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# Research Article Incorporation of Phenol-formaldehyde-based Black Liquor as an Adhesive on the Performance of Plywood

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# Abstract

**Background and Objectives:** Adhesives are one of the most important ingredients in plywood industry. Black liquor (BL), a liquid comprising waste from the pulping process, was used to reduce adhesive consumption in plywood production. The characteristics of BL and properties of phenol-formaldehyde-based black liquor as an adhesive on the performance of jabon (*Anthocephalus cadamba* Miq.) plywood were investigated. **Materials and Methods:** Adhesive mixes were prepared using phenol-formaldehyde (PF) with the seven kraft BL-based adhesive added (0, 5, 10, 15, 20, 25 and 30% (w/w). Three-ply panels were produced at glue spread of 180 g cm<sup>-2</sup>, then pressurized at 10 kg f cm<sup>-2</sup> at a temperature of 140°C for four minutes (PF-BL plywood). The physical and mechanical properties of PF-BL plywood were analyzed according to Standard Nasional Indonesia (SNI) and Japanese Agricultural Standard (JAS). **Results:** The crystallinity of BL was 25.88%, mean while methanethiol (CAS) mercaptomethane, ethanedioic acid (CAS) oxalic acid, methanethiobis-(CAS) 2-thiapropane, phenol 2-methoxy-CAS guaiacol, phenol 2,6-dimethoxy-CAS 2,6 dimethoxy phenol were the dominant of chemical components in BL. Jabon plywood bonding with PF-BLadhesiveup to 30% of BL addition, met the SNI and JAS standards. The best treatment for PF-BL plywood was reached at 15% of BL addition. **Conclusion:** Up to 30% BL addition can be used to reduce PF resin in order to produce PF based plywood. BL was a potential material for decreasing the use of PF-based adhesive for plywood.

Key words: Black liquor, phenol-formaldehyde, adhesive, Anthocephalus cadamba Miq., plywood

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**Competing Interest:** The authors have declared that no competing interest exists.

Data Availability: All relevant data are within the paper and its supporting information files.

#### INTRODUCTION

Currently, raw materials for veneer plywood employ a large range of wood produced from community forests, along with a declining timber supply from natural forests. In 2009, the Indonesian plywood industry export value was 3.1 million m<sup>3</sup>. This figure excluded domestic plywood products, representing 1.5 million m<sup>31</sup>. Thee<sup>2</sup> reports that in the past decade, the plywood industry reached a value of more than 5.6 million m<sup>3</sup> and 5.1 million m<sup>3</sup> for the export market.

In the plywood industry, adhesives are not only an important product, it is also extremely costly. Of the total cost of various forms and types of composite wood production, adhesives represented 32% of overall cost<sup>3</sup>. To reduce the use of base adhesives, extenders can be added as a substitution matter. An extender is an adhesive material that is added to the adhesive base to improve strength and reduce the costs of production<sup>4</sup>.

Black liquor (BL) is a liquid derived from the waste products of the pulping process for pulp and paper making. It is a sticky material, as it contains significant quantities of lignin. Indonesian pulp mills produce black liquor at a rate of more than 23 million tons per year<sup>4</sup>. Approximately 48% of the pulp cooking solution is black liquor and 18% of black liquor is made up of lignin<sup>5</sup>.

BL has been used as materials for energy<sup>6</sup>. On the other hand, BL has been used as an adhesive for various applications in the field of composites. Several studies have employed BL for making adhesives<sup>7-10</sup>, as well as a medium for mushroom cultivation<sup>11</sup>. Lignin isolated from black liquor can be utilized as a raw material in thermosetting adhesives for composite wood product<sup>7-9</sup>. Lignin was copolymerized with resorcinol formaldehyde (lignin resorcinol formaldehyde/LRF)<sup>12,13</sup>. In contrast to previous research studies, in this study BL was added to phenol-formaldehyde adhesive without isolation and copolymerization. These process will reduce the cost and more efficient.

Research on black liquor characterization and its utilization of phenol-formaldehyde glue extender for community forest plywood remains limited. Therefore, in an effort to highlight utilization of BL waste as an extender for adhesives, the present study was carried out. The objectives of the recent study were to characterize BL and to evaluate the physical and mechanical properties of jabon *(Anthocephalus cadamba* Miq.) plywood bonded with PF resin at various levels of BL addition. Jabon wood was preferred, since it is a fast-growing species widely grown by people in Indonesia. A jabon tree at seven years of age can reach a diameter of 38 cm. People harvest jabon wood at five-to-seven years, due to the high demand for it, making it useful for increasing farmers' income. Fast-growing jabon from community forests is generally rotary-cut to produce veneer for plywood, com-ply and LVL<sup>14</sup>.

#### **MATERIALS AND METHODS**

The study was carried out in the Laboratory of Wood, School of Life Sciences and Technology, Institut Teknologi Bandung, Indonesia and PT. Sumber Graha Sejahtera, Tangerang, Indonesia, from June to October, 2017, according to the following scheme.

**Materials:** Jabon veneer with a thickness of 2 mm and a density of 0.4 g cm<sup>2</sup> was obtained from PT. Sumber Graha Sejahtera, Tangerang, Indonesia. Veneer was dried in oven until reaching a moisture content of 8-10%. The kraft black liquor (BL) with solid content (SC), lignin content and pH values of 48, 17.6 and 12.1%, respectively, was obtained from PT. Pindo Deli Pulp and Paper Mills, Karawang, Indonesia. Phenol-formaldehyde (PF) resin with SC, viscosity, gelation time, specific gravity and pH of 41-45%, 1.8 poise to 2.4 poise, 5-15 min, 1.19-1.20 and 10-13, respectively, was supplied by PT. Pamolite Adhesive Indonesia (PAI), Jakarta, Indonesia.

#### Methods

**Characterization of black liquor:** Gas chromatograph-mass spectrometry (GC-MS), Shimadzu GCMS-QP (USA) was employed to examine the chemical compounds of BL. The observation process was carried out by placing 2 mg of a sample of a powdery gap inserted into a seal capsule, then inserted into quartz in a unit of pyrolysis. The sample was heated in an oxygen-free environment with a retention time of 60 min, at a temperature of 400°C.

Crystallinity of black liquor was identified using a X-ray Diffractometer (XRD) (Philips PW 1050 X-pert Diffractometer, Germany), 40 kV angle voltage 2 10-80 with a speed of  $2^{\circ}$ C min<sup>-1</sup>. The crystallinity was measured by comparing the crystalline region with the amount of crystalline and amorphous regions, according to Eq. 1 below:

$$Crystallinity (\%) = \frac{Crystalline region}{Crystalline + amorphous regions} \times 100$$
(1)

**Preparation of mixed adhesives from PF Resin and BL:** Exactly 0, 5, 10, 15, 20, 25 and 30% (w/w) black liquor was added to PF resin (PF-BL). The mixture (PF resin and black liquor) was compounded using a mechanical stirrer (Haake Model Rheodrive 500) at 3000 rpm, for 15 min. The PF-BL was employed to analyze and characterize the properties of this adhesive, these properties include gravity, solid content, gelation time and viscosity, according to SNI 06-4567-1998.

**Preparation of PF-BL plywood:** Jabon wood veneers (300 mm by 300 mm by 2.00 mm) with a moisture content of 5% (dry weight basis) was used. A roll spreader (Black Bros. Inc.,) was used to apply the adhesive mix to core veneers at a rate of 180 g-2, using a double glue line basis. Three-ply panels were immediately assembled in sets pressurized at 10 kgf cm<sup>-2</sup>, at a temperature of 140°C for four minutes to produce a PF-BL-based plywood (PF-BL plywood). All specimens were conditioned at an ambient temperature of  $25\pm3^{\circ}$ C (room temperature) and at a relative humidity of  $30^{\circ}$ C $\pm 2\%$  for two weeks prior to testing.

#### **Characterization of PF-BL plywood**

**Moisture content and density:** The moisture content (MC) of plywood was evaluated according to JPIC<sup>15</sup>. Three-pieces of  $50 \times 50$  mm specimens in each panel were used to determine the density and MC using the gravimetric method. The MC and density were calculated using Eq. 2 and 3:

$$MC (\%) = \frac{Mass before drying (g)-Oven-dried mass}{Oven-Dried mass (g)} \times 100$$
 (2)

Density 
$$(g \, cm^{-3}) = \frac{Air - dried mass}{Air - Dried volume (cm^{-3})}$$
 (3)

**Shear strength:** Shear strength specimens of  $25 \times 80$  mm were prepared and tested using a testing machine (Instron Model 4204, USA) for type I test, according to SNI 01-5008-2-2000. The lengths, widths and thicknesses of the samples were measured and recorded. Samples were tested at a crosshead speed of 10 mm min<sup>-1</sup> and span of 240 mm.

**Determination of formaldehyde emission:** The Japanese standard method (JAS 1751-2008) was employed as a 24 h desiccator method. Emission of formaldehyde was determined by placing test pieces of a known surface area in a desiccator at a controlled temperature and measuring the quantity of emitted formaldehyde absorbed in a specified volume of water during 24 h. The interior volume of the desiccator was 11 L and  $50 \times 150$  mm specimens were used for each desiccator test. The specimens were cut from sample panel of panel segments to obtain adequate representation of

area within the panel or panel segment. The released formaldehyde was caught in the distilled water, which was analyzed using a UV spectrophotometer, after treatment with acetyl acetone and acetyl acid ammonium. The specimens were then conditioned on an edge and spaced apart, so air could freely circulate across all surfaces for seven days at  $20^{\circ}C\pm1.5$  and  $50^{\circ}C\pm10\%$  relative humidity.

**Delamination test:** The delamination test was conducted following the method of JAS standard No.1751-2008. Test pieces were immersed in boiling water for four hours and dried at a temperature of  $60\pm3^{\circ}$ C for 20 h and were then immersed in boiling water for four hours and dried at a temperature of  $60\pm3^{\circ}$ C for 3 h. The delamination ratio was calculated using Eq. 4:

 $Delamination ratio(\%) = \frac{\text{Total length of dela min ation (mm)}}{\text{Total length of glueline (mm)}} \times 100$  (4)

**Morphology of bonding properties:** A macro microscope (NIKON SMZ 745T, Japan) was used to analyze morphological images of how the glue spread on the veneer in jabon plywood materials. Images from this analysis employed 30×magnification.

#### **RESULTS AND DISCUSSION**

#### **Characteristics of black liquor (BL)**

**Chemical components:** The chemical components of black liquor are shown in Table 1. Methanethiol (CAS) mercaptomethane, ethanedioic acid (CAS) oxalic acid, methane thiobis-(CAS) 2-thiapropane, phenol 2-methoxy-CAS guaiacol, phenol 2,6-dimethoxy-CAS and 2,6-dimethoxy phenol were the dominant chemical components of BL. Meanwhile, black liquor from kraft pulping contained lignin, hydroxy acid, acetic acid, resin and fatty acid, hemicellulose and sugar, formic acid, sodium and other components<sup>16</sup>. Lignin compounds were isolated from black

Table 1: Chemical components of black liquor

Compound	Concentration (%)
Ethanedioic acid (CAS) oxalic acid	25.21
Oxirane (CAS) Epoxyethane	3.62
Methanethiol (CAS) Mercaptomethane	43.55
Methane, thiobis-(CAS) 2-thiapropane	12.76
Ammonium penta methylene dithiocarbamate	1.85
Phenol 2-methoxy-CAS guaiacol	4.09
Phenol 2,6-dimethoxy-CAS 2,6 dimethoxy phenol	4.12
Trans-hexa-2,4 dienyl acetate	1.24
Zink dicyclopentyl-CAS	2.70
Butane, i-chloro-3-methyl-CAS 1-Cloro-3-methyl butane	0.86



Fig. 1: XRD pattern of crystalline and amorphous black liquor ranging from 20 (5-90°)

Table 2: Properties of PF-BL adhesive						
Black liquor (BL)			Viscosity	Gelation		
addition (%)	SG*	SC**(%)	(poise)	time (min)		
0	1.19	43.10	3.4	30.0		
5	1.21	43.70	4.5	29.0		
10	1.21	43.90	5.4	27.0		
15	1.22	44.20	6.5	26.5		
20	1.22	44.90	8.0	25.5		
25	1.23	45.30	11.5	24.0		
30	1.24	46.00	13.5	23.0		

\*Specific gravity, \*\*Solid content

liquor consisting of phenol, 2-methoxyphenol (guaiacol), 2,6-dimethoxy phenol (syringol), 4-ethyl-2-methoxyphenol, 4-hidroxy-3,5 dimethoxybenzaldehyde and 1-(4-hydroxy-3-methoxyphenyl) ethanone<sup>17</sup>.

**Crystallinity:** The crystallinity of BL is shown in Fig. 1. Based on Fig. 1, the degree of crystallinity of kraft black liquor was 25.88% and the amorphous area of this BL was 74.12%. When comparing the amorphous and crystalline regions, the amorphous region was found to be larger than the crystalline region. Crystallinity changed according to heat and heating was able to degrade the hydroxyl group of cellulose that began in the amorphous region, then the semi-crystalline region and terminated in the crystalline region<sup>18</sup>.

**Characteristics of PF-BL adhesive:** The test results for the PF-BL adhesive properties including specific gravity (SG), solid content (SC), viscosity and gelation time are presented in Table 2.

The PF-BL adhesive was liquid and a blackish-brown color. The range value of SG, SC and the viscosity of PF-BL adhesives ranged from 1.19-1.24, 43.10-46.00% and 3.4-13.5 poise, respectively. The highest specific gravity, solid content and viscosity were obtained at a 30% BL addition, while the lowest values were found at 0% BL addition. The higher the addition of black liquor, the more SG, SC and viscosity of PF-BL mixed adhesive tended to increase. This was thought to be related to a higher concentration of black liquor (48.00%) than that of PF resin (43.10%). Solid content and viscosity decreased alongside an increasing extension level, it appeared that decreasing the viscosity of the adhesive caused a decrease in viscosity<sup>19</sup>. The viscosity value will affect the adhesive's ability to flow from one surface to another on a wood that is bonded to form a continuous thin film, causing it to spread evenly on the entire surface<sup>20</sup>.

Furthermore, gelation time of the PF-BL mixed adhesive at a temperature of 100°C was 23-30 min. The fastest gelation time was obtained at 30% BL addition, while the longest was found at 0% BL addition. The higher the addition of black liquor, the faster the gelation time of PF-BL adhesive. The gelation time is the time it takes to convert the liquid adhesive into a gel.

#### **Characterization of PF-BL plywood**

**Moisture content and density of plywood:** The results of moisture content (MC) and the density tests of PF-BL plywood are presented in Fig. 2. Based on Fig. 2, the range value of moisture content for jabon plywood ranged from 12.61-13.53%. The variation between these values was not very large. However, it was observed that with the addition of black liquor, the moisture content of plywood tended to increase. Kraft black liquor material consisted of lignin. The presence of hydroxyl function groups, primarily phenolic hydroxyl groups, resulted in the increased hydrophilicity of lignin<sup>16</sup>. The moisture content of plywood met SNI and JAS requirements. MC values of all plywood were lower than 14%.

The density values of jabon plywood ranged from 0.47-0.48 g cm<sup>-3</sup>. These results indicate that the density of PF-BL plywood was higher than that of solid jabon wood (0.40 g cm<sup>-3</sup>). The pressing process was thought to increase the density of plywood. Adhesives and pressing load have been reported as creating higher density plywood, compared to the same processes for solid wood<sup>20.21</sup>.



Fig. 2: Moisture content and density of PF-BL plywood



Fig. 3: Shear strength and wood failure of PF-BL plywood

Table 3 <sup>,</sup> Formaldeh	vde emission	(FF) of PF-BI	plywood
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Black liquor	FE	Classification of
Addition (%)	(mg L <sup>-1</sup> )	FE (JAS)
0	0.29	F****/F4S
5	0.20	F****/F4S
10	0.19	F****/F4S
15	0.16	F****/F4S
20	0.11	F****/F4S
25	0.08	F****/F4S
30	0.08	F****/F4S

FE: Formaldehyde emission, \*: The value met the JAS classification, the number of star showed the good quality from plywood-based on formaldehyde emission

Shear strength and wood failure of PF-BL plywood: The influence of BL addition on the shear strength of jabon plywood in the type I test is shown in Fig. 3. The shear strength and wood failure of plywood were found to be in the range of 7.28-11.51 kg cm<sup>-2</sup> and 28.3-46.7%, respectively. The highest shear strength and wood failure of plywood were achieved by PF-BL-bonded plywood at 15% BL addition and the lowest were achieved by PF-BL-bonded plywood at 30% BL addition. All plywoods met SNI requirements (shear strength value >7 kg cm<sup>-2</sup>).

Based on Fig. 3, addition of BL up to 15% tended to increase shear strength and wood failure. The increasing shear strength effectively were 8.3%. On the contrary, the

addition of BL from 15% up to 30% tended to decrease shear strength and wood failure in jabon plywood. This was believed to be related to the bonding guality of the PF-BL mixed adhesive. Excess use of an extender will degrade the guality of plywood. The addition of an extender tends to decrease the bonding strength of plywood, the higher the level of extension, the lower the bonding strength of plywood<sup>4,19</sup>. Black liquor is a potential material for decreasing the use of PF resin. Up to 30% of PF resin can be replaced by black liquor in the case of jabon plywood. This result is similar to previous research, which found phenol could be replaced by kraft lignin up to a total of 30%, without any major changes in plywood shear strength<sup>9</sup>. Black liquor from the pulping of bagasse was used as a partial substitute for phenol in the preparation of a phenolic adhesive for plywood made from Vateriaindica and Ulmuswalliciana and up to a 50% substitution proved satisfactory for meeting the requirements of boiling water resistant test standard<sup>22</sup>. The use of lignin in black liguor as an adhesive for bamboo plywood can cut 28.69% of costs<sup>10</sup>.

The morphology of wood and adhesive bonding from jabon plywood panels with 0, 15 and 30% BL addition is presented in Fig. 4 (a-c). The addition of 30% black liquor to PF resin (w/w) caused wood adhesive bonding to not remain

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Fig. 4(a-c): Morphology of wood bonding, observed using a macro microscope at 30× magnification: (a) 0% BL, (b) 15% BL and (c) 30% BL



Fig. 5: Delamination ratio of PF-BL plywood

even (Fig. 4c). There were voids between veneer and adhesive in the glue line, which meant the bonding was not as strong as it used to be. The addition of BL therefore affected the quality of the adhesive.

**Delamination ratio:** The value of delamination ratio is presented in Fig. 5. The range value of the delamination ratio was 0-2%. Thus, the value of delamination for all plywoods was less than 5%, indicating these plywoods passed the delamination test according to the JAS standard. The more black liquor was added to PF, the higher the value of jabon plywood delamination. The highest delamination ratio was reached at 30% BL addition. Smaller delamination ratios indicated better performance for jabon plywood. It was clear

that jabon plywood that bonded with the PF-BL adhesive with a 15% BL addition had the highest shear strength and lowest delamination ratio.

**Formaldehyde emission of jabon plywood:** As can be seen in Table 3, a high amount of FE, measured by gas analysis method, was observed from PF-BL plywood. The gas analysis values for 0, 5, 10, 15, 20, 25 and 30% BL-addition ranged between 0.08 and 0.29 mg L<sup>-1</sup>.

The lowest value of formaldehyde emission was obtained at 30% BL addition, while the highest was found at 0% BL addition. The formaldehyde emission of jabon plywood met the F4S (F\*\*\*\*) classification of the JAS standard, indicated by the emission values lower than 0.3 mg L<sup>-1</sup>. Therefore, formaldehyde emission of all jabon plywood did not pose serious environmental consequences. A serious drawback of formaldehyde-based resin has been the emission of the hazardous formaldehyde during cure<sup>23</sup>.

#### CONCLUSIONS

Chemical components of kraft black liquor with 25.88% crystallinity were dominated by methanethiol (CAS) mercaptomethane, ethanedioic acid (CAS) oxalic acid, methane, thiobis-(CAS) 2-thiapropane, phenol 2-methoxy-CAS

guaiacol, phenol 2,6-dimethoxy-CAS 2,6 and dimethoxy phenol. Moisture content and shear strength of plywood bonding with PF-BL adhesive met the standard of SNI. Delamination ratio and formaldehyde emission of jabon plywood bonding with the PF-BL adhesive met the JAS requirements. Jabon (*Anthocephalus cadamba* Miq.) plywoods bonded with PF-BL adhesive up to 30% of BL addition and also met the SNI and JAS standards. The best treatment of PF-BL plywood was reached at 15% BL addition. Up to 30% BL addition can be used to reduce PF resin, in order to produce PF-based plywood. Black liquor is a potential material for decreasing the used of PF-based adhesive for plywood.

#### SIGNIFICANCE STATEMENT

This study discovered the chemical components and crystallinity of kraft black liquor (BL). BL material consists of lignin, which is a phenolic compound. Additionally, this study discovered the maximum addition of BL to phenol-formaldehyde (PF) for jabon plywood application, which meet SNI and JAS standards. This study can also assist the plywood Industry in Indonesia to decrease PF adhesive cost through the utilization of black liquor as an extender of PF adhesive for jabon plywood application.

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