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Original Article

Effect of Phenol Formaldehyde Impregnation on The Physical and Mechanical Properties of Soft-Inner Part of Oil Palm Trunk¹

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ABSTRACT

The objective of this study was to improve physical and mechanical properties of soft-inner part of oil palm trunk (S-OPT) after impregnation with phenol formaldehyde (PF) resin and densification by close system compression (CSC) method. Effect of different methods of PF resin impregnation (*i.e.*, no vacuum-pressure, vacuum, and vacuum-pressure) was evaluated. The results showed that PF resin impregnation and CSC significantly improved the physical and mechanical properties of S-OPT up to: (1) 176% in density; (2) 309% in modulus of rupture (MOR); (3) 287% modulus of elasticity (MOE); and (4) 191% in the compressive strength. Physical and mechanical properties of S-OPT showed their best performances when PF resin impregnated with vacuum-pressure method as shown by higher weight gain, density, MOR, MOE, compressive strength, and lower recovery of set due to better penetration of PF resin into S-OPT. Combining PF resin impregnation and densification by CSC method could be a good method to improve physical and mechanical properties of S-OPT.

Keywords: close system compression, phenol formaldehyde impregnation, physical and mechanical properties, soft-inner part of oil palm trunk

1. INTRODUCTION

Oil palm has developed into one of the most economically important plantation crops in Southeast Asia (Koh and Wilcove, 2007). Among countries in this region, oil palm in-

dustry in Indonesia was able to grow rapidly with the plantation expanding at an annual rate of more than 12% from 1990 to 2005 (Hashim *et al.*, 2012). In 2013, the total oil palm plantation in Indonesia stands at 10.46 million hectares, with more than 63% of the oil palm plan-

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tations located in Sumatera Island and 30% are planted in Kalimantan Island (Directorate General of Estate Crops, 2014). The crude palm oil (CPO) production in 2013 peaked at 27.78 million tons, contributing to more than 50% of world production and positioned the country as the world's biggest producer of palm oil.

Oil palm trees have an economic life span of 24-26 years (Ismail and Mamat, 2002). After this age, it is usually felled for replanting due to decreased oil productivity. In Indonesia, 450,000 ha of oil palm plantation area are expected to be replanted annually during the next 25 years. Such large area will generate large amount of potential biomass particularly in the form of trunks. During replantation the land owners are face with a big problem of disposal of felled oil palm trunks (OPT). These biomasses are commonly dumped off by burning or lands filling which put significant negative impact on the environment due to pollution created. Therefore, the potential utilizations of OPT should be explored to develop productive uses for OPT and mitigating environmental problems from waste biomass.

Previous studies have reported properties of various panel products from OPT, such as sawntimber (Bakar *et al.*, 2006), particleboard (Nadhari *et al.*, 2013; Lamaning *et al.*, 2014), medium density fiberboard (Ibrahim *et al.*, 2014), laminated veneer lumber (Sulaiman *et al.*, 2009), and plywood (Abdul Khalil *et al.*, 2010a; Feng *et al.*, 2011; Hoong *et al.*, 2012). However the utilization of OPT as raw materials for such products limited by abrupt differ-

ences in physical and mechanical properties between the outer parts and inner parts of OPT. For example, Bakar et al. (1998) reported that the green moisture content (MC) of outer part was 258%, while the inner part was as high as 575%. Lim and Khoo (1986) stated that the density of OPT decreases linearly towards the center of the trunk, showing density values over twice those of the inner part. Previous studies revealed that only one-third of the OPT diameter and three-fourths of the height could be potentially used for light-weight building materials and furniture, while the remaining inner two-thirds of the trunk generally becomes waste due to its low dimensional stability (high moisture content, high shrinkage-swelling), low strength, low durability, and low machining properties (Bakar et al., 2006; Erwinsvah, 2008; Iswanto et al., 2010).

Densification by compression method is a promising method to improve the properties of the soft-inner part of oil palm trunk (S-OPT). In previous work, we have studied the densification of the inner part of S-OPT by close system compression (CSC) under different temperature and duration (Hartono et al., 2011; Hartono et al., 2016). The results showed that compression by the CSC method successfully increased the density, modulus of elasticity (MOE), modulus of rupture (MOR), and compressive strength of S-OPT up to 90%, 190%, 155%, and 120%, respectively (Hartono et al., 2016). The impregnation of phenol formaldehyde (PF) resin into OPT and wood has been reported to improve dimensional stability

Table 1. Properties of PF Resin

Properties	Unit	Value	
pH (at 25℃)	-	12.55	
Specific gravity (25℃)	-	1.20	
Viscosity (at 25°C)	Poise	2.25	
Gelation Time (at 135℃)	Min', Sec"	12'45"	
Resin Content* (at 135℃)	0/0	42.50	
Molecular weight	Mn	4000	

^{*} Non-volatile solid content

(Furuno *et al.*, 2004; Rowell, 2005), physical and mechanical properties (Shams *et al.*, 2006; Bakar *et al.*, 2013), and increased the resistance against fungal decay and termites attack (Nabil *et al.*, 2016). The use of PF resin for OPT impregnation has many advantages, such as low viscosity, lack of odor, low volatility, less heat generation at polymerization, good polymerization completion, and low in price (Abdul Khalil *et al.*, 2010b). To further improve the physical and mechanical properties of S-OPT, in this study we attempted to apply hybrid methods through the combination of PF resin impregnation and compression by CSC method.

2. MATERIALS AND METHODS

2.1. Materials

OPT was collected from 40-year-old plantation in Bogor, Indonesia. The samples of S-OPT were sawn into pieces of lumbers having dimensions of 150 mm \times 50 mm \times 20 mm in length, width, and thickness, respectively. To determine their initial density, samples were dried at 60 $^{\circ}$ C until they reached a constant weight, and then the weight and dimensions

were measured. The average density of the samples after drying was 0.34 ± 0.01 g/cm³.

Phenol formaldehyde (PF) resin was impregnated into the cell lumen of S-OPT. The PF resin was purchased from the Palmolite Adhesive Company in Indonesia. The properties of the PF resin used in this study are shown in Table 1.

2.2. Methods

2.2.1. PF resin impregnation and densification by CSC method

Before PF resin impregnation, a group of samples were hot-pressed to a compression ratio of 50% at 100°C for 4 h and another group of samples were not hot-pressed for comparison. Aqueous solution of 20% PF resin was prepared for impregnation. Three methods of PF resin impregnation were applied, including:

- No vacuum-pressure method: samples were soaked in the PF resin solution for 24 h, no vacuum or pressure was applied,
- Vacuum method: samples were soaked in the PF resin solution and vacuumed at 600 mmHg for 1 h,
- 3. Vacuum-pressure method: samples were

soaked in the PF resin solution and vacuumed at 600 mmHg for 1 h. Pressure of 1 N/mm² for 30 min was then applied.

Four samples were used for each treatment. After PF resin impregnation, curing was performed in an oven-dryer at 60°C for 15 hours.

The samples were then compressed by CSC method. CSC is a compression method to improve permanent fixation of compressive deformation in a short time by hygrothermal treatment using moisture in wood or other lignocellulosic materials (Inoue et al., 1993). Detail description of the CSC method can be seen in our previous publication (Hartono et al., 2016). The samples were compressed with a target compression ratio of 50% at 135℃. After 10 min, hot water vapor inside CSC frame was released and then the compression process was continued for another 10 min. After compression by CSC method, the samples were conditioned under a relative humidity of 70 \pm 5% at 25 \pm 2°C for 2 weeks before further testing. The final size of the samples after compression was 150 mm × 50 mm × 10 mm in length, width, and thickness, respectively.

2,2,2. Physical properties evaluation

The air-dry density of samples after PF resin impregnation and compressed by CSC method was determined by measuring their air-dry weight and volume using sample dimensions of $20 \text{ mm} \times 20 \text{ mm} \times 10 \text{ mm}$. Weight gain percentage (WG%) was determined by measuring samples weight before impregnation (w_o) and weight after PF resin impregnation (w_i), and

then calculated using the equation:

$$WG (\%) = \frac{w_i - w_o}{w_o} \times 100\% \cdots (1)$$

The anatomical structure after PF resin impregnation and compression by CSC was observed. Sliding microtome was used to make sections with 30 µm in thickness. The sections were stained with Safranin-Astra blue, dehydrated in a graded series of alcohol solutions (50%, 70%, 90%, 95%, and 99%), mounted in Canada balsam, and observed with an optical microscope (Nikon Eclipse E600, Japan).

The recovery of set (RS) was obtained using samples with dimensions of 30 mm \times 20 mm \times 10 mm by soaking the samples in room-temperature water (20 \pm 3°C) for 24 h and then in hot water (100 \pm 3°C) for 30 min (Dwianto *et al.*, 1999). The RS was calculated as follows:

$$RS \ (\%) = \frac{T_r - T_c}{T_o - T_c} \times 100\% \cdots (2)$$

where T_0 is the initial thickness, T_c is the thickness after compression, and T_r is the thickness after recovery. All the dimensions of the samples were measured in the oven-dry condition.

2,2,3, Mechanical properties evaluation

Modulus of rupture (MOR), modulus of elasticity (MOE), and compressive strength tests were conducted using a universal testing machine (Model 4482, Instron, USA). The sample dimensions for the MOR and MOE tests were 150 mm × 10 mm × 10 mm. Three-point bending was applied over an effective span of 100 mm at a loading speed of 5 mm/min. The

Impregnation method -	WG (%)		Density (g/cm ³)		RS (%)	
	Without HP	With HP	Without HP	With HP	Without HP	With HP
Control	-		0.34 ^A (0.01)	-	-	
No vacuum-pressure	11.52 ^A (2.47)	10.59 ^A (2.12)	0.70 ^{BC} (0.04)	0.66^{B} (0.06)	40.25 ^D (6.40)	26.11 ^C (6.78)
Vacuum	28.37 ^C (2.90)	17.39 ^B (2.60)	0.77 ^{DC} (0.04)	0.72^{BCD} (0.05)	3.56 ^A (1.40)	13.55 ^B (2.24)
Vacuum-pressure	50.94 ^E (6.38)	36.68 ^D (4.01)	0.94 ^E (0.06)	0.80 ^D (0.07)	1.92 ^A (0.34)	2.50 ^A (1.33)

Table 2. Physical properties of S-OPT after impregnation with PF and compression by CSC method

Notes: HP: hot pressing prior to PF resin impregnation; means within a parameter (two columns) followed by the same capital letter are not significantly different at 1% significance level using Duncan's multiple range test. Numbers in parenthesis are standard deviations.

dimensions of the samples for the compressive strength test were 40 mm \times 10 mm \times 10 mm. The MOR, MOE, and compressive strength (R) were calculated as follows:

$$MOR (N/mm^2) = \frac{3 \times P \times L}{2 \times b \times t^2} \cdots (3)$$

$$MOE (N/mm^2) = \frac{P_p \times L^3}{4 \times Y_p \times b \times t^3} \cdots (4)$$

$$R (N/mm^2) = \frac{P}{b \times t} \cdots (5)$$

where P is the maximum load (N), P_p is the load at the proportional limit (N), Y_p is the deflection (mm), L is the span length (mm), b is the sample width (mm), and t is the sample thickness (mm).

2.2.4. Data analysis

The experimental design was a completely randomized design (CRD). The results of the properties tested were submitted to an overall analysis of variance (ANOVA) using SPSS version 16.0. The homogeneity of the means among combinations was tested using the Duncan's Multiple Range Tests.

3. RESULTS AND DISCUSSION

3.1. Physical properties

Physical properties of S-OPT before and after PF resin impregnation and CSC are shown in Table 2. The results showed that the WG and density of samples without hot pressing was significantly higher than samples with hot pressing prior to PF resin impregnation, and the results were consistent for all three impregnation methods. Hot pressing prior to PF resin impregnation made the S-OPT structure become more compact, resulting in the difficulty for PF resin to penetrate. For impregnation method with no vacuum-pressure, samples with hot pressing showed lower RS compared to samples without hot pressing prior to PF resin impregnation. However, when using and vacuum-pressure impregnation methods, the samples with hot-pressing showed significantly higher RS than samples without hot pressing. Lower RS indicated better dimensional stability and better fixation.

The combination of PF resin impregnation

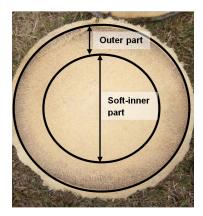
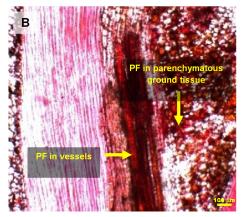


Fig. 1. Soft-inner part of oil palm trunk (S-OPT) used for this study.

and CSC of S-OPT resulted in higher density achieved compared to only CSC. The combination of PF resin impregnation and CSC increased the S-OPT density by 94-176%, significantly higher than the increase of density by using only CSC of 3-90% (Hartono *et al.*, 2016).

The PF resin impregnation method (i.e., without hot-pressing and with vacuum-pressure) of S-OPT resulted in the highest WG and density with the lowest RS. The values indicated that the treatment resulted in a better the PF resin penetration, higher compression ratio, better distability, better fixation. mensional and Vacuum-pressure method resulted in better WG, density, and RS in both samples without and with hot-pressing prior to PF resin impregnation. In addition, statistical analyses showed that WG and density of S-OPT treated without hot-pressing prior to PF resin impregnation were significantly higher than S-OPT treated with hot-pressing, while RS values showed no significantly different.



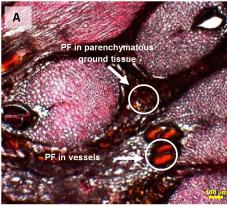


Fig. 2. Micrographs of S-OPT at (A) transverse section and (B) radial section: PF impregnated parenchymatous ground tissue and vessels of S-OPT (20× magnification).

During vacuum-pressure, vacuum could evacuate the air inside of S-OPT, and pressure forced PF resin to penetrate into S-OPT. The pressure may opened up parenchyma and vessel, and it makes PF resin easier to enter, hence the WG value was the higher. Vessels and parenchyma cells filled out with PF resin and after compression by CSC method can be seen in Fig. 2.

Similar to wood, OPT is a hygroscopic material. The hydrophilic properties of OPT

Table 3. Mechanical properties of OPT after impregnation with PF and compression by CSC method

Impregnation method	MOR (N/mm²)		MOE (N/mm²)		Compressive strength (N/mm²)	
	Without HP	With HP	Without HP	With HP	Without HP	With HP
Control	12.08 ^A (1.16)	-	1191.26 ^A (64.06)	-	7.80 ^A (0.96)	-
No vacuum-pressure	37.75 ^{BC} (3.17)	35.24 ^B (2.30)	2882.89 ^{BC} (366.15)	2479.98 ^B (273.88)	16.65 ^{BC} (2.15)	14.32 ^B (1.89)
Vacuum	42.37 ^D (1.83)	40.96 ^{CD} (3.10)	3861.00 ^D (200.01)	3204.00 ^C (546.62)	18.69 ^C (0.74)	15.86 ^B (1.61)
Vacuum-pressure	49.47 ^E (2.87)	44.81 ^D (2.93)	4615.94 ^E (443.91)	4187.68 ^{DE} (217.94)	22.75 ^D (1.45)	21.37 ^D (2.01)

Notes: HP: hot pressing prior to PF resin impregnation; means within a parameter (two columns) followed by the same capital letter are not significantly different at 1% significance level using Duncan's multiple range test. Numbers in parenthesis are standard deviations.

may be caused by its thin cell walls and large lumens (Erwinsyah ,2008, Abdul Khalil *et al.*, 2008). The S-OPT became moisture-resistant by the polymerization of the cured resins; hence more PF resin penetration or higher WG resulted in the increase of dimensional stability as shown by an increase of density and reduction of RS. The results were in agreement with previous studies. Furuno *et al.* (2004) reported an increase of dimensional stability of wood and bamboo after impregnation of PF resin. Gabrielli and Kamke (2008) stated that PF resin impregnation contributed to an increase of dimensional stability and reduction of thickness-swelling of wood.

3.2. Mechanical properties

The control sample of S-OPT had MOE of 12.08 N/mm² and compressive strength of 7.8 N/mm² and classified as very poorly strong (Strength Class V) according to Indonesian Standard SNI 01-0608-1989 (BSN, 1989). The

PF resin impregnation with vacuum-pressure method could increase the MOR strength class into Strength Class III (moderately strong) and compressive strength into Strength Class IV (poorly strong). Overall results showed that the impregnation of PF resin and compression by CSC method significantly increased mechanical properties of S-OPT by 191-309%, 108-287%, and 83-191% for MOR, MOE, and compressive strength, respectively (Table 3). The increase of mechanical properties using the combination of PF resin impregnation and CSC of S-OPT was higher than that using only CSC with MOE, MOR, and compressive strength of 43-192%, 14-155%, 19-123%, respectively (Hartono et al., 2016).

Table 3 shows that S-OPT treated without hot-pressing prior to PF resin impregnation had better mechanical properties than that of S-OPT treated with hot-pressing. The MOR, MOE, and compressive strength increased in order after the sample treated with no vacuum-pressure, vacuum, and vacuum-pressure, respectively with

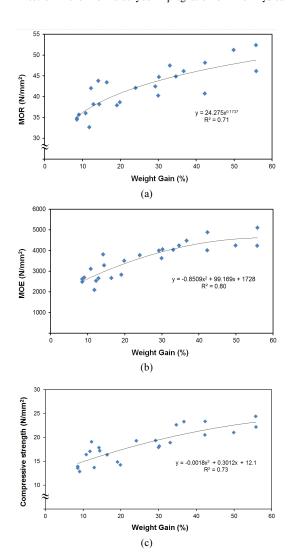


Fig. 3. Weight gain of S-OPT after PF resin impregnation and compression by CSC versus: (a) MOR, (b) MOR, and (c) compressive strength.

the highest value achieved by S-OPT treated with vacuum-pressure method.

Mechanical properties were attributable to WG value. The higher WG resulted in the higher MOR, MOE, and compressive strength. Fig. 3 shows the relationship between WG with

mechanical properties. The regression analysis showed that the correlation between WG and MOR could be described by power fit, while the relationship between WG and MOE, WG and compressive strength could be described by a second-order polynomial.

4. CONCLUSION

The S-OPT was impregnated by PF resin and compressed by CSC method. PF resin impregnation by no vacuum-pressure, vacuum, and vacuum-pressure method significantly improved physical and mechanical properties of compressed OPT. The assessed physical and mechanical properties showed their best performances when PF resin impregnated with vacuum-pressure method. Combining PF resin impregnation and compression by CSC method could be a good method to improve physical and mechanical properties of soft inner part of OPT.

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