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Evaluation of surface properties of betung bamboo (*Dendrocalmus asper*) strands under various heat treatment duration and temperature

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Abstract: The abundance, rapid growth characteristics and good mechanical properties have made betung bamboo as good alternative materials to replace the slow growing wood. To address the susceptibility to insects and water attack, the making of bamboo composites, using resin as matrix, has been widely practiced. Thus, the surface properties of bamboo strands are crucial to determine the optimum interaction with resin. This study evaluates the effect of heat treatment to improve the surface properties of bamboo strands, such as the wettability and the color change. Beforehand, the freshly cut bamboo was cut, cleaned and sand-grinded. The heat treatment was done at 140° and 160°C for 1, 2, and 3 hours. After cooling down to room temperature, the contact angle of bamboo's surface was measured by sessile drop method. The wettability was indicated by constant of contact angle change rate, ***K***. Besides, the change of color was also determined. After the experiments, it was obtained that the bamboo surface tends to be more hydrophobic and has darker color with longer duration of heat treatment. Higher temperature and longer duration of heat treatment can cause more evaporation of liquid inside bamboo and decrease hydrophilicity of the surface.

Keywords: *betung bamboo (dendrocalmus asper), heat treatment, surface properties, wettability, color change*

Introduction

Since the prehistoric era, wood has been known as excellent materials for building, transportation, firewood, etc. As the human population keeps growing, so does the demand for wood. However, slow growing wood cannot fill the human needs and causes deforestation that destroys the ecosystems [1]. Among other plant candidates, bamboo appears as excellent an alternative to replace wood. While mature wood can be obtained after 20 – 30 years of planting, bamboo only requires 3 – 5 years at the height of 30 m and culm diameter of 20cm. Furthermore, bamboo can be easily planted on low nutrient soil [2]. Because of that, bamboo can be easily found from Russia to Australia and India. However the majority of bamboo species are originally from Asia [3]. Besides its cultivation characteristics, bamboo has similar mechanical properties as wood. In fact, bamboo is more ductile and easier to form than wood even though its values are varied as a function of distance from root [4].

In Indonesia, there are 161 bamboo species listed [5]. The abundance of bamboo in Indonesia is due to its ability to grow in poor soil nutrient. Thus, bamboo can grow in any type of soil except coastal land [2], [4]. Based on the report from Statistic Center of Indonesia (*Badan Pusat Statistik*) on 2017, Indonesia produced 14 million bamboo rods. People have used bamboo as construction material since a long time ago because of its availibility, light-mass, and good mechanical properties. However, the application of bamboo is limited to its relatively small diameter [4].

Bamboo belongs to the family of grasses with microscopic structure shown in **Figure 1**. For structure application, people often use the hollow culm of bamboo. Between nodes, there is a cavity. Each species of bamboo can be characterized by the average diameter of the culm and the stem wall thickness [6]. The bamboo culm wall contains a chain of cellulose and hemicellulose inside a matrix of lignin [7]. Bamboo can also be classified based on vascular bundle. Betung bamboo has the vascular bundle type IV in which it consist of three parts: a vascular bundle center and two fibre bundle [8].

Similar to wood, bamboo also possesses weakness of bio-organism and water attack. One of the solution is to prepare bio-composites using resin as matrix. Among many, bamboo-oriented strand boards (BOSB), which is prepared by compressing the resin-wetted-bamboo-strands at specific temperature and pressure, has been widely applied [9]. Previously, the addition of phenolic resin onto the strand of large-diameter-betung-bamboo to prepare BOSB has been improving perfomance and application of bamboo [10], [11]. However, this method proposes a new challenge: the interaction between bamboo strand surface and resin, in order to create strong physical bond that determines the final mechanical properties. Thus , the efficient and enviromentally friendly hygrothermal method can be applied as pre-treatment to the bamboo strands [7].

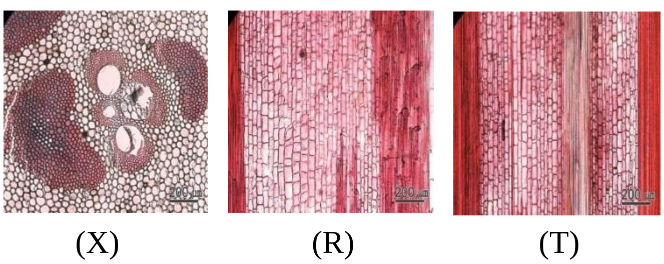


Figure 1. Microscopic structure of betung bamboo (*Dendrocalmus asper*) (X: cross section; R: radial section; T: tangential section) [8]

Hygrothermal treatment is a treatment in which superheated steam is applied to the bamboo strands in order to improve the surface interaction between bamboo strands as filler and polymeric resin [12]–[14]. At the beginning of heating, the lignin was softenned due to depolymerization and it contributed to the adhesion of bamboo [15]. When the temperature reaches the glass transition temperature, the mechanical properties of bamboo are increasing with the improvement of cell wall cristallinity due to autocatalytic reaction [16], [17]. A previous report showed that the rigidity of bamboo was increased by 3.8 – 8.8% with the heating at 100° – 140°C [18]. However, further increase of temperature at 180°C only improved the hardness but the Young's modulus was stagnant [19]. In addition, heat treatment at temperature above the glass transition temperature of bamboo will cause degradation of the polymeric chain, causing increase of brittleness [20]. In other hand, the influence of heat treatment to the surface properties of bamboo, such as wettability and color change has not well understood. In fact, this information is crucial in bamboo composite making. Thus, an experiment of betung bamboo heat treatment was designed at 140° and 160°C whose duration is 1, 2, and 3 hours.

Method

Materials

Four years old betung bamboo (*Dendrocalmus asper*) are harvested in Sukabumi, West Java, Indonesia. The density of bamboo culm is 0.57 g/cm^3. The mechanical method was used to obtain the bamboo strands. In this research, we used bamboo strands whose length, width, and thickness of 70, 25, and 0.8 mm respectively. Furthermore, phenol formaldehyde (PF) resin was used as polymeric resin for wettability test.

Bamboo Heating Treatment

After the betung bamboo strands were obtained mechanically, they went through two steps of drying: (1) sun dry for 7 days continously, (2) oven dry for 3 days at 75° – 80°C. This drying process is to minimize the moisture content that can decrease the hardness [21] and induce bio-organism attack. Then, the heat treatment was applied by blowing hot air (hygrothermal method) at 140° and 160°C for 1, 2, and 3 hours. After cooling to room temperature, the bamboo strands were re-dried in the oven for 3 days at 75° – 80°C to obtain 5% of moisture content.

Wettability Test

Wettability is the ability of surfaces to spread and to be penetrated by liquid. In bamboo composite making, it decides the amount of resin needed to bind all bamboo strands. The wettability test was done using sessile drop technique [22] to measure the contact angle of phenol formaldehyde (PF) drop on the bamboo surface. The image of 0.02 ml PF drop was obtained by high-speed camera that positioned parallelly to the sample holder. Continuous images were captured for 170s to determine the dynamic contact angle (θ). Equation 1 is used to calculate the equilibrium contact angle (θ_e) and the constant of contact angle change rate (K).

$$\theta = \frac{\theta i.\theta e}{\theta i+(\theta e-\theta i)exp\left[K\left(\frac{\theta e}{\theta e-\theta i}\right)t\right]} \quad \textbf{(1)}$$

θ_i is the contact angle at 0 second. The statistical analysis was done by PROC NLIN program from SAS.

Color Parameter Analysis

Heating may improve the color change of bamboo surface as it indicates the depolymerization of polymeric chain structure. The quantification of color change was done by a portable color difference meter CDX – 105 to determine the brightness intensity of the combination of red – green and blue – yellow. Equation 2 is used to compute the change in color intensity.

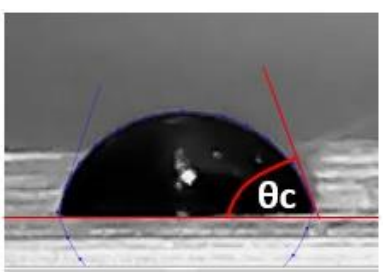
$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad \textbf{(2)}$$

ΔE is the color intensity change, ΔL is the brightness change, Δa is the color difference of red – green, and Δb is the color difference of yellow blue. The value of $\Delta L, \Delta a, \Delta b$ were obtained from equipment.

Results And Discussion

The main purpose of bamboo heat treatment is to eliminate the extractive compounds, such as inorganic compound, sugar, tanin, and starch. The extractive compounds is started to evaporate at 140ºC [23]. The lose of extractive compound may modify the surface properties of bamboo.

Figure 2. Measurement of PF contact angle to bamboo strand surface.



In the contact angle measurement, when the contact angle between liquid drop and the surface is below 90º, it means the surface is hydrophilic. While, if the contact angle is above 90º, the surface is classified as hydrophobic.

Firstly, as seen in **Figure 2**. the line marking was made to indicate the baseline (contact line between the flat bamboo surface and resin droplet) and drop outline. Then, the contact angle was computed as the tangent of the intersection between the baseline and the drop outline. Contact angle was obtained on the left and right side of the droplet.

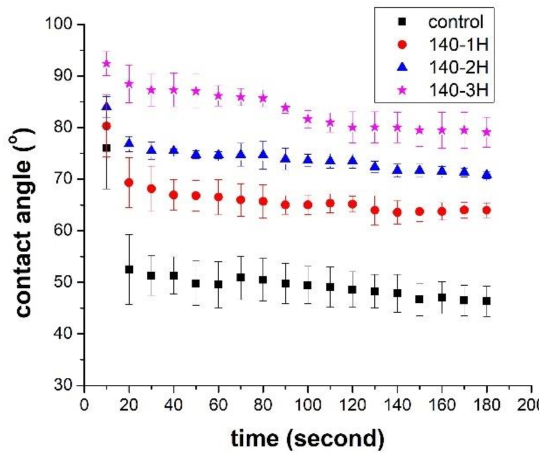


Figure 3. Measurement of PF contact angle to bamboo strand surface after heat treatment at 140ºC as function of time

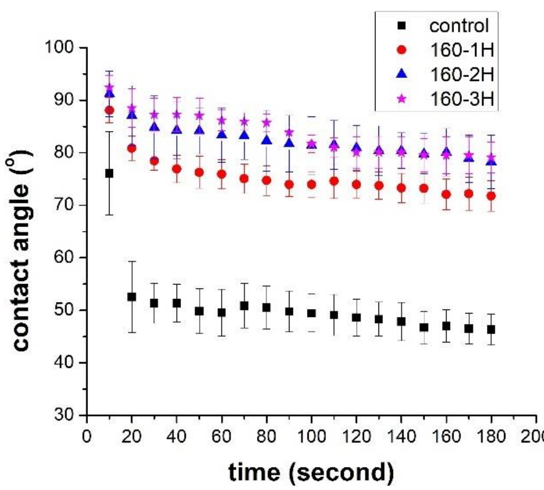


Figure 4. Measurement of PF contact angle to bamboo strand surface after heat treatment at 160ºC as function of time

Contact angle follows the time function. As the time goes by, liquid droplet has more time to spread and penetrate the surface porous. When it reach equilibrium state, the contact angle has been considerably constant. **Figure 3**. shows the evolution of contact angle of bamboo strands as function of time, both untreated and heat-treated at 140ºC. In the span of 180 seconds, the contact angle of all samples was slowly decreasing as the surface absorb the liquid. However, the initial contact angle was varied with the duration of heat treatment.

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Longer duration of heating causes the surface become more hydrophobic (higher contact angle).

Figure 4. shows the change of contact angle with time for samples treated at 160°C. In general, the surface behaves similarly as the contact angle decreases with time. When the initial contact angle is compared as a function of heating duration, the result is also similar with the samples heated at 140°C. The 3 hours of heating modifies the surface to be more hydrophobic. However, the value obtained after heating at 160°C is higher than the one measured after 140°C heat treatment.

In previous study reported that the wettability of bamboo surface could be decreased by heat treatment at 100 to 180 °C based on their experiment [24]. The same phenomenon happened in our experiment at temperature 140 and 160 °C. Lignin is thermoplastic amorphous polymer whose glass transition temperature of 150°C [15]. Previously, it is reported that the chain of cellulose and hemicellulose are more subtle to the increase of temperature since heat can induce hydrolysis reaction, resulting shorter chain of oligomers and monomer [25]. When the temperature increases, the hydroxyl groups in microfibril start to degrade, resulting in a decrease of hygroscopicity of bamboo [26], [27]. Hygroscopicity is the ability of surface to attract and hold water molecule through absorption. At the same time, the acetyl groups are separated from the polymeric chain, obtaining carbonic acid groups [28]. Extractive contents and hemicellulose that have a low molecular weight were decreased by the heat treatment [29]. As the cellulose and hemicellulose chains are decreasing, the content of lignin is increasing [26] causing the softening effect to the bamboos's structure [15]. Thus, autocatalytic reaction is occuring in the cell wall, contributing to the improvement of polymer's crystallinity [16], [17]. Consequently, the mechanical properties of bamboo strands are increasing.

Wettability properties of the surface is quantified by constant *K* to indicate the wetting rate of liquid on the surface. Low value of constant K means the surface is more hydrophobic or low wettability [30]. Inversely, the high value of K constant shows that the surface can be easily spread and penetrated by liquid [31].

As lignocellulosic materials, such as wood, are continuously heated below 140°C, there will be water loss and evaporation of volatile extractive components. Macroscopically, the heating around glass transition temperature (T_g) of bamboo can improve the mechanical properties of bamboo since the polymeric chains undergo chain straightening. Consequently, bamboo structures become denser and the porosity decreases [32] resulting in difficulty for liquid to spread and penetrate the surface. In other words, heating at T_g can improve the hydrophobicity of the bamboo surface. Because of that, heating at higher temperature, without surpassing the limit of T_g, and longer duration can increase the contact angle of bamboo strands (see **Table 1**). A previous report stated that heating at temperature between 100° – 160° C can move the fat and waxes along the axial parenchyma cells to the surface [33]. However, heating at temperature above T_g will cause depolymerization or the breaking of long polymeric chains into oligomers and monomers. Because of that, the porosity increases and the bamboo strands fraction decreases. Thus, mechanical properties in general are declining [34].

Table 1. Color intensity change of untreated and treated betung bamboo strands

Treatment	Time	θi	θϵ	*K* (L/s)
Control	-	89.71	57.93	0.082
HT 140 °C	1 hour	80.320	63.986	0.062
	2 hours	83.991	70.838	0.026
	3 hours	92.436	79.110	0.019
HT 160 °C	1 hour	88.101	71.779	0.019
	2 hours	91.191	78.263	0.015
	3 hours	92.436	79.11	0.013

In fact, heating modifies not only the wettability of the bamboo surface, but also the color of bamboo. The quantification of color change parameters was done by CIELAB method (eq. 2). The parameter of brightness change (*L*) is ranged between pure black 0 to pure white 100. Meanwhile, parameter *a* that indicates the red – green color is ranged between bright red *+a* to bright green- *a.* Parameter *b* spans from bright yellow *+b* to bright blue *-b* [35]. As shown in **Table 2** the value of parameter ΔE is declining when the samples heated for longer duration, indicating that the bamboo is darker when it is heated at higher temperature and longer duration. Inversely, the parameter *a* is increasing and it is related with condensation and oxidation of cellulose, hemicellulose, and lignin. Condensation of lignin results in the formation of by-products, contributing to the intensity of reddish color [36].

The value of color intensity change (ΔE) was considerably small since the bamboo was heat treated at dry condition (5% moisture content). Heat treatment of wood with higher moisture content was found to give dramatic color change (high value of ΔE parameter) [37].

It was reported in wood that the degradation of amorphous microfibril and the increased amount of lignin can modify the surface to be darker [36], [38], [39]. Further heating can results in a decrease in surface brightness of wood [40]. Assume that the majority composition of bamboo are similar with wood, the same explanation to the bamboo surface color change can be applied

Table 2. Color intensity change of untreated and treated betung bamboo strands

Treatment	Time	L*	a*	b*	△E
Control	-	71.465	6.019	28.741	-
HT 140 ºC	1 hour	66.923	8.135	32.453	1.286
	2 hours	58.355	10.256	31.155	-6.459
	3 hours	50.995	10.959	29.019	-15.253
HT 160 ºC	1 hour	65.283	8.409	31.129	-1.405
	2 hours	44.007	11.423	26.593	-24.202
	3 hours	42.808	11.391	26.599	-25.427

Conclusions

The effect of heat treatment of bamboo strands to the surface properties has been investigated. The wettability of surfaces is quantified by computing the value of *K* parameter or the constant of contact angle change rate. Bamboo that was heat treated at 160ºC for 3 hours was found to have the lowest value of *K,* indicating the surface is more hydrophobic. Besides, the bamboo strands that were treated at higher temperature for longer duration also result in darker color or lower value of parameter ΔE. Thus, heat treatment at 160ºC for 3 hours is more recommended as pre-treatment in bamboo oriented strand board (BOSB). The temperature chosen is 10 degrees higher than the glass transition temperature of bamboo fiber, so the softenning effect can be minimized. The wettability of bamboo strands after heating at this condition is considered to be optimum to minimize the use of expensive polymer resin while maintaining the strong interaction between bamboo and resin. More hydrophilic surface (smaller contact angle) requires a higher volume of resin, causing an increase in BOSB production cost. Further mechanical properties analysis of BOSB made of heat treated betung bamboo strand are required to formulate the optimum matrix / fiber ratio.

Conflicts of interest

There are no conflicts of interest to declare.

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