concentrated HCl were added, and the mixture was heated at 90 °C for 85 min. The resulting solution was vacuum-filtered, and the solid residue was washed with hot water, dehydrated, oven-dried at 105 °C, and weighed. The Stiasny number was determined according to the following equation:

$$\text{Stiasny precipitation number} = \frac{\text{Dry weight of solid residue}}{\text{Dry weight of extract}} \times 100 \qquad (1)$$

Analysis of Tannin Extract

The tannin extract was analyzed by Fourier transform infrared (FTIR-1600, Shimadzu, Japan) spectroscopy to determine the functional groups, and tannin compounds were analyzed by pyrolysis gas chromatography–mass spectrometry (GCMS; Py-GCMS-QPXP-2010, Shimadzu, Japan).

Preparation of Mangium Tannin Adhesive

Mangium tannin adhesives were made by mixing mangium tannin extract and formaldehyde (10 mL formaldehyde per 100 mL of tannin extract). The mixture was then stirred for approximately 15 min.

Preparation of Laminated Composite

Laminated composite was made from 5- to 7-year-old trees of two wood species, sengon (*Falcataria moluccana*) and jabon (*Anthocephalus cadamba*), harvested from Bogor, West Java, Indonesia. The lumber was processed into lamina sheets that were 1 cm × 6 cm × 120 cm in thickness, width, and length, respectively. The laminas were dried naturally and then kiln-dried to a moisture content of approximately 12%. Lamina sorting was performed by modulus of elasticity (MOE) prediction using a non-destructive device (Panther version MPK-5) for quality sorting. Laminas used for the face and back layers of laminated composite had higher MOE values than the core laminas. Each sheet was bonded using either mangium tannin adhesive, which had a solids content of 10% and a viscosity of 95 cP, or isocyanate, a water-soluble polymer consisting of a base resin and hardener. Glue was double spread at 280 g/m^2 for MDI and 200 g/m^2 for tannin adhesive, then the laminas were pressed using a cold press machine at a pressure of 12 MPa for 4 h and then clamped for 20 h. The resultant five-layer homogeneous panel measured 5 cm × 24 cm × 120 cm in thickness, width, and length, respectively. For comparison purpose, solid wood representing a material without adhesive was used for control. The size of the solid wood was the same as the laminated composite. Three test specimens were used for each treatment.

Laminated Composite Testing Methods

Sample preparation of laminated composite

The physical and mechanical properties of laminated composite were tested according to the Japan Agricultural Standard (JAS) for glue laminated timber (JAS 234-2003 (2003)). Laminated composite cutting models are shown in Fig. 1

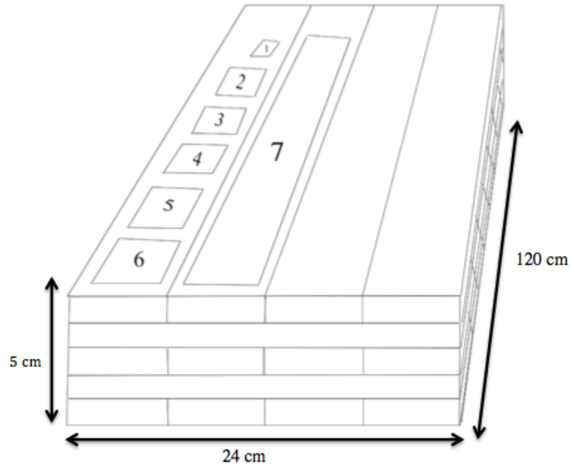
Physical and mechanical properties

Physical properties were tested based on the moisture content, density, and width shrinkage, and mechanical properties were tested with a Universal Testing Machine (Instron type 3369, USA) to determine the modulus of rupture (MOR), MOE, and shear strength. For MOR and MOE tests, centre-point loading was applied with a span of 75 cm and a load speed of 3 mm/min.

Delamination ratio

The cold water delamination test was carried out by soaking test specimens in water at room temperature for 6 h, then putting the specimen into the oven at a temperature of 40 ± 3 °C for 18 h. Hot water delamination was done by boiling the test specimen in water (100 °C) for 4 h, then soaking it in water at room temperature for 1 h before placing it in an oven at 70 ± 3 °C for 18 h. The delamination ratio was calculated using Eq. 2.

$$\text{Delamination ratio (\%)} = \frac{\text{Sum of delaminated lengths of two cross sections}}{\text{Sum of gluing lengths of two cross sections}} \times 100\% \tag{2}$$



1. Sample testing for formaldehyde emission (2.5 cm × 5 cm × 5 cm)
2. Sample testing for shear strength (5 cm × 5 cm × 5 cm)
3. Sample testing for density (5 cm × 5 cm × 5 cm)
4. Sample testing for moisture content (5 cm × 5 cm × 5 cm)
5. Sample testing for delamination (in hot water) (7.5 cm × 7.5 cm × 5 cm)
6. Sample testing for delamination (in cold water) (7.5 cm × 7.5 cm × 5 cm)
7. Sample testing for MOR and MOE (5 cm × 80 cm × 5 cm)

Fig. 1. Cutting model of laminated composite for each test specimen

Formaldehyde emissions

To test formaldehyde emissions, samples sized 2.5 cm × 5 cm × 5 cm were hung in a bottle containing 25 mL of distilled water. The samples were not in contact with the water. The bottle containing the sample was placed in an oven at 40 ± 2 °C for 24 h. The formaldehyde concentration in the sample solution was measured with a spectrophotometer at λ = 412 nm, and 10 mL of sample solution was added to 10 mL of acetyl ammonium acetate reagent.

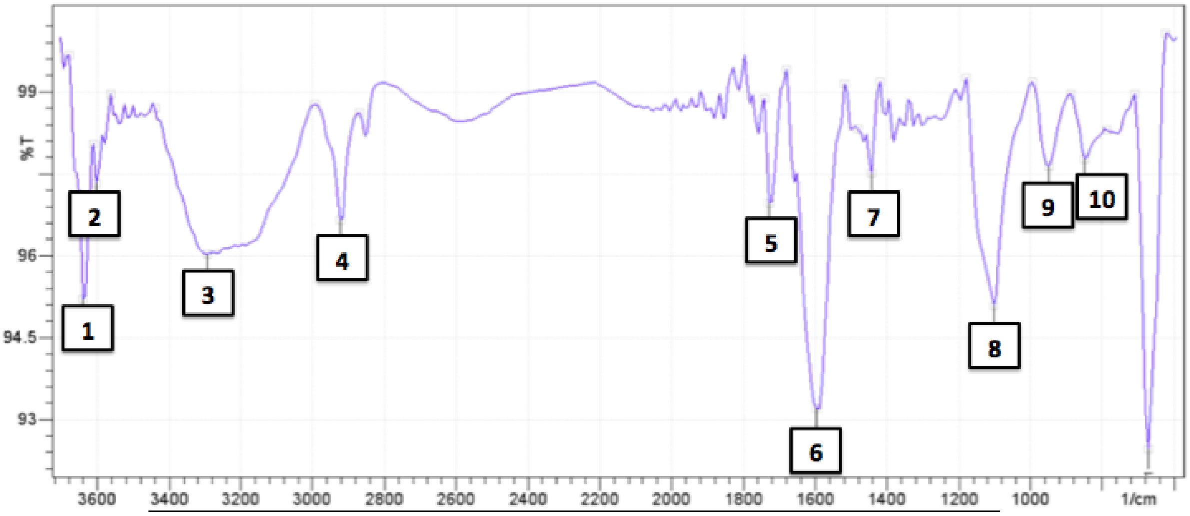
RESULTS AND DISCUSSION

Chemical Analysis of Mangium Tannin Extract

The Stiasny number indicates the reactivity of tannin with formaldehyde. The reactivity can be affected by the bark freshness, source of wood, tree habitat, and extraction methods (Achmadi 1990). Tannin extract accounted for 198.3 g of 1 kg mangium bark or 19.83%, and it had a Stiasny number of 83.48% (w/w), which was higher than that for mahogany extract (79.7%; Lestari *et al.* 2015). This finding indicated that mangium extract has a high amount of reactive polyphenol available for polymerization and would be appropriate for use as a resin.

FTIR spectroscopy

Figure 2 shows a spectrograph of mangium extract in which the wave number 3294 cm^{-1} indicates that mangium tannin extract contains hydroxyl groups, which are typically indicated by peaks between 2500 and 3500 cm^{-1} (Table 1). The wave number at 2924 cm^{-1} indicates a C-H alkane group, with the reference standard of absorbance at 2960 cm^{-1} . The wave number at 1728 cm^{-1} indicates a carbonyl group compound, with the reference range being 1650 to 1800 cm^{-1}. Wave number 1597 cm^{-1} is an aromatic ring vibrational wave, with a reference range from 1500 to 1675 cm^{-1}. The wave number 1443 cm^{-1} indicates an aromatic aldehyde group (reference range, 1300–1475 cm^{-1}, and a wave number of 1103 cm^{-1} indicates an ether group (reference range, 1000–1300 cm^{-1}).



Wave number, υ (cm^{-1})

1	2	3	4	5	6	7	8	9	10
3641	3603	3294	2924	1728	1597	1443	1103	949	849

Fig. 2. Spectrograph of mangium tannin extract

Table 1. Spectrophotometric Absorption Band of Tannin Standard and Mangium Tannin Extract

Sl. No.	Tannin standard*	Mangium tannin extract	Range standard of absorbance**	Description**
1	3335	3294	2500–3500	Hydroxyl group
2	2725	2924	2960	C-H alkane group
3	1710	1728	1650–1800	Carbonyl group
4	1610	1597	1500–1675	Aromatic ring vibration
5	1440	1443	1300–1475	Aromatic aldehyde
6	1320	1103	1000–1300	Ether group
7	754	849	600–1000	=C-H group

*Hindriani (2005); ** Supratman (2010).

GCMS pyrolysis

The GCMS pyrolysis chromatograph (Fig. 3) indicates that mangium tannin extract contains phenol, based on a retention time of 7.81 min, at a concentration of 3.26%, as shown in Table 2. The retention time of 9.38 min corresponds with *o*-cresol phenol compounds at a concentration of 0.51%; 9.81 min is a *p*-cresol phenol compound at 1.58% concentration; 10.00 min is 2-methoxyphenol (guaiacol), at a concentration of 0.37%; 11.99 min is 1,2-benzenediol (pyrocatechol) at a concentration approaching 7.49%; 12.50 min is 3-methoxyphenol (*m*-guaiacol) at 0.31% concentration; 13.03 min is 3-methyl-1,2-benzenediol (3-methylpyrocatechol) at 0.48% concentration; 14.47 min is 1,3-benzenediol, 5-methyl-orcinol at 3.93% concentration; 14.80 min is 1,2,3-benzenetriol (CAS) 1,2,3-trihydroxybenzene at 14.56% concentration; 15.62 min is 4-ethyl-1,3-benzenediol (4-ethylresorcinol) at 1.55% concentration.

Table 2. Chromatic Photometric Absorption Bands of Tannin Extract

Compound	Retention time (min)	Concentration (%)
Phenol (izal)	7.81	3.26
2-Methylphenol, (*o*-cresol)	9.38	0.51
4-Methylphenol, (*p*-cresol)	9.81	1.58
2-Methoxyphenol (guaiacol)	10.00	0.37
1,2-Benzenediol (pyrocatechol)	11.99	7.49
3-Methoxyphenol (*m*-guaiacol)	12.50	0.31
3-Methyl-1,2-benzenediol (3-methylpyrocatechol)	13.03	0.48
1,3-Benzenediol, 5-methyl-Orcinol	14.47	3.93
1,2,3-Benzenetriol (1,2,3-trihydroxybenzene)	14.80	14.56
4-Ethyl-1,3-benzenediol (4-ethylresorcinol)	15.62	1.55
Total phenolic compounds		34.04

The FTIR analysis and the pyrolysis GCMS yielded similar results, indicating that the mangium tannin extract was dominated by hydroxyl group, aromatic group, and ether. Pyrolysis GCMS revealed that phenolic compounds accounted for 34.04% (w/w) of the extract. The tannin extract contained lot of polyphenolic compounds that could react with formaldehyde to form phenol formaldehyde resin. Therefore, the extract could potentially be used as an adhesive for organic composite products.

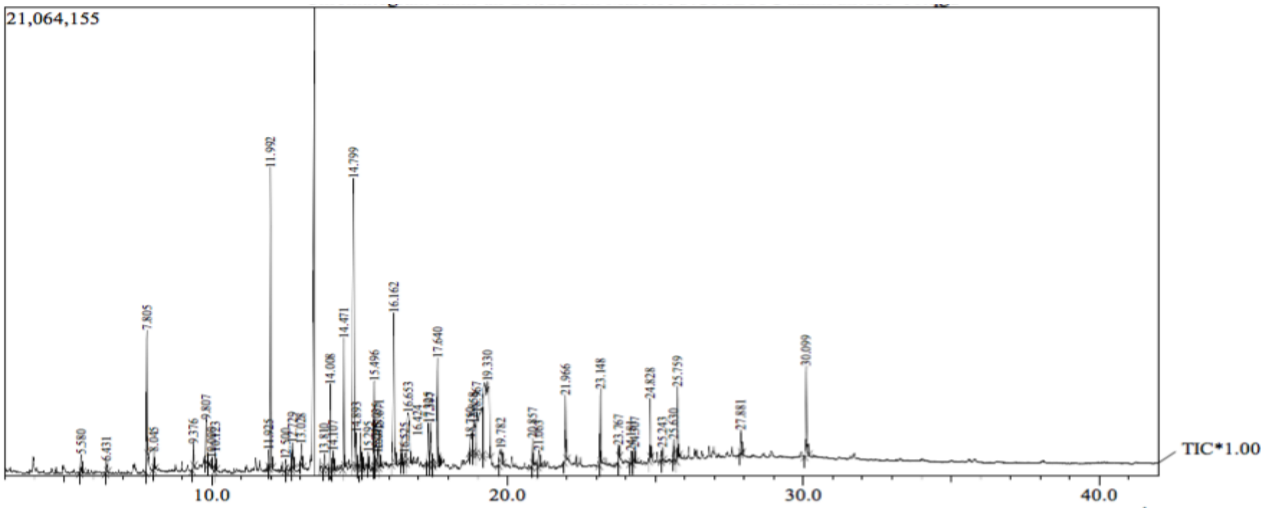


Fig. 3. Chromatograph of mangium tannin extract

Physical Properties of Laminated Composite

Density

The physical properties of laminated composite and solid wood are shown in Table 3. Laminated composite and solid jabon wood had higher densities (0.46 ± 0.04 g/cm^3) than laminated composite and solid sengon wood (0.32 ± 0.01 g/cm^3). The density of the wood component affected the density of laminated composite, and an analysis of variance showed that sengon and jabon had significantly different densities (Table 4). The type of adhesive did not significantly affect the density and the three types of product (solid wood, laminated composite with tannin, and laminated composite with MDI) had the same density values. In short, the glue line, which was very thin, did not affect the weight of the laminated composite.

Moisture content

Table 3 shows that the moisture content of the jabon laminated composite was 13.31 ± 0.33% and that of the sengon laminated composite was 12.21 ± 0.45%. The moisture content fulfilled the JAS 234 (2003) standard which sets the allowable moisture content at less than 15%. According to the analysis of variance shown in Table 4, the moisture content of laminated composite was affected by the wood species but not by the type of adhesive. Jabon wood was shown to have a higher density than sengon wood, so solid jabon wood and jabon laminated composite panels had higher moisture content than solid sengon wood and sengon laminated composite panels; the wood with greater density dried to a lesser degree up than the wood with a lower density (Supartini 2012).

Table 3. Physical Properties of Laminated Composites

Wood species	Adhesive	Density (g/cm^3)	Moisture content (%)	Width shrinkage (%)
Sengon	Solid	0.31 ± 0.01	11.25 ± 1.92	1.82 ± 0.55
	Tannin	0.31 ± 0.02	11.94 ± 0.21	0.37 ± 0.30
	MDI	0.32 ± 0.01	12.48 ± 0.51	0.24 ± 0.04
Jabon	Solid	0.46 ± 0.05	12.27 ± 1.19	2.94 ± 0.22
	Tannin	0.47 ± 0.05	13.06 ± 0.12	0.42 ± 0.37
	MDI	0.44 ± 0.04	13.56 ± 0.29	1.81 ± 5.24
JAS standard			Max 15	

Width Shrinkage

Laminated composite had an advantage in dimensional stability owing to the board layers being placed in orthogonally alternating orientation to neighboring layers. Table 3 shows less width shrinkage for laminated composite made from sengon and jabon than for solid wood. Anti-shrink efficiency (ASE) is a measure of how well a technique compares to a control experiment in which the wood is allowed to air-dry (Bernick 1998). The ASE of jabon laminated composite with tannin was 85.6% and that of jabon laminated composite with MDI was 38.4%. The ASE of sengon laminated composite with tannin was 79.5% and that of sengon laminated composite with MDI was 86.4%, demonstrating that laminated composite panels had higher dimensional stability than solid wood. Width shrinkage of sengon laminated composite (0.81 ± 0.77%) was lower than that of jabon laminated composite (1.72 ± 1.11%).

According to the analysis of variance shown in Table 4, the width shrinkage of laminated composite was affected by the wood species and the type of adhesive. Generally, high-density wood will shrink more than low-density wood. The density of sengon was 0.32 g/cm^3, and previous research reported shrinkage in radial and tangential directions of 2.5% and 5.2%, respectively (wet to dry kiln) (Iskandar 2006). Jabon, which had a density of 0.46 g/cm^3, was previously report to have shrinkage in the radial and tangential directions of 3.03% and 5.41%, respectively (wet to dry kiln) (Lempang 2014). Width shrinkage was also affected by the type of adhesive. Based on additional analysis by Duncan's test (Table 5), panels made with tannin and MDI adhesives were not significantly different from each other in terms of width shrinkage, but both were different from solid wood. The width shrinkage of panels was smaller than that of solid wood, indicating that laminated composite panels had more dimensional stability than solid wood.

Table 4. ANOVA Physical and Mechanical Properties of Laminated Composite

Parameter	Wood species	Adhesive
Density (g/cm^3)	**	NS
Moisture content (%)	**	NS
Width shrinkage (%)	**	**
MOE (MPa)	NS	NS
MOR (MPa)	NS	NS
Shear strength (MPa)	NS	**
Delamination in cold water	NS	NS
Delamination in hot water	NS	**

**Very significant ($P < 0.01$); NS = not significant.

Table 5. Duncan's Test Physical and Mechanical Properties of Laminated Composite

Bonding system	Width shrinkage (%)	Shear strength (MPa)
Tannin	0.39a	2.52a
MDI	1.03a	2.81a
Solid wood	2.38b	5.19b

Remarks: Values followed by the same letters within a column are not significantly different.

Mechanical Properties of Laminated Composite

Modulus of elasticity

The MOE values of solid wood and laminated composite are shown in Table 6. None of the panels met the standard JAS 234 (2003), which sets the allowable MOE at more than 7354.99 MPa. The MOE of jabon solid wood (6414.21 MPa) in this study was higher than that found by Hadi *et al.* (2013) (3868.92 MPa), but both values were very low because of low-density wood.

Table 6. Mechanical Properties of Panels

Wood species	Adhesive	MOE (MPa)	MOR (MPa)	Shear strength (MPa)
Sengon	Solid	4,633.09 ± 903.62	24.92 ± 7.71	3.16 ± 0.36
	Tannin	3,640.52 ± 643.01	24.35 ± 2.37	1.96 ± 0.44
	MDI	5,043.54 ± 299.99	32.02 ± 7.17	4.98 ± 0.08
Jabon	Solid	6,414.21 ± 1,410.14	43.90 ± 6.73	5.42 ± 0.15
	Tannin	4,253.26 ± 1,441.99	21.06 ± 6.52	3.08 ± 0.77
	MDI	3,448.54 ± 343.66	25.55 ± 2.44	2.47 ± 1.28
JAS standard		Min 7354.99	Min 29.42	Min 5.29

The low quality of sengon wood might be attributed to the fact that the mass of wood was dominated by juvenile wood. Juvenile wood is characterized by lower density, shorter tracheids or fibres, lower percentage of latewood, thinner cell walls, smaller tangential cell dimensions, lower cellulose content, lower strength, higher longitudinal shrinkage, higher microfibril angel (MFA), larger cell lumen, more reaction wood, more spiral grain and higher degree of knottiness compared with mature wood. (Panshin and de Zeeuw 1980).

The laminated panels were made from fast-growing species, which have low density and low-value mechanical properties. The MOE of laminated composite made with tannin adhesive was lower than that of the solid wood. The MOE of laminated composite (4096.43 ± 955.76 MPa) was also lower than the MOE of glulam (4726.61 MPa) made from jabon and sengon wood with mahogany adhesive (Lestari *et al.* 2015). Based on Supartini (2012), the MOE of laminated composite made from jabon with mangium adhesive was slightly lower than that of jabon cross laminated timber with MDI (4,824.87 ± 0.18 MPa). Although laminated composite has high dimensional stability, the MOE is lower because of the orientation of the laminas. According to the analysis of variance shown in Table 5, the MOE of laminated composite was not affected by the wood species or the type of adhesive.

Modulus of rupture

The MOR for laminated panels and solid wood panels is shown in Table 6. All panels using tannin adhesive had lower MOR values than MDI and solid wood. However, only laminated panels made from sengon with MDI (32.02 MPa) had an MOR that met JAS 234 (2003), which sets the allowable MOR at more than 29.42 MPa, which is higher than the MOR of solid jabon wood (24.91 MPa). Based on the analysis of variance presented in Table 5, MOR values were not significantly different based on the species of wood or the type adhesive.

Shear strength

Shear strength tests were carried out in dry conditions. Table 6 shows that laminated panels (2.67 ± 0.85%) had lower shear strength values than solid wood (5.19 ± 0.26 %). All panels failed to meet the JAS standard of more than 5.29 MPa for shear strength. According to analysis of variance (Table 5), the type of adhesive affected the shear strength of laminated panels. Based on the results of Duncan's test (Table 6), tannin adhesive and MDI did not have significantly different effects. However, a higher shear strength could be achieved for laminated panels owing to the higher resin content of mangium extract.

Delamination Test

The results of cold-water delamination (Table 7) indicate that jabon laminated panels with tannin and MDI adhesives had the highest ratio of delamination.

Table 7. Laminated Composite Delamination Test

Wood species	Adhesive	Delamination (%)	
		Cold water	Hot water
Sengon	Tannin	0.00 ± 0.00	1.42 ± 2.46
	MDI	0.00 ± 0.00	8.39 ± 11.86
Jabon	Tannin	4.11 ± 3.45	7.40 ± 5.02
	MDI	12.32 ± 5.25	15.82 ± 21.34
JAS Standard		Max 5	Max 10

Laminated panels made from sengon had no delamination, because sengon has a low density and the glue could penetrate more deeply into the wood compared with jabon. According to JAS 234 (2003), sengon and jabon laminated composites with tannin adhesive met the Japanese standard of less than 5% delamination.

In the hot-water delamination test, laminated panels made with MDI had the highest delamination value. However, sengon laminated panels still met the JAS 234 (2003) requirement of less than 10% delamination. As in the cold-water test, sengon laminated panels showed no delamination. For exterior use, sengon laminated panels fulfilled JAS standards for both cold-water (maximum 5%) and hot-water conditions (maximum 10%). According to the analysis of variance (Table 4) showing a significantly different performance between tannin adhesive and MDI, the tannin adhesive was better than MDI in the hot-water delamination test.

Formaldehyde Emissions

Whatever formaldehyde that has not reacted with the resin in the adhesive is eventually released into the environment. When formaldehyde is present in the air at levels higher than 0.1 parts per million, some people may have health effects, such as watery eyes; a burning sensation in the eyes, nose, and throat; coughing; wheezing; nausea; and skin irritation (American Cancer Society 2014). Table 8 shows that formaldehyde emissions of jabon and sengon laminated composites can be classified as F****, which is the lowest emission and the best class according to JAS 234 (2003).

Table 8. Formaldehyde Emissions from Laminated Composite

Wood species	Formaldehyde emissions (mg/L)	Grade*
Jabon	0	F****
Sengon	0	F****

*Categorized by standard JAS 234 (2003)

CONCLUSIONS

1. Mangium extract has a high amount of reactive polyphenol available for polymerization and would be appropriate for use as a resin due to its high Stiasny number (83.48%), and high phenolic compounds (34.04%).
2. The adhesion quality of laminated composite glued with tannin adhesive from mangium bark was the same as those of glued with MDI. In addition, the tannin adhesive was even better than MDI in ASE and hot-water delamination.
3. All the laminated composite panels failed to fulfill the JAS 234 (2003) standard.
4. All laminated panels made from fast-growing species glued with mangium tannin adhesive and MDI performed low formaldehyde emissions and classified as F**** according to JAS 234 (2003).

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