Composite Flooring Quality of Combined Wood Species Using Adhesive from Merbau Wood Extract

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Abstract

Polyphenol chemical components extracted from merbau (*Intsia* spp.) wood exhibit a strong affinity for resorcinol and formaldehyde in alkaline conditions, forming a copolymer that could serve as an adhesive. A liquid extract of merbau wood (M) was allowed to copolymerize with resorcinol (R) and formaldehyde (F) under alkaline conditions, with an M:R:F weight ratio of 100:5:10. The course of the reaction and potential mechanisms of copolymerization were scrutinized using Fourier transform infrared spectroscopy, X-ray diffraction, and differential thermal analysis. The adhesive was used to manufacture three-ply composite flooring consisting of a back layer of rubberwood (*Hevea brasiliensis*), a core layer from sengon (*Paraserianthes falcataria*) wood, and a face layer with one of seven wood species, namely, sengon, sungkai (*Peronema canescens*), mangium (*Acacia mangium*), rubberwood, mahogany (*Swietenia* spp.), kempas (*Koompassia malaccensis*), and merbau. The adhesive was spread on the face and back layers of 170 g/m² single glue line, which was followed by pressing at 11 kg/cm² for 3 hours at room temperature. After conditioning for 10 days, the resulting composite flooring was examined to assess its physical and mechanical properties, shear strength, and formaldehyde emission. Results revealed that the bioadhesive exhibited crystallinity of 23.32 percent and a melting glass transition of 115.31°C. IV-meter intrinsic viscosity tools were used to determine the product's molecular weight of 49,658. Physical–mechanical properties and shear quality of the composite flooring were similar to products that use synthetic phenolic adhesive and belong to the exterior quality type with E_0 or F**** types of low formaldehyde emission.

L he use of adhesive is essential in products such as glue laminated timber, laminated veneer lumber, cross-laminated timber, block board, particleboard, finger joint board, and other composite wood products. In Indonesia resorcinolbased adhesives used in the manufacturing process of wood products are mostly imported from other countries, e.g., Belgium, and the price of these adhesives continues to rise. Resorcinol-formaldehyde (RF) and phenol-resorcinol-formaldehyde (PRF) cold setting adhesives are primarily used in the manufacture of structural, exterior grade glulam, finger joints, and other exterior timber structures (Pizzi 2003), and research is currently under way to obtain more economical adhesives from renewable resource materials. Recently, formaldehyde has been classified as a probable human carcinogen, and its use could be limited in the near future. In order to reduce formaldehyde emissions from wood panels, new environmentally friendly adhesives need to be developed. The main natural resins used as wood panel binders are vegetal tannin adhesives, lignin adhesives and, more recently, soy protein adhesives. Tannins, which are natural polyphenolic compounds, are present in large

concentrations in wood barks. In order to develop industrial green wood-based adhesives, tannins need to be easily available and in large quantity (Bertaud et al. 2012).

Wood is a biomaterial that is primarily composed of lignocellulosic components. However, it also contains other chemical compounds that can be extracted by use of polar and nonpolar solvents without destruction of the cellulose and lignin structures. These extracts are dissolvable in organic solvents or water, and known extractive compounds include tannin and other polyphenols, color pigments,

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volatile compounds, fat, resin, wax, gum, and starch. The ability of green adhesives to make a significant impact as a substitute for polymeric materials depends on the availability of low-price biopolymer fractions in large quantities and of high-quality polyphenolic extracts, displaying good chemical reactivity toward cross-linking agents (e.g., formaldehyde and glyoxal; Ping et al. 2012). The amounts of extracts within wood tissues vary from below 1 percent to more than 30 percent, depending on many factors, such as the growth of the tree and the time that the tree is cut down (Donegan et al. 2007).

Adhesive materials can be extracted from wood waste, giving it an added value. The discovery of natural adhesive materials such as tannin from the bark extracts of acacia (*Acacia decurrens, A. mangium*), mangrove (*Rhizophora* spp.), and other trees (Brandt 1953, Coppens et al. 1980, Hashim et al. 2011, Santoso et al. 2012, Zhou et al. 2013) has led to numerous studies on how to obtain alternative bioadhesives from renewable resource materials. Many such compounds are probably still undiscovered and may exist in various parts of numerous tree species. One of the species that likely contains natural adhesive materials is merbau (*Intsia* spp.) wood. When this wood is soaked in water, it leaches a dark-colored extract similar to resorcinol, and research is needed to assess it.

Previous research showed that extractives from merbau sawdust could be made for phenolic-based resin if they react with resorcinol and formaldehyde at base conditions and if the weight mixture was merbau:resorcinal:formaldehyde (M:R:F) = 100:(0-12.5):10 with a maximum extender of tapioca of 10 percent weight ratio of liquid MRF resin (Malik and Santoso 2010; Santoso and Malik 2011a, 2011b). That resin could be applied for glued laminated lumber or laminated bamboo manufacturing with the M:R:F = 100:5:10 formula and with pH 10 to 11 to keep the resin from coagulating too quickly (Santoso and Malik 2011b, Santoso et al. 2013).

The goal of the current study was to characterize the merbau wood liquid extract, copolymerize it with resorcinol and formaldehyde to obtain an adhesive from a renewable resource material, and optimize the adhesive for three-ply composite flooring manufacturing. Physical and mechanical properties, shear strength, and formaldehyde emission of composite flooring were evaluated, and the results were compared with those other composite products made from phenolic synthetic resin.

Methods

Adhesive preparation

Air-dried merbau wood sawdust was mixed with water in a 1:4 ratio (wt/wt) and heated at 80°C for 3 hours. The liquid extract was separated from sawdust by filtering, and the extraction process was repeated twice, with a resulting yield of 6.5 percent (wt/wt, extractive to sawdust both in ovendried weights). MRF had two components, consisting of resin (merbau wood liquid extract, resorcinol technic, and 40% NaOH) and formaldehyde (37%) as a cross-linker or curing agent. Merbau liquid extract (M) was mixed with resorcinol (R) at weight ratios of 100:5, respectively, and NaOH was added to adjust the pH to 10 to 11. The mixture was added to formaldehyde (F) at 10 percent M (wt/wt) and mixed until homogenous, which was followed by a 1-hour conditioning period. In this study the reaction product between merbau extract and formaldehyde is called polymerization, and the reaction product between merbau extract incorporated with resorcinol and formaldehyde is called copolymerization.

Prior to the adhesive's use in composite flooring manufacture, tapioca at 5 percent weight was added as an extender. To verify the reaction of the MRF components, qualitative analyses were done by ultraviolet-visible (UVvis) spectrometry (UV-1700, Shimadzu), Fourier transform infrared (FTIR-1600, Shimadzu) spectroscopy, X-ray diffraction (XRD-7000, Shimadzu), pyrolysis gas chromatography mass spectrometry (Py-GCMS-QPXP-2010, Shimadzu), differential thermal analysis (DT-30, Shimadzu), and viscosimeter (IV-meter, Seiko). Physical and chemical properties of the adhesive were tested by comparison with phenol-resorcinol-formaldehyde (PRF) adhesive (Anonymous 2003) and waterproof resorcinol glue (Anonymous 2002). The measured properties were viscosity, density, visual aspects, contaminants, pH, solid matter contents, and free formaldehyde. Free formaldehyde from resin was determined using the acetyl acetone method (Nash reagent) at a wavelength of 412 nm (Roffael 1993) and the following procedure.

- 1. From a formaldehyde standard solution of 3 mg/liter, we measured a pipetted amount of 0, 5, 10, 20, 50, or 100 mL into six bottles (100 mL each) and then diluted each by adding water up to the 100-mL line.
- 2. Each bottle contained concentrations of 0, 0.15, 0.3, 0.6, 1.5, and 3 mg/liter formaldehyde, respectively.
- 3. We diluted the MRF resin in the water up to 100 times (MRF resin sample).
- 4. We added 10 mL of MRF resin sample to 10 mL of ammonium acetate (20%; 200 g in 1,000 mL), and then we added 10 mL of acetyl acetone (0.4%; 4 mL in 1,000 mL). We put the solution in a water bath at 40°C for 10 minutes, and then cooled it to reach room temperature. The solution was measured with a photometer at 412 nm.

Composite flooring manufacturing

A three-ply composite flooring as a component of parquet flooring was made with sengon (*Paraserianthes falcataria*) wood for the core layer and rubberwood (*Hevea brasiliensis*) for the back layer. The face layer varied and was made from one of seven different wood species, namely, sengon, sungkai (*Peronema canescens*), mangium (*Acacia mangium*), rubberwood, mahogany (*Swietenia* sp.), kempas (*Koompassia malaccensis*), and merbau. The wood from these species can be classified as low density as produced from sengon with a density of 0.37 g/cm³; medium density from sungkai, mangium, rubber, and mahogany with densities of 0.50, 0.51, 0.52, and 0.53 g/cm³, respectively; and high density from kempas and merbau with densities of 0.60 and 0.64 g/cm³, respectively.

The dimensions of the face and back layer components were 90 cm by 14 cm by 3 mm in length, width, and thickness, respectively. These layers consisted of one panel, while the core layer consisted of smaller parts with dimensions of 14 cm by 3 cm by 12 mm in length, width, and thickness, respectively. Fiber orientation of the core layer was perpendicular to the face and back layers. The composite flooring dimensions were 90 cm by 15 cm by 18 mm in length, width, and thickness, respectively, with 1 cm of the core layer being offset on the width to form a tongue



Figure 1.—Three-ply composite for parquet flooring.

and groove on each board for interlocking connections in the parquet flooring, as shown in Figure 1.

In the manufacturing of composite flooring, the adhesive was spread on the face and back layers with an amount equal to a 170-g/m^2 single glue line, followed by pressing at 11 kg/cm^2 for 3 hours at room temperature; conditioning took 10 days. Five replications were made for each wood specimen, and all composite flooring was made in a lumber factory in Central Java, Indonesia.

The evaluations of physical and mechanical properties of composite flooring were done according to Sutton et al. (2011), while shear strength and formaldehyde emissions were based on the Japanese Agricultural Standard (JAS 2003). The shear strength test was done in dry conditions, and it was done in the glue line area, as described in Figure 2, and the value was calculated with the following formula:

$$B = P/A$$

where

- $B = \text{shear strength (N/mm^2)},$
- P = maximum load (N), and
- A = area of shear test (mm²).

Formaldehyde emission was measured with a spectrophotometer at $\lambda = 412$ nm with the reactant acetyl ammonium acetate (WKI method), and the test procedures as follows.

Sample preparation

- 1. We hung a test sample of 25 by 25 by 1 mm using rope.
- 2. We put 50 mL of distilled water in a 500-mL bottle.
- 3. We hung the sample inside the bottle (about 1 cm above the water level).
- 4. We used another bottle containing 50 mL of distilled water as a control.
- 5. We put the bottles in a 40° C oven for 24 hours.



Figure 2.—Test specimen for shear strength.



Figure 3.—Scheme of formaldehyde emission test.

- 6. Formaldehyde emissions that were released from the sample were caught by the water.
- 7. After 24 hours in the oven, we took the bottles out and then immersed them in the water for 30 minutes to increase the chances of catching emissions in the water. The sample was taken out of the bottle, and the water was moved to the volumetric flask.

The scheme of the test is illustrated in Figure 3.

Test of formaldehyde emission

- 1. From a formaldehyde standard solution of 3 mg/L, we pipetted amounts of 0, 5, 10, 20, 50, or 100 mL into six bottles (100 mL each) and then diluted each by adding water up to the 100-mL line.
- 2. The bottles contained concentrations of 0, 0.15, 0.3, 0.6, 1.5, and 3 mg/L formaldehyde, respectively.
- 3. We pipetted 25 mL of sample and put the original formaldehyde solution into the Erlenmeyer flask (100 mL) with a stopper.
- 4. We added 25 mL of acetyl acetone–ammonium acetate to the Erlenmeyer flask (see number 3), and stirred it until homogeneous.
- 5. We heated the solution for 10 minutes in a water bath at 65°C and then cooled it until it reached room temperature.
- 6. The same procedure was done to the control solution.
- 7. The solutions were measured using a spectrophotometer at 412-nm wavelength.

To evaluate the effect of wood species on composite flooring properties, a completely randomized design with a single factor (seven levels) was used to analyze the data. The factor was wood species used for the face layer of composite flooring, namely, sungkai, kempas, mangium, mahogany, rubberwood, merbau, and sengon.

Results and Discussion

Qualitative analysis of merbau wood liquid extract

Analysis by UV-vis spectroscopy (Fig. 4) showed that the liquid extract of merbau wood ($\lambda = 279.50$ nm) consisted of conjugated or aromatic systems (Field et al. 2008), similar to resorcinol, which has $\lambda = 273.50$ nm; the difference is still acceptable because merbau is not a pure substance. Further identification by FTIR spectroscopy (Fig. 5) showed that there were functional groups of hydrogen bonded (–O– H Stretch; wave number 3,134 to 3,366/cm) and aromatic



Figure 4.—Spectrograph of resorcinol (a) and liquid extract (b) of merbau.

C=C double bond (C=C; wave number 1,510 to 1,693/cm; Field et al. 2008).

The Py-GCMS chromatogram (retention time = 22.19 min) showed that the merbau wood liquid extract was dominated by resorcinol compounds, namely, 1,3-benzenediol resorcin (Fig. 6a) with a crystallinity degree of 20.86 percent (Fig. 6b) and a melting or glass transition temperature of 110.89° C (Fig. 6c). All identifications indicated that the merbau extract consisted of phenolic compounds, especially 1,3-benzenediol resorcin, commonly called resorcinol.

Physical and chemical properties of adhesive from merbau wood liquid extract

The adhesive was produced from air-dried sawdust; 1 kg merbau sawdust with a moisture content 13.8 percent had a

yield of 8.5 kg liquid extract, equal to 56 g solid content containing extractives. Regarding the moisture content of sawdust, the yield of extractives as raw material of adhesive reached 6.5 percent.

The adhesive was prepared by grafting merbau wood liquid extract onto the active methylol groups of the low condensation obtained by the reaction of resorcinol with formaldehyde. Copolymerization of the liquid extract with formalin and resorcinol was carried out by an alkaline catalyst at room temperature. The formation of copolymers (Fig. 7b) was identified by FTIR spectroscopy with the appearance of absorption bands for different spectrum numbers compared with the polymer (Fig. 7a) and monomer (Fig. 5). There were bonded C–H and C=O absorption bands below 2,850 to 2,750/cm and 1,820 to 1,600/cm (Fig. 6a), which indicated functional aldehyde (CHO) groups, and the absorption intensity decreased (from 60% to 25%) after



Figure 5.—Fourier transform infrared spectrograph of merbau wood liquid extract.



Figure 6.—Chromatogram (a), diffractograph (b), and thermogram (c) of merbau wood liquid extract.



Figure 7.—Spectrograph of polymerization (a) and copolymerization (b) products from merbau wood liquid extract.

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Figure 8.—Chromatogram of polymerization (a) and copolymerization (b) products from merbau wood liquid extract.

resorcinol was added in excess. In the reaction following the addition of excess resorcinol, the polymer (Fig. 7a) reacts with the –CH₂OH groups to form copolymers (Fig. 7b) in which the terminal resorcinol groups could be a resorcinol or any type of resorcinol compound adhesive. Regarding the previous research results (Santoso and Malik 2011b), adding more resorcinol to a solution of merbau extract and formaldehyde resulted in less free formaldehyde, with a linear equation of y = -0.0153x + 0.4986, $r^2 = 0.8878$, where x represents the addition of resorcinol to solution of merbau extract and formaldehyde.

Py-GCMS results further confirmed the reaction details. The chromatogram showed that the merbau wood liquid extract was dominated by resorcinol compounds, and after polymerization different band peaks were apparent, dominated by resorcinol compound copolymer derivatives, namely, 5-methoxy-2,3-dimethyl-3-methoxy-5,6-dimethylphenol, with 24.75 minutes of retention time (Fig. 8a). After copolymerization, the band peaks indicated phenolic polymer derivatives dominated by 1,3-benzenediol,4,5dimethylresorcinol with 24.38 minutes of retention time (Fig. 8b).

XRD indicated that the compounds within the copolymerized products of merbau wood liquid extract had a crystallinity degree of 23.32 percent (Fig. 9a), relatively close to that of merbau wood liquid extract (20.86%), while the transition temperature was 115.31°C with a decomposition or dissociation temperature of 468.77°C (Fig. 9b), which indicates the formation of copolymer. Copolymer



Figure 9.—Diffractograph (a) and thermogram (b) of copolymerization products from merbau wood liquid extract.

Table 1.—Physical and chemical properties of adhesive from merbau wood liquid extract.

Properties	Copolymer	Standard, PRF ^a	
Appearances			
Phase	Liquid	Liquid	
Color	Red-brown	Red-brown	
Smell	Phenol	Phenol	
Viscosity (poise)	56	3.40	
Acidity (pH)	11	8	
Molecular wt	49,658	b	
Depolymerization temp. (°C)			
Glass point	115.31	161	
Decomposition, dissociation	468.77		
Crystallinity degree (%)	23.32	51.53	
Retention time (min) ^c	24.38		
Specific gravity	1.14	1.15	
Solid content (%)	29.05	57.03	
Free formaldehyde (%)	0.011	0.04	
Gel time of MRF resin at 135°C (min) ^d	128	85	

^a PRF = phenol-resorcinol-formaldehyde (Anonymous 2003).

 $^{\rm c}$ Retention time is the elapsed time from the introduction of the solute to the peak maximum.

 d MRF = merbau-resorcinol-formaldehyde .

formation was further confirmed by using IV-meter intrinsic viscosity tools, which determined the product's molecular weight was 49,658, much heavier than that of the polymerized product of merbau wood liquid extract (9,308). An overview of the copolymer characteristics of merbau wood liquid extract can be found in Table 1. From this overview it may be concluded that merbau wood liquid extract is similar to PRF resin in terms of physical and chemical properties, although there are some differences in terms of crystallinity, solid content, viscosity, and depolymerization temperature. These differences are likely a result of the different raw material used.

Properties of composite flooring

The properties of composite flooring specimens made with different wood species bonded together using merbau extract adhesive are presented in Table 2. Moisture content of products varied from 10.66 to 13.41 percent, and sengon wood was the lowest one because it had the lowest wood density, with a value of 0.37 g/cm^3 , while the other six wood

species had 12.48 to 13.41 percent moisture content with wood density varying from 0.50 to 0.64 g/cm³. The moisture content of all products fulfills Indonesian standard SNI 01-5008.2-2000 requiring less than 14 percent (National Standardization Bureau 2000).

The composite flooring shear strength of low-density wood (sengon) was 3.23 N/mm² with wood failure of 50 percent for the wet test and 6.53 N/mm² with wood failure of 90 percent for the dry test, while the composite flooring with medium-density woods (sungkai, mangium, rubberwood, and mahogany) had an average shear strength of 2.09 N/mm² with wood failure of 10 percent for the wet test and 4.41 N/mm² with wood failure of 35 percent for the dry test. The composite flooring of the high-density woods (kempas and merbau) had an average shear strength of 1.57 N/mm² for the wet test with wood failure of 0 percent and 4.34 N/ mm² with wood failure of 15 percent for the dry test. In comparison with products that used a phenolic synthetic adhesive and yielded 1.78 N/mm² for the wet test and 7.72 N/mm² for the dry test (Santoso and Rachman 2004), composite flooring from sengon wood was found to have similar values. All shear strengths in dry test fulfill Indonesian standards for all types of plywood (SNI 01-5008.2-2000) with a minimum requirement 0.7 N/mm² (National Standardization Bureau 2000).

Statistically, the wood species used for the face layer affected composite flooring properties (Table 2). The products with a face layer made from sengon wood had the highest shear strength, whereas those with the lowest strength were made from the rubberwood and merbau wood. The low values suggested a high nonpolar extractive compound was present within rubber and merbau wood, which prevented molecular bonds between adhesives and the wood.

The modulus of rupture (MOR) of the three-ply composite flooring ranged from 17.91 to 43.79 N/mm² (Table 2). The highest MOR value belonged to the products with the face layer made from sengon wood; however, statistically the value was not significantly different from similar products with face layers made from rubberwood (38.02 N/mm²), mahogany (40.05 N/mm²), and kempas (41.67 N/mm²). The products have MOR values similar to typical properties of cross-laminated timber for building materials described by Sutton et al. (2011), which had an average value of 24.0 N/mm². The lowest MOR value was associated with products with a face layer made from mangium wood (17.91 N/mm²).

Table 2.—	Properties	of composite	flooring. ^a

Face layers	Properties ^b								
	Moisture content (%)	Density (g/cm ³)	Shear strength (N/mm ²)		Wood failure (%)		MOR	MOE	Formaldehyde emission
			Dry test	Wet test	Dry test	Wet test	(N/mm^2)	(N/mm^2)	(mg/liter)
Sengon	10.66 C	0.37 D	6.53 A	3.23 A	90	50	43.79 AB	5,139 C	0.08 CD
Sungkai	13.40 A	0.50 C	3.52 D	1.76 D	40	0	37.17 B	6,500 A	0.35 A
Mangium	13.18 A	0.51 C	5.58 B	2.95 B	40	20	17.91 D	6,128 AB	0.26 B
Rubber	12.48 B	0.52 C	3.24 DE	1.24 E	40	20	38.02 AB	5,667 BC	0.09 CD
Mahogany	13.41 A	0.53 C	4.44 C	1.57 D	20	0	40.05 AB	5,654 BC	0.10 C
Kempas	12.74 B	0.60 B	4.36 C	2.54 C	20	0	41.67 AB	6,120 AB	0.04 D
Merbau	13.16 A	0.64 A	2.89 E	1.57 D	10	0	25.40 C	6,017 AB	0.22 B

^a Delamination for all samples = 0 percent.

^b Values followed by the same latter are not significantly different. MOR = modulus of rupture; MOE = modulus of elasticity.

 $^{^{}b}$ — = no data.

The three-ply composite flooring specimens had modulus of elasticity (MOE) values ranging from 5,139 to 6,500 N/ mm^2 (Table 2). Statistically, the products with face layers made from sungkai wood (6,500 N/mm²) were not significantly different from those with face layers made from mangium wood (6,128 N/mm²), kempas (6,120 N/ mm^2), and merbau (6,017 N/mm²). The products with face layers made from rubberwood (5,667 N/mm²) were relatively equal to similar products with face layers made from mahogany (5,654 N/mm²), kempas (6,120 N/mm²), mangium (6,128 N/mm²), and merbau (6,017 N/mm²). Formaldehyde emission on the three-ply composite flooring ranged between 0.04 and 0.35 mg/liter, which places the products among the lowest in terms of emission (F****; JAS 2003). Statistically, the products with a face layer made from sengon wood (0.08 mg/liter) were not significantly different from similar products with a face layer made of rubberwood (0.09 mg/liter), whereas products with a face layer made from mangium wood (0.26 mg/liter) were equal to products with face layers of merbau wood (0.22 mg/liter).

The physical-mechanical properties and formaldehyde emission characteristics of the composite flooring bonded with merbau wood liquid extract (bioadhesive) mostly fulfilled the criteria for laminated wood bonded with the conventional, but not renewable, adhesive (e.g., PRF), with the exception of MOE. This MOE drawback could be improved by using a greater amount of the adhesive and/or imposing longer pressing time. The much lower formaldehyde emission from the adhesive compared with the conventional PRF adhesives strongly suggests it could potentially be used for gluing wood or other lignocellulosic products, which have four-star formaldehyde emission (JAS 2003).

In summary, the bioadhesive could provide as effective a performance as the conventional PRF adhesives in composite wood. In this way, the bioadhesive can be expected to sooner or later replace the conventional PRF adhesives derived from nonrenewable materials.

Conclusions

Based on qualitative analysis using UV-vis spectroscopy, FTIR spectroscopy, and Py-GCMS and physical analysis using DTA, XRD, and IV-meter intrinsic viscosity, merbau wood waste liquid extract is predominantly 1,3-benzenediol resorcin, or resorcinol. Copolymerization of the liquid extract with formaldehyde and resorcinol in alkaline conditions produced 1,3-benzenediol,4,5-dimethylresorcinol, with a molecular weight of 49,658, as resin. Physical and mechanical properties of three-ply composite flooring have high performance as a building component material, and the shear strength was equal to phenol-resorcinolformaldehyde synthetic adhesives with low formaldehyde emission.

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