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Research Article

Effect of Heating at Oven-Dry State on Steam Treated Bamboo Powder Thermal Fluidity

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In hot molding processes of woody material, it is important to understand the effect of oven-dry heating on the property of woody biomass material, such as thermal fluidity. In this study, thermal flow tests of untreated and steam treated bamboo powder were conducted to investigate the effects of heating at an oven-dry state on thermal fluidity. The test temperature was set to 200°C. Before the thermal flow test, powder was dry-heated in a capillary rheometer at 200°C with a variable heating time. Thermogravimetry was conducted to understand the thermal changes of the powder during an increasing temperature and constant temperature. Fluidity of untreated powder was improved with a short dry-heating but decreased with a long dry-heating. In contrast, steam treated powder fluidity was high compared to untreated one, but its fluidity did not improve from dry-heating. From these thermogravimetry results, the chemical changes associated with component volatilization relate with the thermal fluidity. Therefore, the decrease in fluidity from dry-heating occurred because fluidity related components escape from the powder through volatilization.

1. Introduction

Effective use of woody biomass resources is environmentally friendly because woody biomass resources are carbon neutral and sustainable. However, applications for woody biomass materials are limited because of some inherent practicality issues. One issue is material workability. Woody biomass is generally processed through machining, bending, or compacting. However, processing complex shapes is difficult, and some problems, such as limited extraction rates and productivity, exist with these methods.

In order to resolve these problems, various research studies on woody material processing methods have been conducted. For example, wood plastic composites (WPC), which are made by mixing woody materials and plastics, can be processed with general plastic processing methods such as extrusion or injection molding [1–3]. Methods where petroleum-derived materials were not used have also been developed. It was reported that molding of only woody materials containing water was possible at 170–180°C because woody materials have thermal fluidity and self-adhesiveness due to the hydrolysis of the material's chemical components [4–6]. In addition, it was reported that thermal fluidity and self-adhesiveness could be improved by steam treatment [7–10]. It is considered that these woody properties changes occur due to water soluble components increasing with the hydrolysis of the materials chemical components, such as hemicellulose, during steam treatment [11–13]. Injection molding is possible without water by using steam treated woody powder, and strength of injection molded product came up to that of polypropylene [14, 15]. This way, thermal fluidity of woody material can be controlled for the improvement of moldability by heating at the wet state, such as steaming.

Woody material properties are affected by heating not only during the wet state, such as steaming, but also during the dry state. For example, research about woody material dry-heating has shown that dynamic viscoelastic properties of dry wood change in relation to its heating temperature or time [16]. In addition, thermal changes to the physical

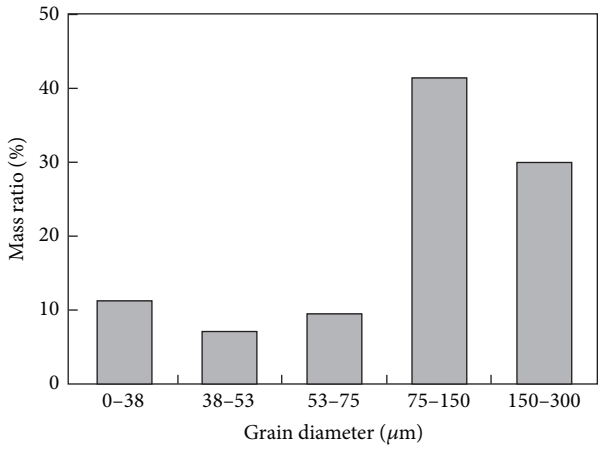


Figure 1: Particle size distribution of bamboo powder.

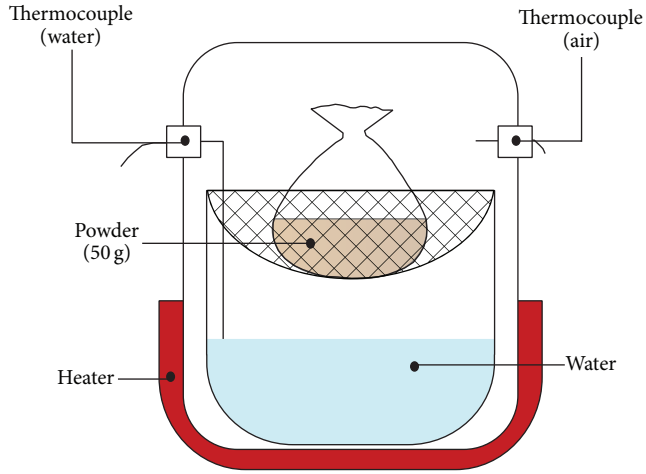


Figure 2: Schematic diagram of steam treatment.

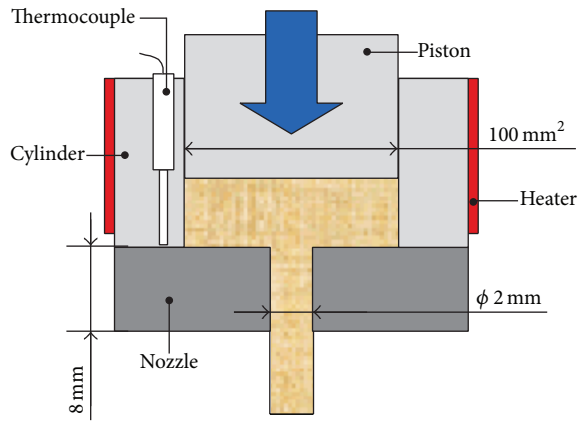


Figure 3: Schematic diagram of the capillary rheometer.

property, such as thermal softening, in the dry state are different from those in the wet state [10, 17]. Accordingly, it is possible that the thermal flowability of woody material is affected by dry-heating. However, the effect of dry-heating on the thermal fluidity of woody material is not fully understood. For actual hot molding processes for woody material, it is important to understand these effects.

In this study, a thermal flow test for woody materials dry-heated for different treating time was conducted using a capillary rheometer. Bamboo powder, which was untreated or steam treated, was studied because an effective use of the timber from bamboo thinning is desired in Japan [18]. In addition, it was reported that bamboo fluidity is high compared to other woody materials [19]. This reason is the fact that hemicellulose of bamboo is mainly xylan [20] and xylan having acetyl group produces acid which catalyzes hydrosis [12]. Thermogravimetry for bamboo powder was conducted in order to investigate the thermal change effects during dry-heating. From these results, the relationship between thermal fluidity and chemical changes in bamboo powder during dry-heating is discussed.

2. Materials and Methods

2.1. Preparation of the Materials. Moso bamboo powder was used. To obtain the powder, a stem was shaved and shavings were then milled in a pin mill. The powder passed through a ϕ300 μm screen during the milling stage. Figure 1 shows the powder particle size distribution, which was predominately in the range of 75–300 μm.

Figure 2 shows a schematic diagram of the bamboo powder steam treatment. The powder was treated with saturated water vapor at a high temperature by heating a small pressure vessel containing the powder and water. First, the vessel was heated to a target temperature of T_s (°C). Next, the temperature T_s was held for a predefined time t_s (min). The vessel was then cooled to 100°C. The powder was tapped off from the vessel and air-dried at room temperature. Air-dried powder was dried additionally in an oven at constant 105°C to achieve an oven-dry state before the thermal flow test.

2.2. Thermal Flow Test. A capillary rheometer (CFT-500D, Shimadzu) was used to dry-heat the powder and for the thermal flow test. Figure 3 shows a schematic diagram of the capillary rheometer. First, the capillary rheometer was heated to a test temperature of 200°C because bamboo fluidity is activated at 200°C [4]. Powder with a mass of 1.5 g was placed in the cylinder followed by a piston. The powder was then heated for a predefined time t_h (min) in the cylinder. The piston was compressed at 49 MPa and material was extruded from the nozzle. The piston movement during the material extrusion was evaluated.

2.3. Thermogravimetry. Thermogravimetry was conducted with a thermogravimetry/differential thermal analyzer (TG/DTA 6200, Seiko Instruments Inc.). The air-dried bamboo powder was placed in an aluminum pan, and an empty aluminum pan was used as a reference. Figure 4 shows the temperature program. First, drying at 105°C for 30 min with a dry nitrogen gas purge was performed to ensure that moisture state of the powder was oven-dry in the device. The temperature was raised to 200°C at a constant rate of 10°C/min and held for 60 min. Changes in mass loss rate

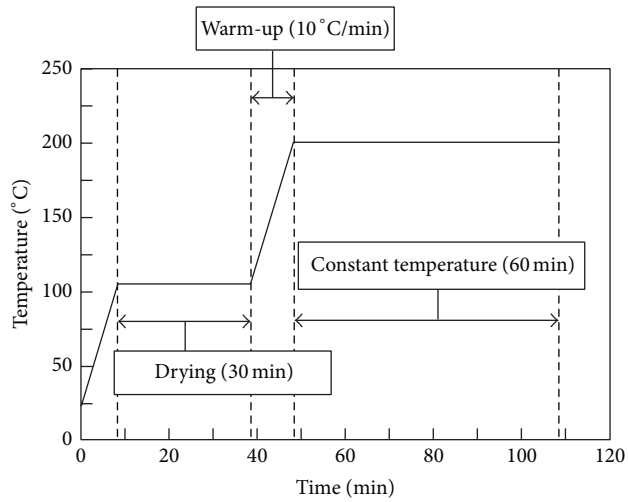


Figure 4: Thermogravimetry temperature schedule.

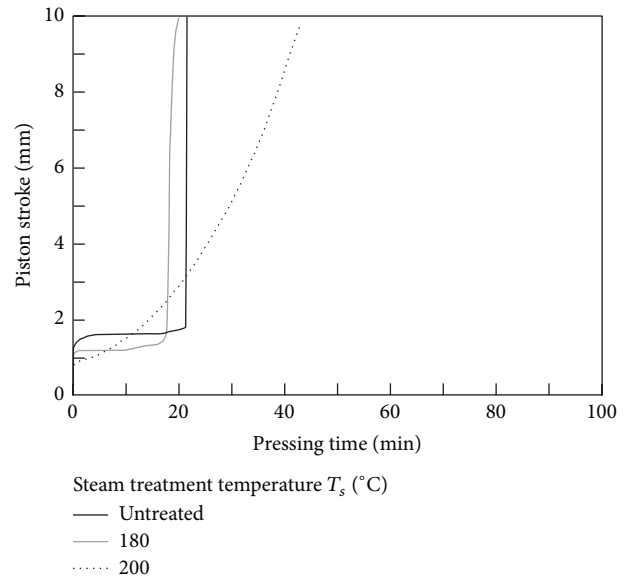


Figure 5: Effect of steam treatment temperature T_s on the thermal flow curve. (t_s = 20 min and t_h = 5 min).

(%/min) based on the oven-dry mass were evaluated during the warm-up period and the constant temperature period.

3. Results and Discussion

3.1. Thermal Flow Curve of Untreated and Steam Treated Bamboo Powder. Flow behavior of untreated and steam treated bamboo powder was investigated. Figure 5 shows the relationship between press time and piston stroke motion when the steam temperature T_s was changed. The steam treatment time t_s was set at 20 min and the heating time t_h was set at 5 min. It was considered that the powder temperature achieved 200°C with a 5 min heating interval because we confirmed that the temperature of the powder near the surface was 200°C after 3-4 min.

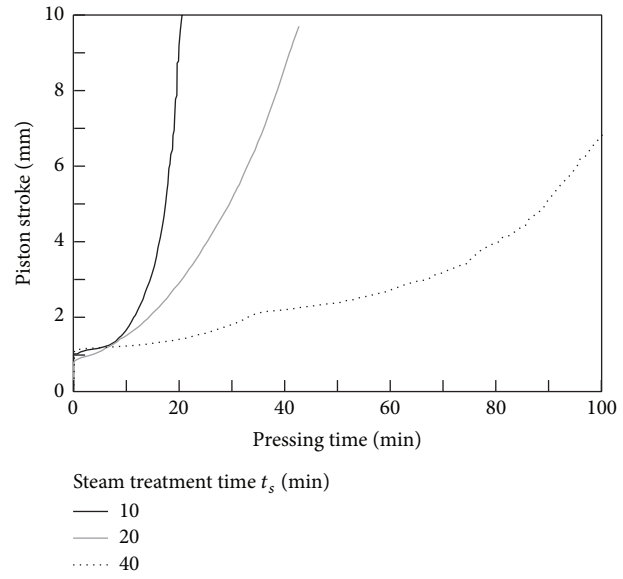


Figure 6: Effect of steam treatment time t_h on the thermal flow curve. (T_s = 200°C and t_h = 5 min).

As shown in Figure 5, the untreated and T_s = 180°C powder did not flow immediately but rapidly increased in rate when the pressing time reached about 20 min. It was determined that the fluidity was provided by thermal decomposition from heat caused by the piston pressure. Gas is exhausted during thermal decomposition of wood biomass material [21], but it is difficult for the gas to escape from the cylinder until the flow starts because the powder is in a high pressure state. Therefore, the material flowed drastically when the gas could escape once the flow started. In contrast, the T_s = 200°C powder flowed immediately once pressed but at a slower flow rate. Accordingly, it was determined that powder fluidity is simple to increase because the powder has flow components produced by the preliminary steam treatment. In addition, a rapid flow rate increase could not have occurred because the gas was able to escape gradually with material flow.

Figure 6 shows the relationship between pressing time and piston stroke motion when the steaming time t_s was changed. In this experimental series, T_s was set at 200°C because flow behavior changed largely with a steam treatment of 200°C, as shown in Figure 5. The flow rate slows with an increase in t_s. This result suggests that a long steam treatment has a negative effect on thermal fluidity.

3.2. Effects of Dry-Heating Time on the Bamboo Powder Thermal Flow Curve. Figure 7 shows the thermal flow curve changes for each powder by dry-heating time t_h. The range of t_h tested was from 1 to 20 min. For untreated powder, flow started sooner when t_h increased from 1 to 5 min, but the start was delayed with a further increase in t_h over 5 min as shown in Figure 7(a). In contrast, for steam treated powder, the flow began soonest when t_h was 1 min, while the flow start was delayed with an increase in t_h as shown in Figures 7(b)–7(e). In particular, powder steam treated at 200°C for 10 and 20 min had no flow when t_h was 20 min and that for 40 min had no

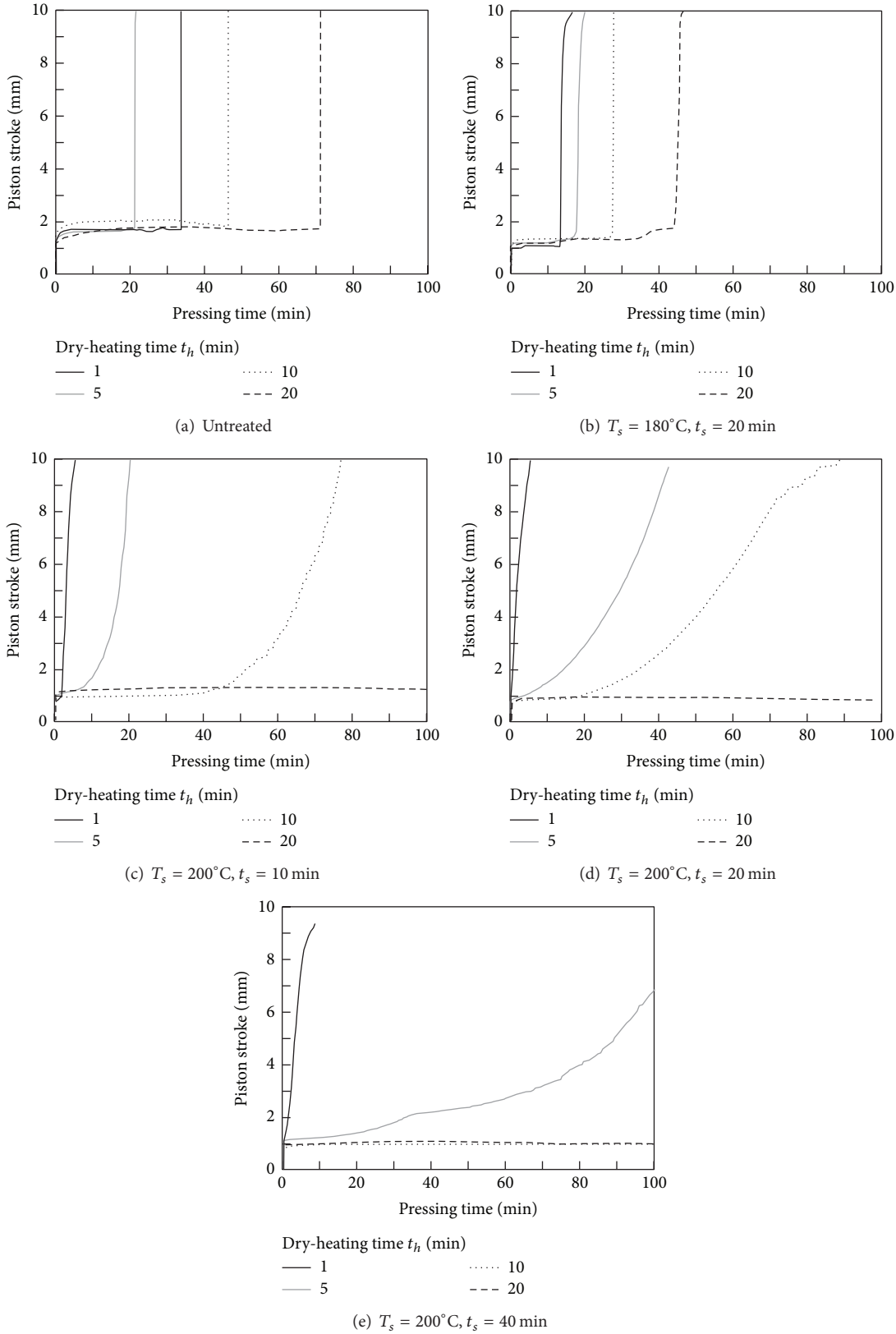
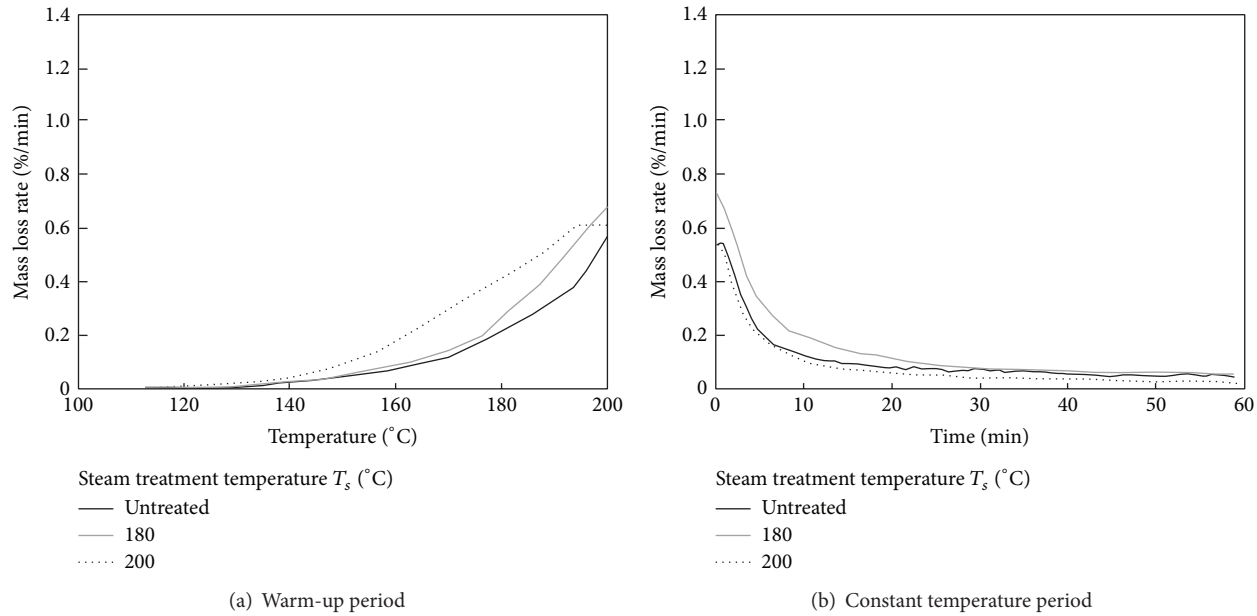


Figure 7: Effect of dry-heating time t_h on the thermal flow curve when the steam treatment temperature is changed.



(a) Warm-up period (b) Constant temperature period

Figure 8: Mass change rates of bamboo powder untreated and steam treated with a variable steam treatment temperature T_s. (t_s = 20 min).

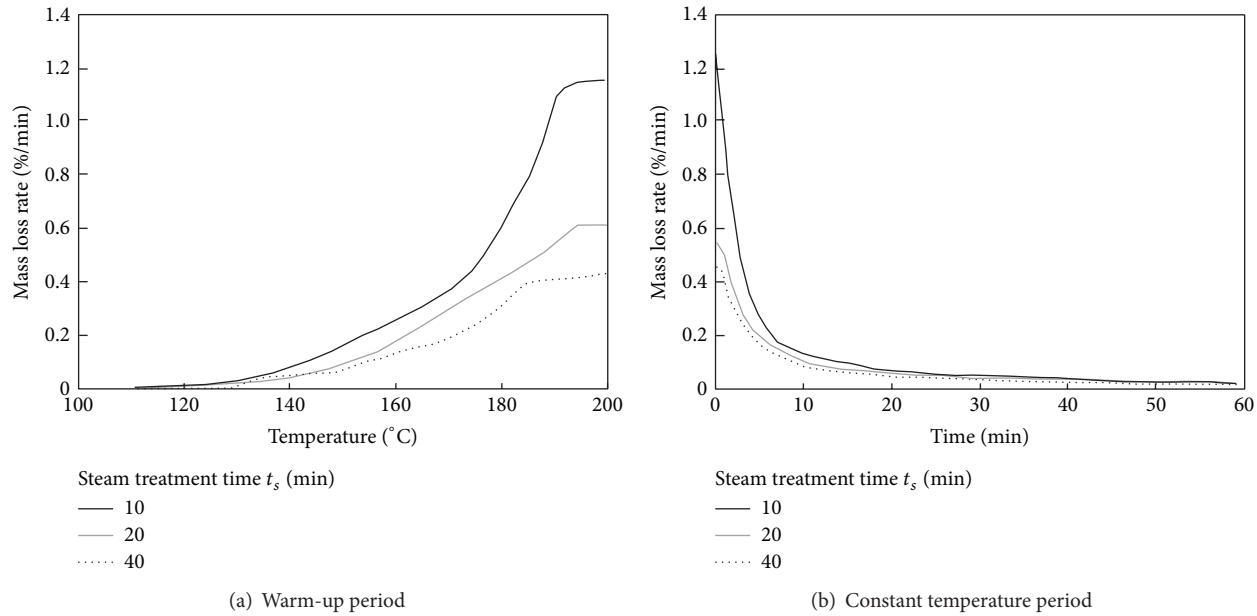
flow when t_h was over 10 min. The powder temperature did not reach 200°C yet when t_h was only 1 min. Accordingly, powder fluidity at T_s = 200°C, which flowed immediately for t_h = 1 min, could have become activated at a temperature under 200°C. These results suggest that untreated powder can be activated by heating under ordinary pressure for a short time. For steam treated powder, it was confirmed that dry-heating has a negative effect on thermal fluidity regardless of steam treatment temperature or time.

3.3. Bamboo Powder Mass Loss during Warm-Up and Constant Temperature Periods. Figure 8 shows the mass loss rate for bamboo powder during the temperature increase to 200°C and when being held constant at 200°C for a variable steam treatment temperature T_s. The steam treatment time t_s of steam treated powder was 20 min. The mass loss rate shows the chemical change intensity because component volatilization occurs from woody material thermal decomposition [21]. As shown in Figure 8(a), mass loss starts when the temperature reaches about 140°C. The amount of mass lost for steam treated powder was higher than that of untreated powder. In particular, the mass loss rate for T_s = 180°C surpassed that of untreated powder when the temperature reached about 160°C, where it continued to increase until 200°C. This result suggests that chemical changes in powder are activated by a 180°C steam treatment. For T_s = 200°C, the mass loss rate surpassed the untreated powder at about 145°C, and it continued to increase until 190–195°C. This suggests that the chemical changes become active at a lower temperature and the intensity relaxes at 190–200°C. With constant temperature, the mass loss rate of each powder decreases with time and ceases after about 30 min as shown in Figure 8(b). The mass loss rate for T_s = 180°C powder is comparatively high until 30 min. This result indicates that chemical changes continued for 30 min after increasing the temperature to 200°C, and T_s = 180°C powder was the most active at constant temperature.

Figure 9 shows the bamboo powder mass loss rate for T_s = 200°C during the temperature increase to 200°C and when being held constant at 200°C for a variable steam treatment time t_s. In the warm-up period, as shown in Figure 9(a), the amount of mass lost decreases with increasing t_s. The mass loss rates for t_s = 10 and 20 min become constant at 190–195°C, while t_s = 40 min becomes constant at about 185°C. This result suggests that the chemical change intensity decreases with an increase in t_s. For constant steam treatment temperature, as shown in Figure 9(b), the mass loss rate for each powder decreases and becomes constant at 30 min.

It was reported that water-soluble components are produced from hemicellulose by steam treatment and some of these components change to volatile components, such as furfural, from high temperature at a prolonged exposure time [11]. Thus, an increase in mass loss from steam treatment could have been due to an increase in volatile water-soluble components. For long t_s, it is possible that powder loses its water-soluble components through dissolving with water or volatilization during steam treatment. For this reason, it was determined that the amount of mass loss decreases with an increase in t_s.

Relationship between thermal fluidity and mass loss during warm-up and constant temperature periods is discussed. As shown in Figures 5 and 8, T_s = 200°C powder flow starts early compared to the other steam treatment temperatures and mass change begins at a lower rheometer temperature. It was reported that chemical changes associated with component volatilization relate with woody material thermal fluidity [13, 19]. It was determined that T_s = 200°C powder fluidity was adequately activated during the 5 min dry-heating. For



(a) Warm-up period

(b) Constant temperature period

Figure 9: Mass loss rate for steam treated bamboo powder with a variable steam treatment time t_s. (T_s = 200°C).

untreated and steam treated powder at 180°C, fluidity was not adequately activated after 5 min of dry-heating. Thus, flow started after about 20 min of pressing time and T_s = 180°C powder flow start began earlier than the untreated powder because chemical changes at T_s = 180°C were more active with a constant rheometer temperature. The thermal fluidity changes from t_s, as shown in Figure 6, were also caused by the intensity differences in the chemical changes associated with the component volatilization. T_s = 200°C powder flow started immediately after pressing because the chemical changes caused by the 5 min dry-heating activated the fluidity, but the chemical change intensity decreases with an increase in t_s as shown in Figure 9(a). Therefore, the flow rate decreases with an increase in t_s.

Next, we discuss the effect of dry-heating time on thermal fluidity in an oven-dry state and the chemical changes during dry-heating. For untreated powder as shown in Figure 7(a), it was determined that the flow started early when t_h increased from 1 to 5 min because the fluidity was activated by a 5 min dry-heating. However, the flow started later with an increase in t_h from 5 to 20 min. It is possible that this is because of component volatilization. It was considered that volatile components have a large effect on thermal fluidity, but the volatile components escape from the powder during dry-heating because dry-heating was conducted under atmospheric pressure. As shown in Figure 8(b), it can be seen that volatilization occurred for 30 min in each powder. Accordingly, the powder fluidity decreases with an increase in t_h because of volatile components escaping from the powder during dry-heating. By contrast, component volatilization could have been inhibited during pressing due to the powder's high pressure state. Thus, fluidity was activated through pressing and heating from occurring chemical changes, while the volatile components remain in the powder.

For steam treated powder, the flow showed a tendency to start later at a slower rate as t_h changed from 1 to 20 min as shown in Figures 7(b)–7(e). The volatilization amount was larger than that of untreated powder until the temperature reached 200°C, as shown in Figures 8(a) and 9(a). Therefore, the fluidity of steam treated powder was activated with ease until the temperature reached 200°C, but the negative effect volatilization has on fluidity was greater than the heating effect for fluidity activation when the temperature reached 200°C.

4. Conclusions

In this study, thermal flow tests of untreated and steam treated bamboo powder were performed using a capillary rheometer to investigate the effect of heating at an oven-dry state on thermal fluidity. The test temperature and dry heating temperature were set at 200°C. Untreated and steam treated bamboo powder at 180°C started to flow several minutes after the start of pressing. Steam treated powder at 200°C flowed immediately after pressing. When the powder dry-heating time increased in an oven-dry state before pressing, the untreated powder flow began earlier with an increase in dry-heating time from 1 to 5 min but started later with an increase in dry-heating time from 5 to 20 min. The flow of the steam treated powder started later with an increase in dry-heating time from 1 to 20 min. From the thermogravimetry results during the increasing and constant temperature periods, it was confirmed that chemical changes associated with component volatilization relate with thermal fluidity. For untreated powder, fluidity is improved from chemical changes during a short dry-heating, but fluidity decreases because the powder loses its fluidity related components from volatilization with a long dry-heating. For steam treated powder, fluidity was

high when the dry-heating was short because of the chemical change intensity associated with volatilization. It was easy to slow fluidity with dry-heating because the powder readily loses its fluidity related components due to the large amount of volatilization.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgment

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