Wettability of *Dendrocalamus asper* under Various Heating Time during Composites Making Process

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Abstract. Bamboos are often used due to their abundance, fast growth rate, cheap price, ductility, and formability in the sector of transportation, musical instrument, cooking ware, etc. For construction purposes, *Dendrocalamus asper*, (locally known as “Bambu betung”), is used as bamboo composites. The properties of bamboo are highly improved by using phenolic resins (phenol-formaldehyde) as an adhesive in bamboo composites. Bamboo is stronger and more susceptible to any liquid. The liquid is the most common enemy to plant-based structure due to the softening effect. Thus, the wettability of bamboo is an important matter as it indicates the ability of a liquid to spread and penetrate on the surface. The objective of this study was to analyze the effect of various heating times to wettability properties of heat-treated *D. asper* strands during the bamboo-composite making process. Before testing, the bamboo was cut, cleaned, and sand-grinded. The bamboo strands were heated at 140°C for 1, 2, and 3 hours. Then, the surface of bamboo was evaluated by measuring the contact angle based on the sessile drop method. The constant contact angle was obtained by calculating the regression formula between time (t) and contact angle (θ). The wettability was indicated by the value of K. The color of heat-treated bamboo strands was measured by portable color difference meter model CDX 105 and characterized by CIE Lab. The results showed that the longer the holding time, the surface became more hydrophobic, showed by a higher contact angle. Longer heating gives more time to the resin to spread more evenly into the pores of bamboo so that the hydrophobicity of bamboo composites is increased. The lightness value (L*) of heat-treated bamboo strands tended to decrease with increasing temperature and time of heat treatment. To conclude, for further making the process of bamboo composites using *D. asper*, we recommend curing the composites for 3 hours.

Keywords: bamboo composite materials, *Dendrocalamus asper*, heating holding time, heat treatment, wettability
1. Introduction

The augmentation of human population has increased the demand of wood for construction, transportation, paper-making, firewood, and recently for biomass. However, the availability of wood supply cannot keep up with the demands [1]. Consequently, the alternative materials to replace the wood become the urgent need to fulfill. The appropriate raw materials should have similar range of strength, rigidity, and ductility as wood. Besides, the price, the cultivation characteristic, and the processing method are also taken into consideration. After long research, bamboo is considered as fitted to be wood substitution due to its high growing rate, wide spread, and inexpensiveness.

The majority of industrial application exploit the culm of bamboo which consist of hollow stem and visible nodes. The cavity is found in internodes part [2]. The variation of stem wall thickness and diameter characterize each species of bamboo. Similar with wood, bamboo is also a renewable material that consist of polymeric chain of cellulose, hemicellulose, and lignin [3]. However, bamboo comes under family of grasses Poaceae, subfamily Babusoideae [4] and has distinct morphology and cellular structures as shown in Fig. 1.

Compared with wood that requires 20 – 30 years to be harvested, bamboo can be produced only in 3 – 5 years [6]. Beside, bamboo can be planted on soil with soil nutrient (Hunter, 2003). The habitat of bamboo is stretched from Russia to North Australia and west India though 65% of bamboo population are found in Asia [7]. Among approximately 1250 bamboo species in the world, Indonesia is the home for 100 species [8]. The Statistic Indonesia (locally known as Badan Pusat Statistik) reported on 2017 that Indonesia produced approximately 14 million bamboo stems from island of Java, Bali, Nusa Tenggara, Sumatera, and Sulawesi [9]. Regarding the mechanical properties, bamboo is more ductile thus it has better formability than wood. However, due to its relatively small diameter, the application of bamboo is limited. Besides that, the physical properties of bamboo has great variation for culm in the middle and end part. In addition, bamboo is susceptible of organism attack [10]. Thus, instead of using bamboo as single materials, it is highly recommended to use bamboo as filler in composites materials.

Among the existing engineered bamboo products, bamboo-oriented strand board (BOSB) has been widely developed for construction materials. BOSB is produced by compressing the aligned the bamboo strands at specific pressure and temperature [11]. The strands are previously coated by polymeric resin adhesive to improve the performance and application of bamboo. Several publications have reported the opportunity to use betung bamboo (Dendrocalmus asper) as raw material of OSB due to its culm size [12], [13]. Matured betung bamboo can rise up to 30 m high with 20 cm diameter [8].

In order to improve the dimensional stability an durability of bamboo as raw materials of OSB, the hygrothermal treatment was applied. This method is known to be efficient and environmentally friendly [3]. Besides, it can also treat not only the solid materials but also the composites. The application of superheated steam to BOSB can improve the binding between bamboo strands and polymer resin [14]–[16]. It has been also reported that heat
treatment of bamboo at 100 – 140°C is improving the Young’s modulus by 3.8 – 8.8% compared to the sample control [17]. At higher temperature of 180°C the Young’s modulus has become constant while the hardness is slightly increasing [18]. Further heating cause the degradation of bamboo polymeric chain, thus the bamboo became brittle [19]. The change in mechanical properties due to heat treatment is related to the respond of chemical component. Given the fact that the initial heating can induce autocatalytic reactions of the cell wall and consequently can improve the relative cristalinity [20], [21], heating can also modify the surfacic properties of bamboo strands. The surface modification of bamboo can control its ability to spread and to bind with the liquid adhesive. So, we design an experiment to investigate the effect of heating duration of 1, 2, and 3 hours at 140°C to the relative wettability of bamboo strands to the adhesive component.

2. Materials and Method

2.1 Materials
Betung bamboo (Dendrocalamus asper) are collected after four years from bamboo forest in Sukabumi, West Java, Indonesia. Early characterization has found the density of bamboo at 0.57 g/cm3. The bamboo strands are produced traditionally, using mechanical technique. Then, the bamboo strands are classified based on length, width and thickness of the strands. Only the strands with length, width and thickness of 70, 25, and 0.8 mm respectively, used in this research. As for the determination of wettability properties, phenol formaldehyde (PF) with 42% solid content was used. PF has been chosen as in the latter step, PF has been decided as adhesive in bamboo oriented strand board making.

2.2 Bamboo Strand Modification Method
Hygrothermal method was chosen to modify the strand as it is proven to be efficient and environmentally friendly. The previous research reported that the heating treatment can influence the structure of cellulose, hemicellulose, and lignin chain in bamboo. The heat may decrease the hydroxyl group so that it may improve the stability of bamboo dimension [3].

Firstly, the bamboo strands were dried in under sunlight for ± 7 days and continued with oven drying at 75 – 80°C for 3 days. Later, the bamboo strands were heat treated at 140°C for 1, 2, and 3 hours. Then the moisture content (MC) of 5% was obtained by re-drying in oven at 75 – 80°C for another 3 days.

2.3 Wettability Test
Based on sessile drop technique, wettability properties was quantified by measuring the contact angle of PF drop on bamboo strand surface. The wettability test apparatus consists of high-speed camera which is put parallel to sample holder. 0.02 ml PF was dropped on each surface. The liquid dropping was recorded for 170 seconds to observe the change of dynamic contact angle (θ) by the time. The equilibrium contact angle (θe) and the constant of contact angle change rate (K) was obtained by using equation 1. (θi) is the initial contact angle (t = 0 s). The statistical calculation was done by program PROC NLIN from SAS.

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

2.4 Color Parameter Analysis
The analysis of color parameter was done by measuring the brightness intensity of red-green and blue-yellow combination using portable color difference meter model CDX-105. Each sample was scanned at five different points to obtain the value the brightness change (ΔL), the red-green color difference (Δa), and yellow-blue color difference (Δb). Then, the color intensity change (ΔE) can be computed by using th Eq. 2.

\[ \Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \]
3. Results and Discussion
Surface is considered hydrophobic if the contact angle is higher than 90°, inversely contact angle lower than 90° is found in hydrophilic surface. Figure 2 showed a droplet of bamboo strand surface at 0 and 180 seconds. The contact angle is measured as the angle between the flat surface and the intersection line of the droplet. Measurement was done at the left and right side of the droplet. Later the average value was used for further computation. In the beginning, the contact angle is almost 90°, but by the time the adhesive is slowly spread on the surface so the contact angle is decreasing.

![Figure 2. Contact angle measurement of PF droplet on bamboo strand surface at 0 s (left) and 180 s (right)](image)

We have recorded the contact angle every 10 seconds in order to observe its evolution with the increasing of time (Figure 3). There is a decreasing tendency of contact angle with the increasing of time for all samples. As seen in droplets capture image and plot of contact angle versus time in Figure 3, the surface that was heated longer, is more hydrophobic. Table 1 shown the calculated equilibrium contact angle angle (θₑ) and the constant of contact angle change rate (K), obtained from Equation 1. The K value indicates the ability of liquid to spread on the surface or the decreasing rate of contact angle. Higher K value shows that the surface is more hydrophilic as the liquid easily spread out on the strands. With the longer duration of heat treatment, the K value is decreasing.
Figure 3. Evolution of PF droplet contact angle as function of time (left) and plot of contact angle versus time for untreated and heat treated bamboo strand (right)

| Sample            | $\theta_i$ | $\theta_e$ | $K$ (L/sec) |
|-------------------|------------|------------|-------------|
| Control           | 89.71      | 57.93      | 0.082       |
| 1 hour heating    | 88.099     | 60.89      | 0.027       |
| 2 hours heating   | 95.899     | 73.772     | 0.013       |
| 3 hours heating   | 102.15     | 79.874     | 0.0117      |

The difference in bamboo strands surface properties induced by heat treatment, is due to the change of chemical component structure. Heat application may cause pyrolysis of cellulose, hemicellulose, and lignin chain [20]. The precedent reports mentioned that cellulose and hemicellulose is more sensitive to heat treatment. Previously, it is reported that hemicellulose undergoes hydrolysis due to heating at specific temperature, resulting oligomer and monomer [22]. At the beginning of heating process, there is formation of carbonic acid because of acetyl group separation form hemicellulose chain [23]. There is an increase of lignin after heating while the cellulose and hemicellulose content is decreasing [24]. Consequently, the cell wall structure undergoes autocatalytic reaction that can increase the relative crystallinity of polymeric structure [20], [21]. In addition, the early step of heating can also causes degradation of hydroxyl groups in the amorphous area of microfibril [24], [25] thus it decreases the hygroscopcity of bamboo strands. So, there is a clear evidence of correlation between the chemical component structure change to the modification of surface properties.
Apart of change in wettability properties of bamboo strands, heat treatment can also modify its color. The difference in color is quantified by CIELAB method (Equation 2) and the results are presented in Table 2. The parameter $L^*$ has value range between 0 (pure black) to 100 (pure white). While the parameter $a^*$ represent the color of red (+$a^*$) and the color of green (-$a^*$); and the parameter $b^*$ is also ranged from -$b^*$ to +$b^*$ for yellow and blue respectively [26]. Table 2 shows that the color brightness ($L^*$) of sample is decreasing with the increase of heating duration. This results in are in agreement with the published article which reported that the value of parameter $L$ is inversely proportional to the value of $a^*$ and $b^*$. The degradation of amorphous regions in hemicellulose chain and the increase of lignin in early step of heating, can darken the wood surface [27]–[29]. Thus, higher heating temperature or longer heating duration may cause the wood lose its surface brightness [30]. Since bamboo has similar chemical component as wood, the same phenomenon may be applied on heat treated bamboo.

The values color intensity change ($\Delta E$) found in this research are relatively small and it indicates that the color change is not dramatic. Application of high heating temperature to the wood in wet condition caused drastic color change because it increases the amount of hemicellulose that undergoes hydrolysis (Sehisted-Persson, 2003). Meanwhile, the heating temperature in this experiment is considered to be low and the hygrothermal treatment method was done in dry condition, so, the difference in color less significant.

### Table 2. Color Parameter Analysis of untreated and heat treated bamboo strand

| Treatment   | Time  | $L^*$  | $a^*$  | $b^*$  | $\Delta E$ |
|-------------|-------|--------|--------|--------|------------|
| Control     | -     | 71.465 | 6.019  | 28.741 | -          |
| HT 140 °C   | 1 hour| 66.923 | 8.135  | 32.453 | 1.286      |
| HT 140 °C   | 2 hour| 58.355 | 10.256 | 31.155 | -6.459     |
| HT 140 °C   | 3 hour| 50.995 | 10.959 | 29.019 | -15.253    |

### 4. Conclusion

The bamboo strand heat treatment can be classified into three steps. During the initial heating, the mechanical properties and the hydrophobicity is improved due to the elimination of extractive component. After reaching the optimum condition in term of heating temperature and/or duration, the bamboo strands become softer due to the formation of oligomer and monomer. Further heating can induce the degradation of chemical component and worsen the overall properties. From the result of experiment, it is recommended to heat treat the bamboo strand at 140°C for 3 hours with the purpose of optimization of wettability properties and color stability. Even though hydrophilic surface can improve the binding interaction strands and adhesive, the required volume fraction of adhesive is relatively high. The adhesive liquid can easily spread out and be absorbed through the porous bamboo. Consequently, the strands fraction is minimized and the overall mechanical properties of BOSB is decreasing [31].

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### References

[1] P. Chaowana, “Bamboo: An Alternative Raw Material for Wood and Wood-Based Composites,” *J. Mater. Sci. Res.*, vol. 2, no. 2, 2013, doi: 10.5539/jmsr.v2n2p90.
[2] R. Anokye, E. S. Bakar, J. Rattnasingam, and B. K. Awang, “Bamboo Properties and Suitability as a Replacement for Wood,” *PJSRR Pertanika J. Sch. Res. Rev.*, vol. 2, no. 1, pp. 63–79, 2016, doi: 10.13140/RG.2.1.1939.3048.
[3] C. Ye, Y. Huang, Q. Feng, and B. Fei, “Effect of Hygrothermal Treatment on the Porous Structure and Nanomechanics of Moso Bamboo,” *Sci. Rep.*, vol. 10, no. 1, pp. 1–11, 2020, doi: 10.1038/s41598-020-63524-4.
[4] Z. Qisheng, J. Shenxue, and T. Yongyu, *INDUSTRIAL UTILIZATION ON BAMBOO*, no. 26. Beijing: Colour Max Publisher Limited, 2001.
[5] T. Gangwar and D. Schillinger, “Microimaging-informed continuum micromechanics accurately predicts macroscopic stiffness and strength properties of hierarchical plant culm materials,” *Mech. Mater.*, vol. 130, pp. 39–57, 2019, doi: 10.1016/j.mechmat.2019.01.009.
[6] F. A. Troya Mera and C. Xu, “Plantation Management and Bamboo Resource Economics in China,” *Cienc. y Tecnol.*, vol. 7, no. 1, pp. 1–12, 2014, doi: 10.18779/cyt.v7i1.93.
[7] P. Md. Tahir, *Bonding with Natural Fibres*, vol. 1. Serdang: Universiti Putra Malaysia Press, 2013.
[8] S. Dransfield and E. A. Widjaja, *Bamboos*. Leiden: Backhuys publishers, 1995.
IOP Conf. Series: Earth and Environmental Science 830 (2021) 012012      doi:10.1088/1755-1315/830/1/012012

9. Badan Pusat Statistik, “Statistik Produksi Kehutanan,” Jakarta, 2017.
10. F. Febrianto, I. Sumardi, W. Hidayat, and S. Maulana, Papan Untai Bambu Berarah-Material Unggul untuk Komponen Bahan Bangunan Struktur, 1st ed. Bogor: IPB Press, 2017.
11. S. B. Association, “Oriented Strand Board in wood frame construction,” Ontario, 2005.
12. F. Febrianto et al., “Properties of oriented strand board made from Betung bamboo (Dendrocalamus asper (Schultes f.) Backer ex Heyne),” Wood Sci. Technol., vol. 46, no. 1–3, pp. 53–62, 2012, doi: 10.1007/s00226-010-0385-8.
13. F. Febrianto et al., “Effect of bamboo species and resin content on properties of oriented strand board prepared from steam-treated bamboo strands,” BioResources, vol. 10, no. 2, pp. 2642–2655, 2015, doi: 10.15376/biores.10.2.2642-2655.
14. S. Maulana, B. D. Purusatama, N. J. Wistara, and I. Sumardi, “Pengaruh Perlakuan Steam pada Strand dan Shelling Ratio terhadap Sifat Fisis dan Mekanis Oriented Strand Board Bambu (Effect of Steam Treatment on Strand and Shelling Ratio on the Physical and Mechanical Properties of Bamboo Oriented Strand Board ).”
15. A. Fatrawana et al., “Changes in chemical components of steam-treated betung bamboo strands and their effects on the physical and mechanical properties of bamboo-oriented strand boards,” Eur. J. Wood Wood Prod., vol. 77, no. 5, pp. 731–739, 2019, doi: 10.1007/s80107-019-0426-7.
16. S. Maulana, I. Sumardi, N. J. Wistara, N. H. Kim, and F. Febrianto, “Effects of compression ratio on physical and mechanical properties of bamboo oriented strand board,” IOP Conf. Ser. Mater. Sci. Eng., vol. 935, no. 1, pp. 0–2, 2020, doi: 10.1088/1757-899X/935/1/012063.
17. Y. Zhang, Y. Yu, and W. Yu, “Effect of thermal treatment on the physical and mechanical properties of phyllostachys pubescen bamboo,” Eur. J. Wood Wood Prod., vol. 71, pp. 61–67, 2012.
18. Y. Li et al., “Quasi-static and dynamic nanoindentation to determine the influence of thermal treatment on the mechanical properties of bamboo cell walls,” Holzforschung, vol. 69, no. 7, pp. 909–914, 2015, doi: 10.1515/hf-2014-0112.
19. L. Qin, “Effect of Thermo-Treatment On Physical, Mechanical Properties and Durability of Reconstituted Bamboo Lumber: Ph. D. dissertation, Chinese,” Chinese Academy of Forestry, 2010.
20. M. Nishida, T. Tanaka, T. Miki, T. Ito, and K. Kanayama, “Multi-scale instrumental analyses for structural changes in steam-treated bamboo using a combination of several solid-state NMR methods,” Ind. Crops Prod., vol. 103, pp. 89–98, Sep. 2017, doi: 10.1016/j.indcrop.2017.03.041.
21. H. Yun, K. Li, D. Tu, and C. Hu, “Effect of heat treatment on bamboo fiber morphology crystallinity and mechanical properties,” Wood Res., vol. 61, pp. 227–234, Jan. 2016.
22. O. Bobleter and H. Binder, “Dynamic hydrothermal degradation of wood,” Holzforschung, vol. 34, no. 2, pp. 48–51, doi: 10.1515/hfsg.1980.34.2.48.
23. J. Bourgois and R. Guyonnet, “Characterization and analysis of torrefied wood,” Wood Sci. Technol., vol. 22, no. 2, pp. 143–155, 1988, doi: 10.1007/BF00355850.
24. F. Meng, Y. Yu, Z. Zhang, W. Yu, and J. Gao, “Surface chemical composition analysis of heat-treated bamboo,” Appl. Surf. Sci., vol. 371, pp. 383–390, 2016, doi: https://doi.org/10.1016/j.apsusc.2016.03.015.
25. A. Burmester, “Effect of heat-pressure-treatments of semi-dry wood on its dimensional stability,” 1973.
26. K. Srivivasa and K. Pandey, “Effect of Heat Treatment on Color Changes, Dimensional Stability, and Mechanical Properties of Wood,” J. Wood Chem. Technol. - J WOOD CHEM TECHNOLOG, vol. 32, pp. 304–316, Oct. 2012, doi: 10.1080/02773813.2012.674170.
27. P. H. G. De Cademartori, M. D. Mattos, D. A. Gatto, and S. Maria, “Colour Responses of Two Fast-growing Hardwoods to Two-step Steam-heat Treatments,” Mater. Res., vol. 17, no. 2, pp. 487–493, 2014.
28. H. Pelit, “The effect of different wood varnishes on surface color properties of heat treated wood materials,” Istanbul Universitesi Orman Fakültesi Derg., vol. 67, p. 1. Jul. 2017. doi: 10.17099/jifiu.300010.
29. B. Sundqvist, “Colour Changes and Acid Formation in Wood During Heating,” Lulea University, 2004.
30. X. Huang, D. Kocaefe, Y. Kocaefe, Y. Boluk, and A. Pichette, “A spectrocolorimetric and chemical study on color modification of heat-treated wood during artificial weathering,” Appl. Surf. Sci., vol. 258, no. 14, pp. 5360–5369, 2012, doi: https://doi.org/10.1016/j.apsusc.2012.02.005.
31. Y. Sun, Y. Zhang, Y. Huang, X. Wei, and W. Yu, “Influence of board density on the physical and mechanical properties of bamboo oriented strand lumber,” Forests, vol. 11, no. 5, pp. 1–12, 2020, doi: 10.3390/F11050567.