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DEVELOPMENT OF GREEN COMPOSITES USING DECOMPOSED BAMBOO POWDER

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ABSTRACT

The purpose of this study is to investigate the mechanical properties of green composites made from lignin and cellulose extracted from bamboo. The raw material, consisting of bamboo powder and pure water were put into the reactor and sealed tightly. To decompose the raw material, the temperature of reactor was elevated by the ring furnace to a specified level and maintained for 10 min. For investigate the ability of the decomposed bamboo powder to be molded without the use of a binder, the compression molding technique was employed. The mechanical characteristics of fabricated composites were measured under four point bending load. The Young's modulus of fabricated composites was also measured by using dynamic hardness tester. The change in amount of holocellulose, α-cellulose, and lignin during molding was quantitatively evaluated by using the milled composite. Test results showed that the bending strength was increased with increase of temperature of molding. Fracture surface observation revealed that when molding temperature was high, fine surface was appeared at fractured surface, while rough surface was appeared when molding temperature was low. These results suggested that the thermoset ability of lignin was enhanced when molding temperature was high. By enhancing the thermoset ability of lignin, bending characteristics of composites was improved. Quantitative determination results showed that when molding temperature was higher than decomposed temperature of bamboo powder, the change in composition was appeared. Therefore, excessively high molding temperature is not effective because of the α-cellulose might be decompose.

KEYWORDS: Green composites, cellulose, lignin, binder-less, bending characteristics

INTRODUCTION

Biomass resources are gaining increasing attention because of global environmental problems such as the exhaustion of petroleum supplies or need for reduction of CO_2 emissions. From the viewpoint of material development, many kinds of natural plant biomass resources such as jute [1-2] or kenaf [3–4] have been investigated to fabricate eco-friendly composites materials called "green composites" [5–6]. Among the plant biomass resources, bamboo has received increased interest as an environmentally sustainable resource for material development because of its fast growth rate and robust mechanical characteristics [7–10]. Despite the fact that bamboo consists of cellulose, hemi-cellulose, and lignin, only cellulose was of interest in the reinforcement of composites in which the matrix was made from other polymers [11–15]. However, in spite of being a complex bio-based polymer like phenol, lignin has not been given much attention in the development of eco-friendly materials. Therefore, in order to develop new eco-friendly materials, the use of lignin as an alternative to other polymers is considered to be effective.

The purpose of this study is to propose an effective method of fabricating green composites in which the matrix and reinforcement are composed of lignin and cellulose, respectively. In this study, lignin and cellulose were extracted from bamboo by superheated vapor treatment [16] and were compression-molded to obtain binder-less green composites. The mechanical properties of the resulting composites were characterized by micro hardness and bending tests, respectively.

MATERIALS AND METHODS

Raw material

Dried bamboo powder was obtained from Moso bamboo trees grown naturally in Okayama, Japan, with an average plant age of around four years. Figure 1 shows an optical photomicrograph of the

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bamboo powder. The length (longest dimension) of the bamboo powder grains was measured for 100 randomly selected samples with an optical microscope. The average length of long side of the bamboo powder grains was about 0.8 mm. The amount of hemi-cellulose, a-cellulose, and lignin contained in the raw bamboo powder is described in Table 1.

Decomposition of bamboo powder

Figure 2 shows the experimental apparatus used in this study. The experimental apparatus consisted of a subcritical reactor (OM-Labotec: MM-200) and a ring furnace. The raw material, consisting of 15.0 g of bamboo powder and 150 cc of pure water, were put into the reactor, which was sealed tightly. To decompose the raw material, the temperature was elevated by the ring furnace to a specified level Td at a constant heating rate of 10°C/min, and was maintained for 10 min. During the reaction, the pressure in the reactor rose to the saturated vapor pressure of water at the selected temperature. After decomposition, the reactor was cooled to room temperature at a constant cooling rate of 50°C/min. A black solid residue and a weakly acidic liquid residue with a pH of around 3.5 were obtained, and these were separated by filtration. After filtration, the black solid residue was dried in the oven at 80°C.

Compression molding

Green composites made from decomposed bamboo were fabricated by using the compression-molding technique. To investigate the effects of molding conditions on mechanical characteristics of molded parts, the molding temperature T_m was varied. 7.0 g of the decomposed solid residue, in which the temperature of decomposition was T_d, and 20 cc of ethanol were put into a 60×60 mm2 rectangular mold and heat pressed under a nominal pressure of 30 MPa for 30 min. The fabricated composites were cut by a diamond cutter to prepare a 60×10×2.5 mm^3 coupon-type specimen.

Four-point bending test

A four-point bending test was conducted according to JIS K 7074 by using a universal testing machine, model SHIMADZU EZ-L, shown in Figure 3. The crosshead speed, upper span length, and lower span length of the bending test were set at 5 mm/min, 17 mm, and 51 mm, respectively. Bending strain and stress were simply calculated by following equations:

$$\sigma_B = \frac{FL}{wt^2} \quad (1)$$

$$\varepsilon_B = \frac{4.7\delta t}{L^2} \quad (2)$$

Here, F, δ, L, w, and t denote the applied bending load, deflection, span, width, and thickness of the specimen, respectively. At least five specimens were tested to investigate bending properties.

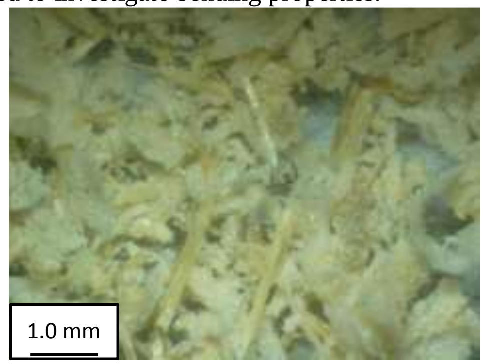

Figure 1. Optical microscope image of raw bamboo powder.

Table 1. Compositions of raw bamboo powder.

Hemicellulose	α-cellulose	Lignin	Other
28.3 %	45.5 %	21.7 %	4.5 %

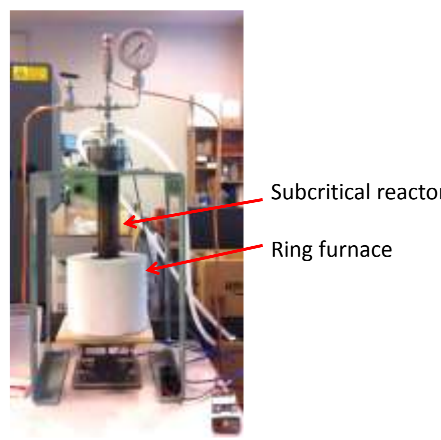

Figure 2. Experimental apparatus.

Figure 3. Four-point bending test apparatus.

Figure 4. Dynamic hardness tester.

Micro hardness test

The mechanical properties of composites were characterized using a dynamic hardness tester (SHIMADZU:DUH-200) shown in Figure 4. The indentation force was applied to the molded products by a diamond indenter until the reaction force increased to 9.8 mN, after which a constant force speed of 1.42 mN/s was maintained. The maximum load was held for 5 s and then released to zero at 1.42 mN/s force speed in the opposite direction. During testing, the stroke of the indenter and applied force were measured. By analyzing the applied force-stroke curve during unloading, the Young's modulus of the molded products was characterized. The Young's modulus at the surface of the molded products was measured at 16 or more different points and then averaged.

Quantitative determination

For a quantitative evaluation of the composites, three types of determinations, holocellulose, α-cellulose, and lignin determination, were performed using milled composites. First, the milled composites were de-lignified with a NaCl solution to determine holocellulose content by measuring the weight change during delignification. Then, hemicellulose was removed from holocellulose using a 1 mol/L NaOH solution, and α-cellulose content was determined by measuring the weight change owing to hemicellulose removal. Finally, lignin content of the milled composites was determined by subtracting the hemicellulose and α-cellulose content from the weight of the milled composites.

RESULTS AND DISCUSSION

Mehanical characteristics of composites

Figures 5 and 6 show the typical bending stress-bending strain (σ_b-ε_b) diagrams of composites in which decomposition temperature T_d was constrained to 180 or 220 °C, respectively. Test results showed that the bending stress increased almost linearly with the increase of bending strain. When the bending stress reached the bending strength, the bending stress rapidly decreased. This result suggests that the failure morphology of composites is similar to brittle

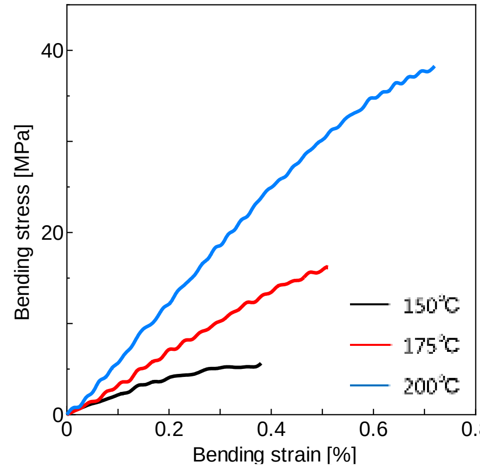

Figure 5. Typical bending stress-bending strain diagrams of composites in which decomposition temperature was T_d=180°C.

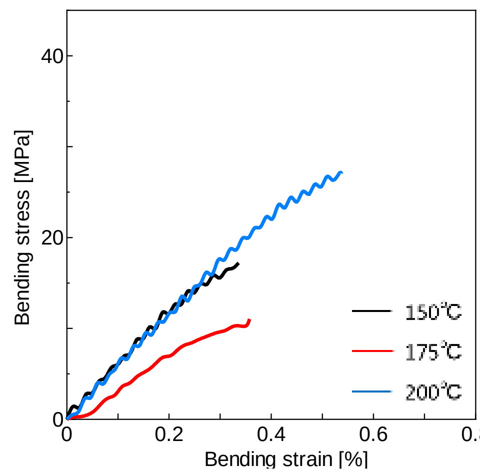

Figure 6. Typical bending stress-bending strain diagrams of composites in which decomposition temperature was T_d=180°C.

fracture. Figures 7 and 8 also show the averaged bending strength and flexural modulus with respect to the decomposed temperature T_d of bamboo powder at various molding temperatures T_m. Test results showed that the bending strength increased with increasing bamboo powder decomposition temperature when the molding temperature was 150°C. When the molding temperature was 175°C, the bending strength was increased until the temperature of bamboo powder decomposition reached 200°C and then decreased. When the molding temperature was 200°C, the bending strength increased until the bamboo powder decomposition temperature reached 180°C and then decreased slightly. For flexural modulus, almost the same tendencies observed for bending strength appeared. Figure 9 shows the Young's modulus of composites with respect to the decomposition temperature T_d of bamboo powder at various molding temperatures T_m. By using bamboo powder which decomposed at temperatures above 180°C, the Young's modulus of composites showed almost the same level even if the molding temperature was different. Figure 10 shows the SEM observation of the fractured surface of composites. When molding temperature was high, a fine surface appeared at the fractured surface, while a rough surface appeared when the molding temperature was low. These results suggested that the thermoset ability of lignin was enhanced when molding temperature was high. By enhancing the thermoset ability of lignin, the bending characteristics of composites were improved.

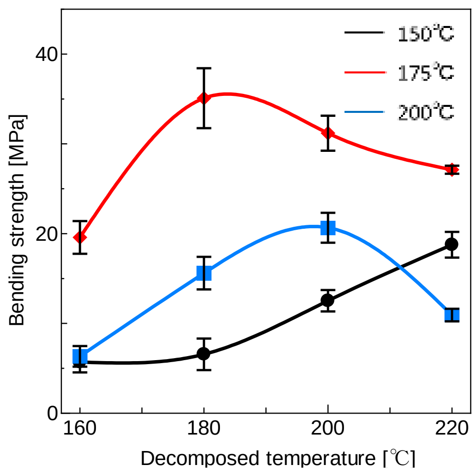

Figure 7. Average bending strength with respect to decomposition temperature T_d.

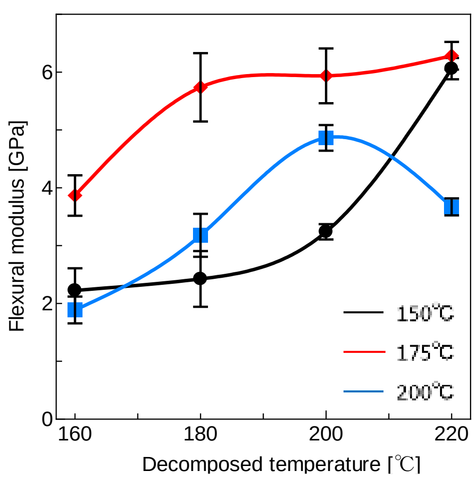

Figure8. Average flexural modulus with respect to decomposition temperature T_d.

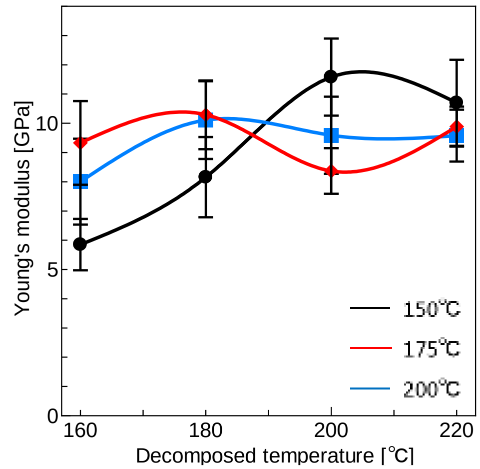

Figure 9. Young's modulus of composites with respect to decomposition temperature T_d.

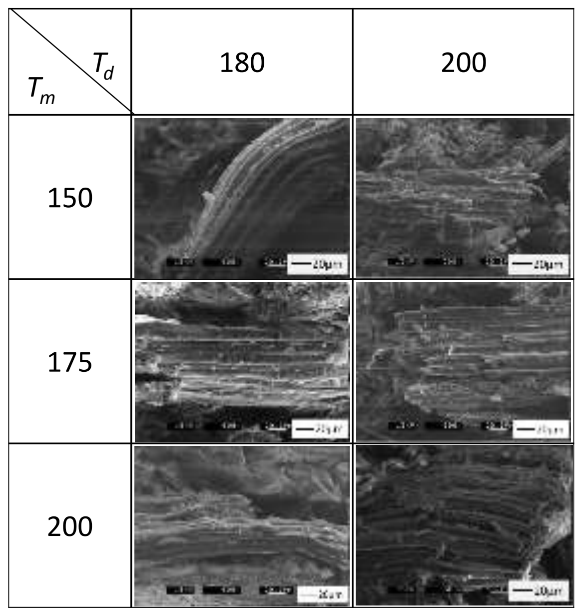

Figure 10. SEM observation of the fractured surfaces of composites.

Change in composition during molding

Figures 11 and 12 show the amount of holocellulose, α-cellulose, and lignin contained in composites made from bamboo powder decomposed at 180°C and 220°C, respectively. Here, for comparison, the composition of bamboo powder without molding is also indicated. Test results showed that when the molding temperature T_m was lower than the decomposition temperature T_d, the composition of the resulting composites was almost same as that of bamboo powder without molding. However, when the molding temperature T_m was higher than the

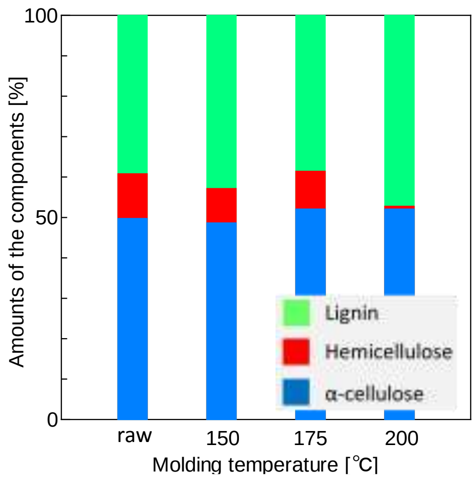

Figure 11. Composition of composites made from bamboo powder decomposed at 180°C.

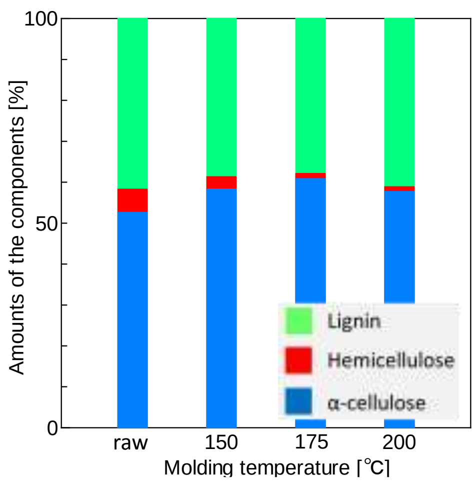

Figure 12. Composition of composites made from bamboo powder decomposed at 220°C.

decomposition temperature T_d, a change in composition appeared. These results suggest that decomposition of bamboo powder occurred during molding. According to our previous research, the decomposition of α-cellulose begins at 220°C and is almost complete at about 250 to 260°C. Therefore, excessively high molding temperature is not effective because α-cellulose present might decompose.

CONCLUSIONS

In this study, the mechanical characteristics of green composites made from decomposed bamboo powder were investigated, and the following conclusions were drawn:

1. The thermoset ability of lignin was enhanced when the molding temperature was high.
2. By enhancing the thermoset ability of lignin, the bending characteristics of composites were improved.
3. Excessively high molding temperature is not effective because α-cellulose might decompose.

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