Effects of Heat Treatment on The Wettability and Color Properties of Betung Bamboo (*Dendrocalamus Asper*) Strand

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Abstract. Betung bamboo (*Dendrocalamus Asper*) has recently attracted interest as a sustainable reinforcement fiber in (polymer) composite materials. Utilization of bamboo has advantages and disadvantages. It has a short growth cycle, lightweight, and good mechanical properties, but with poor wettability, dimensional instability, and low resistance against biodeterioration agents. Some efforts have been devoted to alter and enhance its properties, especially wettability properties. Heat treatment under various temperatures can enhance wettability properties due to chemical changes and migration of extractives, but also can induce color changes. Therefore, this study aims to investigate wettability properties and color changes of heat-treated Betung bamboo strand. The 1/3 middle of Betung bamboo was used as main material and converted into strand. Heat treatment temperatures applied were 140°C and 160°C for 3 hours. The wettability of Phenol Formaldehyde (PF) adhesive on heat treated Betung bamboo strand was measured using a sessile drop contact angle method. Constant contact angle and K-value were used as parameter of wettability. Color change of heat-treated Betung bamboo strand was measured by portable color difference meter model CDX 105 and characterized by CIE Lab. Results showed that the increasing temperature of heat treatment affected wettability and color properties. Contact angle tended to increase with an increase of temperature which is indicated an enhancement of wettability by making wood more hydrophobic. Increasing of temperature made Betung bamboo strand to be darker than that of control sample. Total color change (ΔE) values were 21.06 and 29.23 at 140 and 160°C, respectively.

Keywords: Betung Bamboo (*Dendrocalamus Asper*) strand, color properties, heating time, heat treatment, wettability properties

1. Introduction

In recent years Bamboo as an excellent substitution material for wood due to its use for many applications (Sulaeman et al., 2018). Based on BPS (2017), the availability of bamboo in
Indonesia is high with the production rate reached ± 14 million culms. Bamboo has a rapid growth rate with a short life cycle than wood, compared to wood, bamboo has advantages, including relatively low price, resilience, straight, easy to split, and easy to shape (Febrianto, Fauzi, Sumardi, 2017). Bamboo also has several disadvantages, mainly due to its small diameter which makes it difficult to use especially in wide dimension products.

Composite products are an alternative ways to increase the efficiency of using bamboo as a construction component. Oriented strand board (OSB) is a structural composite panel that substitutes for plywood can substitute using bamboo as raw material. According to SBA (2005), OSB is a structural composite board made of a material in the form of long, complete, and thin strands stacked parallel to each other, bonded with an exterior adhesive under pressure and heat. Also, bamboo OSB (BOSB) has better mechanical properties than wood OSB (F. Febrianto, 2009). However, BOSB still uses MDI adhesive, which is quite expensive. The Study of using adhesives such as phenol-formaldehyde (PF) have been conducted, but higher concentrations are needed for BOSB to meet commercial standards (Febrianto & Sadiyo, 1995). Therefore, to overcome this problem, the pretreatment of strands needs to be done. The treatment of steam (hydrothermal) on the strand is an option in improving the quality of BOSB. Several studies have shown that steam treatment strand at 126°C for one hour has been shown to improve the quality of BOSB produced (Maulana et al., 2017, 2019; Fatrawana et al., 2019; Maulana et al., 2020). Several of studies regarding the steam treatment of strands showed that steam treatment successfully improved the quality of BOSB bonded using PF adhesive. Steam treatment is a heat treatment that belongs to hydrothermal. (Stamm et al., 1946, Inoue et al., 1993, Rowell et al., 2002). Therefore, The main object of this work was to study the influence of heat treatment on the wettability properties strand.

2. Materials and Methods

2.1 Materials

The main material used is four years old betung bamboo from Sukabumi, West Java, Indonesia. The density of betung bamboo is 0.57 g/cm³. The culm of bamboo was converted into strand that manually produced using a sharp-knife and scissor. The length, width, and thickness dimension of the strands were 70, 25, and 0.80 mm, respectively. Phenol formaldehyde (PF) adhesive with 42% solid content was used to determine wettability of control and heat treated strand.

2.2 Strand modification

The strands were modified by the hygrothermal method. Strands were air-dried for ±7 days and followed by oven-dried at 75–80 °C for ± 3 days. Heat treatment was applied on strand at 140°C and 160°C for 3 hours. Heat treated strands were re-oven-dried at 75 – 80 °C for ± 3 days to achieve 5% of moisture content (MC).

2.3 Wettability Test

Wettability was determined through contact angle of the PF adhesive against the heat treated strand. The strand sample was placed on a flat table parallel to the camera. The adhesive was dropped on the surface of samples by using sessile drop method (droplet volume 0.02 ml) in
five different area for each sample. Dropped adhesive was recorded for 170 seconds, cut for every 10 seconds with GOM Player software, and obtained 18 images for each dropping video. Contact angle for every images were measured by using Image-J 1.46 with a drop snake plugin analysis. The curve of contact angle against time was generated. The constant contact angle value was determined by the regression equation between time (x) and contact angle (y) using SAS PROC NLIN program. Then, the K-value of the S/G model (Shi and Gardener 2001) was determined by XLSTAT program. The K-values were obtained using the wetting model equation of the S/G model as follows:

$$\theta = \frac{\theta_i \theta e}{\theta_i + (\theta e - \theta_i) \exp \left[ K \left( \frac{\theta e - \theta_i}{\theta e - \theta_i} \right) t \right]}$$

(1)

Where "\(\theta\)" is the angle at a given time, "\(\theta_i\)" is the first contact angle, "\(\theta e\)" is the equilibrium contact angle, t is time, and K is the constant rate of change of the contact angle.

2.4 Color parameters measurements

Color properties of control and heat treated strand was obtained by determining the brightness (L *), red-green (a*) and yellow-blue (b*) values using portable color difference meter model CDX-105 in five different area. The values obtained were converted online through colorhexa page. Total color change value (ΔE) was calculated based on the CIE Lab method with the following formula:

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$$

(2)

where $\Delta E =$ total color change, $\Delta L =$ the brightness difference, $\Delta a =$ the red or green color difference, and $\Delta b =$ the yellow or blue color difference.

3. Results and Discussion

3.1 Wettability Properties

The contact angle formed between a surface and a liquid provides useful indication related to performance of adhesive wets. Wettability of bamboo is one of key indicators of how substrate responds with liquid. This can be conducted by using contact angles measurement. When the contact angle is zero, a complete wetting occurs, while above 90° indicates lack of wetting. A contact angle value above 90° degrees indicates poor wettability (Bracco & Holst, 2013).
Contact angle changes as a function of time at different temperature is shown on Figure 1(b). The contact angle of heat treated betung bamboo strands markedly increased than that of control samples. The value tended to increase with an increase of temperature. The best treatment condition for betung bamboo strand to achieve maximum contact angle was 160°C for 3 hours. Increasing of temperature can successfully reduce the amount of hydroxyl groups in the cellulose chains, leads to a lower moisture uptake, thus consequently reduce water adsorption, and may affect wettability properties (Pétrissans et al., 2003). This would be increased surface hydrophobicity. Bamboo was sensitive to alkali-based adhesives such as PF, and required proper adhesive formulation to ensure sufficient curing of the resin.

Table 1. Constant contact angle change rate (K-value) at different temperature

| Sample    | \( \theta_i \) | \( \theta_c \) | K (L/sec) |
|-----------|----------------|----------------|-----------|
| Control   | 89.71          | 57.93          | 0.082     |
| 140 °C    | 93.4           | 60.89          | 0.027     |
| 160 °C    | 95.53          | 73.772         | 0.013     |

As shown in Figure 2, it can be seen that the contact angle had changed since the first 10 seconds of observation. Variation of contact angle value would be eventually affected the K-value. K-value is an indicator to describe how fast wetting process by liquid occurred on surface area (Bracco & Holst, 2013). Therefore, it could be explained the accuracy of the wetting process on betung bamboo strand’s surfaces. The lower K-value obtained means lower the wettability (Shi & Gardner, 2001). The greater K-value means shorter time to reach a constant angle and faster a liquid will spread and penetrate into betung bamboo strand’s surface (Y. Huang et al., 2019). Table 1 also showed that heat treatment at 160 °C had the lowest K-value compared to that of 140 °C. Thermal treatment can improve the durability of bamboo against degrading agents (Shi & Gardner, 2001). During this process, free in lignocellulosic materials lead to improve the board properties. Steam treatment of strands has been reported to improve the properties of OSB.
3.2 Color difference

Heat treatment caused color change on betung bamboo strands. The strand turned to be darker as temperature increased. According to Table 2, the L* values of heat treated betung bamboo strand tended to decrease, while the a* and b* values tended to increase compared to that of control sample. The same phenomenon was also reported by (Hon & Minemura, 2001), the a* and b* values have an opposite behaviour with the L* value. According to Srinivas & Pandey, (2012), the L* value represents the brightness (lightness) which ranges from 100 (pure white) to 0 (black), while +a* represents reddish color, −a* represents green, + b* represents yellowish, and −b* represents blue.

| Sample  | L*   | a*  | b*  | ΔE  |
|---------|------|-----|-----|-----|
| Control | 71.465 | 6.019 | 28.741 | -   |
| 140 °C  | 50.995 | 10.959 | 29.019 | 21.059 |
| 160 °C  | 42.808 | 11.391 | 26.599 | 29.235 |

Table 2 shows that the brightness (L*) of heat treated betung bamboo strand decreased with the increase of temperature. This value was lower than that of control. This could be due to degradation of hemicellulose and migration of extractive component. As wood is progressively heated to higher temperature, production of condensable fraction occurs, with loss of water and volatile extractives at temperature below about 140°C. Above this temperature, cellular breakdown occurred and produced acetic acid, formic acid, methanol from hemicellulose and non-condensable gases (mainly CO2), as well as dehydration reaction began to occur leading to a decrease of OH content (Hill, 2006). According to Sandqvist, (2002), Pelit, (2017) and De Cademartori et al., (2014), thermally modified treatment can reduce the L* value due to chemical components changes, especially degradation of amorphous polysaccharides (hemicellulose). The degradation will increase as the temperature increased (Huang et al., 2012). Nuopponen et al., (2003) also confirmed that temperature of heat treatment around 100-160°C is resulting in fat and waxes moved along the axial parenchyma cells to surface area. Meanwhile, the a* and b* values was higher than that of control (Table 2). Higher the a* and b* values were generally associated with condensation, degradation and oxidation of lignocellulosic materials. According to De Cademartori et al., (2014), condensation process occurs in lignin and other extractives and allows the formation of by-products, thereby contributes to the increasing of red tone intencity (reddish color).

Total color change is expressed as ΔE value (Turkoglu et al., 2015). Table 2 shows that total color change of heat treated bamboo strand increased with an increase of temperature. This phenomenon was supported by colorhexa results, for control sample was slightly desaturated orange (● #ccaa7b), while bamboo strands heat treated were both resulted dark moderate orange (● #9a7148 and ● #845d39) at 140°C and 160°C, respectively. The color change occurs due to hydrolysis of hemicellulose which leads to an increases of ΔE value (Sehlstedt-Persson, 2003)). The sampe phenomenon was reported by other researchers, Schneid et al., (2014) stated that *Luehea divaricate* heat treated wood results in total color changes (ΔE) around 1.18-1.85 at
160°C for 2 h. In addition, Akkuş & Budakçı, (2020) stated that ∆E values of beech heat treated are 3.89 and 18.01 at 140°C for 3 h and 160°C for 7 h, respectively.

4. Conclusions
Heat treatment can affect wettability and color properties of betung bamboo strands. The best treatment condition for heat treated betung bamboo strand to achieve maximum contact angle is 160°C for 3 hours. Increasing of temperature will eventually reduce the amount of hydroxyl groups, affect wettability properties and create more hydrophobic surface. Variation of contact angle value will eventually affect the K-value. Heat treatment at 160 °C has the lowest K-value compared to that of 140 °C and control samples. Color properties also changed during this process. The L* values of heat treated bamboo strand tended to decrease, while the a* and b* values tended to increase compared to that of control sample. The ∆E value of heat treated bamboo strand increased with an increase of temperature. This occurred due to degradation of hemicellulose and migration of extractive component to the surface area.

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