

Structural and Compositional Changes of Bamboo Fibers During Super-Heated Steam Treatment Improved Mechanical Properties of Polypropylene/Bamboo Biocomposite

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Abstract

Structural and compositional changes of bamboo fibers, which were prepared through super-heated steam (SHS) treatment at normal pressure and fine grinding, were investigated by infra-red spectroscopy, thermogravimetry, viscosity-average molecular weight (M_v) measurement, and X-ray fluorescence analysis. SHS-treated Moso bamboo resulted in preferential decomposition of hemicellulose without any decrease in the M_v of cellulose crystalline. The SHS-treated bamboo was easily ground to obtain various short bamboo fibers (sBF) suitable for conventional melt processing of composites with polypropylene. The sBF showed good melt-processability and contributed to the mechanical performance of the composites moldings, resulting in 290% increases in flexural modulus.

Keywords: Moso bamboo, superheated steam treatment, short bamboo fiber, chemical composition

1. Introduction

Bamboo is an abundant biomass and available in many countries. Worldwide, bamboo forest area totals 22 million hectares generating over 30 million tons of bamboo fiber per year [1]. Bamboo, with time to maturity of only six to eight months, can regenerate much more rapidly than normal wood, which takes about twenty times longer. Bamboo fiber (BF) is one of the important ligno-cellulosic fibers possessing low-density compared to glass and steel fibers, high tensile modulus, and low elongation at break [2]. Because of these useful physical and mechanical properties, it has attracted worldwide attention as a potential reinforcement for polymer composites. It is anticipated that bamboo fiber will also have low water absorption properties because of its intrinsic fibrovascular bundle structure. Moreover, the blended cellulosic fibers in matrix resins have a significant effect on the mechanical strength of composites. Much research is ongoing concerning its properties of mechanical strength [3] and fiber size [4]; into its use as filler and into the development of appropriate compatibilizers [5].

Since fossil oil is a limited resource, there is a need in the transportation sector to use lighter materials for manufacturing vehicles. This approach can help to reduce fuel consumption in transportation. Therefore, wood-plastic composites are being investigated as replacements for the metal frames and outside panels of vehicles. These light materials with excellent melt processability can be moulded into complex shapes and are ideal for replacing heavier traditional materials. Short fiber

reinforced composites are increasingly being used in many applications because they exhibit the various advantages of continuous fiber reinforced composites and can be processed without difficulty. Many of these short fiber reinforced composites are processed by injection molding [6] so the desired parts can be manufactured with high precision at high speed.

In order to prepare short bamboo fiber (sBF) effectively, physical and chemical treatments of bamboo have been conducted. Phuong and Gilbert [7], and Okubo et al. [8] obtained sBF (average length: L 168 μm and 125-210 μm , respectively) by a mechanical separating method. Tokoro et al. [9] prepared three kinds of sBF: short bundle, alkali-treated filament, and steam exploded filaments (average diameter: D 18.8-39.2 μm , L 215-1740 μm) using a cutting machine, in a 1.5N NaOH at 70 $^{\circ}\text{C}$, and over-heated steam at 175 $^{\circ}\text{C}$ and 0.7-0.8 MPa for each BF preparation. Chattopadhyay et al. [10] treated sBF with NaOH at 160 $^{\circ}\text{C}$ followed by grinding and sieving to obtain sBF (D 90-125 μm , L 1-6 mm). Although the mechanical pulverization is the simplest method, the abrasion of blade is intense due to the hard epidermis of bamboo including silicon. The steam explosion is a popular method to isolate BF, however, a pressure resistant reactor is required and thereby it is difficult to operate in a large scale. The chemical treatment is a desirable method to isolate more pure BF, however, a large amount of liquid waste that must be post-processed is by-produced. In comparison with the above methods, super-heated steam

(SHS) treatment is an effective method to prepare sBF, because SHS is a powerful and clean reagent for biomass transfer to organic materials at normal pressure.

Although the sBF were prepared by various methods as showing above, no report has been carried out into the compositional and structural changes in the ingredients of bamboo during such treatment or into how such changes might affect the properties of the resulting sBF/plastic composites. In this paper, changes in the chemical composition of bamboo and the molecular weight of cellulose during SHS treatment were investigated. Although the SHS treatment was applied to palm biomass to enhance saccharification [11], no report of the SHS treatment of bamboo has been published. Obtained sBF by the SHS treatment was applied to biomass/plastic composites, in which the sBF showed good melt-processability and contributed to the mechanical performance of the composite moldings.

2. Materials and Methods

2.1 Materials

Phyllostachys heterocycla f. pubescens (Moso bamboo) samples (diameter: 10-20 cm, height: 10-20 m) were collected at Yame city in Japan. Cellulose microcrystalline was purchased from Merck KGaA (Germany, Avicel® for thin-layer chromatography) and used to estimate the molecular weight change of cellulose. Sodium chlorite (NaClO₂), sulfuric acid (H₂SO₄), potassium hydroxide (KOH), 1M hydrochloric acid (1M HCl), lithium chloride (LiCl), and dimethylacetamide (DMAC) were obtained from Wako Pure Chemical Industries, Ltd. (Japan). Polypropylene (PP) [Japan Polypropylene Corporation, Japan, extrusion grade NOVATEC-PP FY6, density 0.90 g/cm³ (JIS K7112), melt flow index 2.5 g/10 min (JIS K7210)] was used in this study as a matrix.

2.2 Preparation of sBF

The Moso bamboos were cut to 40 cm in length, treated by superheated steam (SHS) at 190-250 °C for 60 min and at 210 °C for 180 min in 30 min intervals under thermostating within ±5 °C and a constant flow rate of 6 kg·h⁻¹ in a SHS oven model NHL-1 (Naomoto Corp., Japan; inner dimensions: W 590 × D 385 × H 555 mm³) with an internal fan for agitation of atmosphere. SHS treated bamboo was ground with a grinder, RUB Master SRM-15 (Sanken Engineering Co., Ltd., Japan) and a Jiyu mill type M-2 (Nara Machinery Co. Ltd., Japan), then sieved with a sieve shaker model MVS-1 (AS ONE Corp., Japan) using sieves of aperture sizes: 63, 150, and 250 μm. Three kinds of sBF were classified, i.e., 150-250 μm, 63-150 μm, and 0-63 μm in a weight ratio of 20:45:30, which had different aspect ratio (AR) values:

3.20, 9.94, and 2.85, respectively, and a residual larger sBF portion (>250 μm) in a weight ratio of 5. Moisture content of obtained sBF was measured on a moisture balance MOC-120H (Shimadzu Corporation, Japan) at 105 °C.

2.3 Characterization of SHS treated bamboo

2.3.1 Fourier transform infrared (FTIR) analysis

Fourier transform infrared (FTIR) spectra were recorded on a Perkin Elmer GX2000R infrared spectrophotometer (USA) in a range of 500–4000 cm⁻¹ at a resolution of 4 cm⁻¹. Reflection spectra of EFB samples were measured by the single-reflection ATR method on a Golden Gate Diamond attenuated total reflectance (ATR) (10500) module with a germanium crystal.

2.3.2 Thermal properties

Thermogravimetric (TG) and differential TG (DTG) measurements were conducted on a SEIKO Instruments Inc. EXSTAR 6200 TG system (Japan) in aluminum pans (5 mm in diameter) at a prescribed heating rates, ϕ , of 9 °C·min⁻¹ in a temperature range of 50 to 550 °C under a constant nitrogen flow (100 mL·min⁻¹) using about 5 mg of ground powder sample (water content: 4-5 wt%). A blank aluminum pan was used as a reference. The pyrolysis data were collected at regular intervals (about 20 times °C⁻¹) by an EXSTAR 6000 data platform, and recorded into an analytical computer system.

2.3.3 Organic components

The composition of main organic components: cellulose, hemicellulose, and lignin, was analyzed in the same way as Fahma's method [12]. To remove lignin, the ground sBF (1.000 g) was soaked in a 5 wt% sodium chlorite (NaClO₂) solution (100 mL) under an acidic condition (pH 4) using H₂SO₄, stirred at room temperature for 30 min and at 70 °C for 1 h, filtered on filter paper (No. 5B), washed with deionized water, dried in an oven at 70 °C overnight *in vacuo*, and then weighed. Hemicellulose was extracted by bleaching the sBF with a 6 wt% KOH solution at room temperature for 24 h under stirring. After the extraction, the solution was neutralized by a 1M HCl solution and the treated sBF was filtered on filter paper (No. 5B), washed with deionized water, dried in an oven at 70 °C overnight *in vacuo*, and then weighed. Moisture content of sBF was measured after each extraction process using the moisture balance.

2.3.4 Inorganic components

In order to analyze inorganic components in bamboo samples, X-ray fluorescence (XRF) analysis was carried out with a RIGAKU ZSX101e (Japan). Two kinds of bamboo were sampled:

the surface layer of the stem in a thickness range of < 1mm and the bamboo's inner layer. The bamboo sample and polystyrene as a matrix were ground to obtain a mixed powder. The mixed powder was then poured into an aluminum cup (diameter 1.1 cm, height 3 mm) and pressed at 25-30 MPa for 30 s to form a disk-shaped sample and XRF analysis was performed on this solidified disk. Measurements were repeated five times and averaged composition and standard deviation values were calculated for each element.

2.4.5 Molecular weight measurement of cellulose

Molecular weight of cellulose samples was measured by viscometry method as per a previous report [13,14]. The inside of the cellulose sample was replaced by DMAc in the following manner. Cellulose sample (5 g) was soaked in deionized water (100 mL) and stirred at room temperature overnight. The sample was filtered on filter paper (No. 5B), rinsed with acetone, and soaked in acetone (100 mL) overnight at room temperature under stirring, and then filtered on filter paper (No. 5B). The sample was soaked in DMAc (100 mL) and stirred at room temperature overnight. The sample was filtered on filter paper (No. 5B) and dried at 60 °C for 40 h *in vacuo*.

The LiCl/DMAc solution was prepared in the following manner. LiCl was dried at 120 °C overnight *in vacuo* and DMAc was treated with molecular sieves (4A) for 40 h. Dried LiCl (9 g) was dissolved in DMAc (91 mL) and the solution was stored in a desiccator under shading.

The cellulose sample was dissolved in the LiCl/DMAc solution at concentrations of 50, 75, and 100 mg/50 mL under stirring at room temperature for 24 h. The relative viscosities of the sample solutions were measured with an Ubbelohde viscometer (Shibata Scientific Technology Ltd., Japan, SU-8306-1C) at 30 °C in a thermostated water bath. The molecular weight of cellulose sample was estimated from its intrinsic viscosity by using Eq. 1:

$$[\eta]=kM^a \quad (\text{Eq. 1})$$

where, k and a are constants for cellulose using 0.128×10^{-3} mL/g and 1.19, respectively [14].

2.4.6 Melt-blending with polypropylene and flexural properties of composites

In order to evaluate the contribution of sBF to the mechanical properties of composites, three kinds of sBF, classified by grinding and sieving from SHS treated bamboo at 210 °C for 3 h, were melt-blended with polypropylene (PP) in a weight ratio of sBF:PP = 30:70 (wt/wt). The SHS treated bamboo at 210 °C for 3 h was selected due to the lowest temperature, at

which temperature almost hemicellulose degraded, while no cellulose crystalline degraded.

A twin-screw extruder IMC-160B (Imoto Machinery Co., Ltd., Japan, screw diameter 20 mm, L/D 25) equipped with an air vent was used for blending under temperature profiles of 80, 190, 190, and 190°C for four zones, respectively, from hopper to die with a screw rotational speed of 15 rpm. Extruded strands were cut to pellets with a pelletizer.

Injection molding of the pellets of sBF/PP composites was carried out with a simple injection molding machine IMC-18D1 (Imoto Machinery (Kyoto, Japan)) at 190 °C/12 MPa for 30 s, resulting in the preparation of specimens ($20 \times 5 \times 2$ mm³) for flexural strength tests. The flexural strength test was conducted on a compact tensile & compression tester IMC-18E0 (Imoto Machinery Co., Ltd., Japan) at a bending rate of 10 mm/min.

3. Results and Discussion

Among the many different kinds of natural fibers used in composite materials, bamboo is deemed to have one of the most favorable combinations of good mechanical properties. The specific strength and stiffness of bamboo fibers are comparable to those of glass fibers [15].

3.1 SHS treatment of bamboo

Bamboo comprises three main ingredients: cellulose (40-48%), lignin (11-27%), and hemicellulose (23-30%) [16,17,18]. Hemicellulose consists of various saccharides having many branches which are embedded in the cell walls of plants and bind to cellulose to form a network of cross-linked fibers. When the hemicellulose was removed selectively, cellulose fibers are more easily obtainable to apply as reinforcing fibers, e.g., for wood plastic composites.

It has been reported that hemicellulose is easily degraded and removed from the main stem [19]. Cellulose, however, is more stable, with lignin being the most stable [20] due to its aromatic ring network having various branch structures. This means that the hemicellulose is preferentially degradable under controlled heating conditions to liberate cellulose fibers. In order to decompose the hemicellulose preferentially, the Moso bamboo was treated by super heated steam (SHS) at 190-250 °C, resulting in the bamboo changing in color and physical toughness by turning dark brown and splintering easily in a lengthwise direction, respectively. Shao et al. explained that a major component of the brown-colored fraction of steam-exploded at 210 °C was estimated as lignin [18]. Thus, it is considered that the color change of bamboo after the SHS treatment is induced by lignin-derived degradation products.

3.2 Changes in organic ingredients

3.2.1 Thermal gravimetric analysis

Figure 1 shows TG and derivative TG (DTG) curves of SHS treated bamboo samples at 190-250 °C for 60 min. The TG profile shifted to a higher temperature range and higher residual weight with increase in the steam temperature (Figure 1). The DTG curves showed the two main pyrolysis steps of hemicellulose and cellulose in temperature ranges of 200-300 and 300-380 °C, respectively. It is assumed that degradation of the lignin occurs in a wide temperature range [21]. The DTG peak at 200-300 °C based on the degradation of hemicellulose, gradually diminished and disappeared after treatment at 240 °C for 60 min. Moreover, the DTG peak at 300-380 °C based on the degradation of cellulose shifted to a higher temperature region by 10 °C and more. When the SHS treatment was prolonged for up to 180 min at 210 °C, the DTG peak of hemicellulose degradation was diminished and disappeared with time similar to the changes in peaks with increasing temperature found in Figure 1. Similarly, the temperature range of cellulose degradation also shifted to a higher temperature range by 10 °C and more. These results indicate the preferential degradation of hemicellulose and suggest the presence of interactive mechanisms on the thermal degradation between hemicellulose and cellulose. By hydrothermal removal of the hemicellulose, cellulose may be liberated from the interaction, resulting in the apparent stabilization.

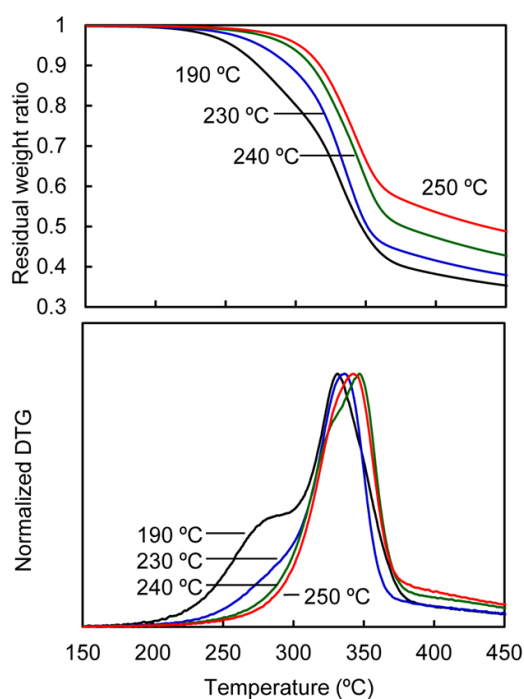


Figure 1 : TG and DTG curves of original and SHS treated Moso Bamboo. SHS treatment: at 190-250 °C for 60 min.

3.2.2 FT-IR analysis

Changes in chemical structure during the SHS treatment were analyzed with FT-IR. In Figure 2, FT-IR spectra of the SHS treated bamboo samples at 210 °C for 180 min in 30 min intervals with their difference spectra are shown. The FT-IR spectrum after 30 min treatment was employed as reference of the differential spectra in order to exclude influences of various physical and chemical changes occurring at lower temperatures until 210 °C. Remarkable decreases in peaks at 1730 and 1240 cm^{-1} corresponding to the specific absorption bands: $\nu\text{C}=\text{O}$ and $\nu\text{C}-\text{O}-\text{C}$ of carboxyl groups of the hemicellulose, were observed [17,22]. In the differential spectra, intensity of a characteristic absorption peak around 1390 cm^{-1} based on C-H bending of amorphous cellulose [23] was decreased with the SHS treatment, suggesting the degradation of amorphous cellulose. On the other hand, the specific absorption bands of lignin at 1614 and 1456 cm^{-1} ($\nu\text{C}=\text{C}$, aromatic skeletal) showed a relative increase [21], and the bands of cellulose remained without definite changes in intensity [22,24]. These results also indicate the preferential degradation of hemicellulose by SHS treatment. Similarly, the SHS treatment of bamboo at 190-250 °C for 60 min induced the preferential degradation of hemicellulose with increase in the treatment temperature.

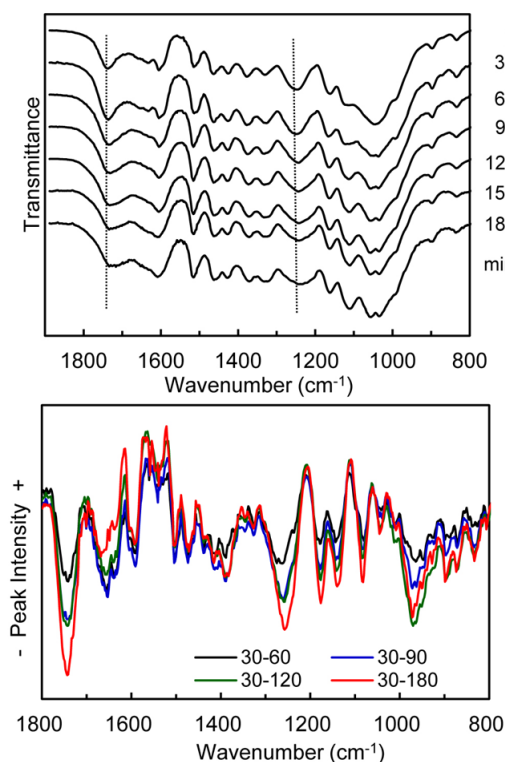


Figure 2 : FTIR spectra and differential spectra of original and SHS treated Moso Bamboo. SHS treatment: at 210 °C for 3 h. Differential spectra were based on the sample of 30 min treatment.

3.2.3 Composition analysis

Changes in composition of main organic ingredients were examined quantitatively during SHS treatment at 210 °C for 3 h, at which temperature the DTG peak based on hemicellulose finally disappeared. Results were illustrated in Figure 3. At first, the composition of cellulose, hemicellulose, and lignin was 44:29:27 (wt%) in agreement with previous reports [16]. After the SHS treatment, the content of hemicellulose decreased linearly from 29 wt% to less than 10 wt%, while the cellulose content remained at 44 wt% and the lignin content increased from 27 wt% to around 50 wt%. These results demonstrate the preferential degradation of hemicellulose and the contrastive increase in lignin content over the duration of SHS treatment. The content of cellulose did not decrease unlike hemicellulose, nor did it increase linearly unlike lignin. This suggests that a small quantity of cellulose such as amorphous cellulose was also removed during the hemicellulose degradation.

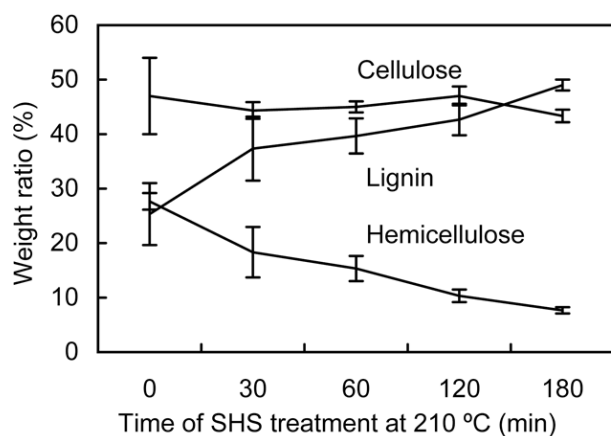


Figure 3 : Quantitative changes in main organic components of bamboo during SHS treatment at 210 °C for 3 h.

3.2.4 Changes in molecular weight of cellulose microcrystalline

In order to clarify influences of the hemicellulose degradation temperature on cellulose, changes of cellulose microcrystalline were evaluated under the same conditions of SHS treatment. In Figure 4, no definite changes in FT-IR spectrum of the cellulose microcrystalline were detected during treatment at 210 °C for 3 h. The DTG curve of microcrystalline became narrow in an early stage up to 30 min, however no change in the curve was observed during the period 30 min - 3 h (Figure 5). The early narrowing of the curve is due to the disappearance of a shoulder in a lower temperature region around 300 °C, indicating the degradation of a small amount of

the easily degradable amorphous part of the microcrystalline as suggested above.

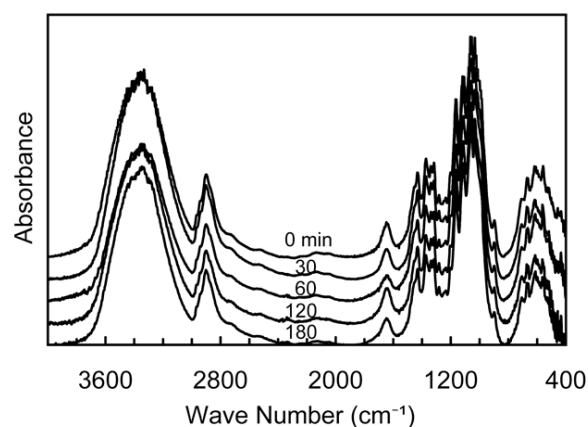


Figure 4 : FTIR spectra of SHS treated cellulose microcrystalline. SHS treatment: at 210 °C for 3 h.

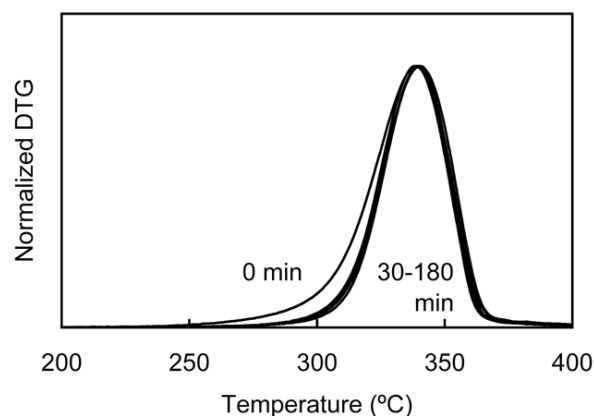


Figure 5 : DTG curves of SHS treated cellulose microcrystalline. SHS treatment: at 210 °C for 3 h.

Molecular weight of the cellulose microcrystalline was evaluated as viscosity-averaged molecular weight (M_v) using its LiCl/DMAc solution. Changes in M_v value during the SHS treatment at 210 °C for 3 h were plotted in Figure 6. The plot demonstrates that the M_v value of cellulose microcrystalline was stable over the SHS treatment without any degradation. This result clearly shows that the cellulose crystalline region in the bamboo tissue was not affected under the conditions of 210 °C for 3 h, under which conditions hemicellulose is degraded preferentially, and the amorphous parts of cellulose might also decompose in the same way as the hemicellulose.

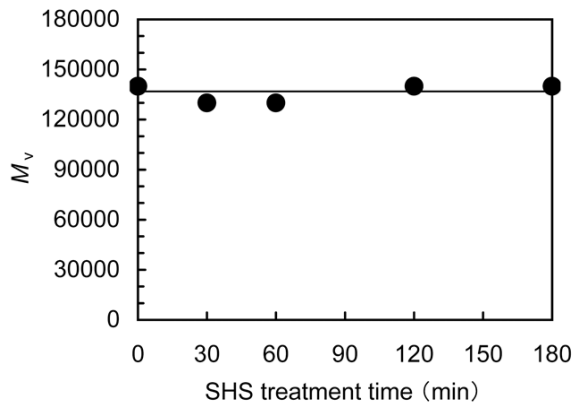


Figure 6 : Changes in viscosity-average molecular weight (M_v) of SHS treated cellulose microcrystalline. SHS treatment: at 210 °C for 3 h.

3.3 Changes in inorganic components

Scurlock et al. [16] studied the composition of ash elements as oxides after combustion at 600 °C for nine bamboo samples, reporting that K_2O and P_2O_5 were main components in ranges of 31-50% and 11-21%, respectively. In order to confirm the elemental composition in raw and SHS treated bamboo samples, XRF analysis was conducted without any combustion process. In Figure 7, the distribution of inorganic components at the surface and inner layers of bamboo are illustrated. In confirmation of Scurlock's data, potassium, which may relate to electrical properties of biocomposites, was detected as the main element of total bamboo. Calcium and chlorine were major elements in the inner layer. Chlorine, which may induce chlorine corrosion of inner surfaces of an extruder during melt-blending, in particular was detected only in the inner layer. Interestingly, in the surface layer, there was a concentration of silicon as a main element of around 50%, which is more than the concentration of potassium. Li reported that silica content was the highest in the epidermis of bamboo culm [25]. Silicon is a major reason of the abrasion of blade when the mechanical pulverization was applied to raw bamboos. Phosphorous, sulfur and aluminum were also detected as minor elements of total bamboo. These results indicate that the surface layer is a specific layer comprising different elements to those constituting the total bamboo stalk.

When the bamboo stalk was treated by SHS at 210 °C for 3 h, no significant change in the elemental composition was observed not only for surface layer, but also for the inner layer. Thus, it is concluded that the SHS treatment produces few changes in the inorganic components of bamboo.

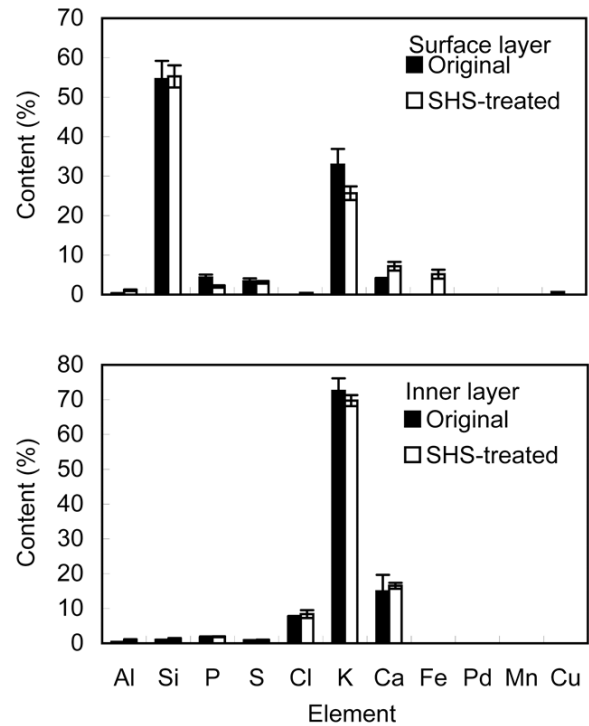


Figure 7 : Changes in inorganic components of SHS treated bamboo. SHS treatment: at 210 °C for 3 h.

3.4 Preparation of sBF

The SHS treated bamboo was easily ground to obtain sBF and a fine powder, which was made up of not only sBF dust, but also of amorphous particles. The sBF and amorphous particles were less than 750 μm in length and less than 50 μm in diameter, and were derived from vascular bundles and parenchymatous ground tissue, respectively (Figure 8) [26]. The moisture content of ground products was in a range of 3-5%.

Figure 9 shows the length and diameter distribution histograms of sieved components: 150-250 μm , 63-150 μm , and 0-63 μm prepared by grinding after the SHS treatment at 210 °C for 3h. Since almost all the sBF were less than 750 μm in length and less than 200 μm in diameter, the dimensions of the sBF were adequate to be used as a composite component for injection molding. When the sBF was used in injection molding, the sBF has to pass through a screen of extruder, and narrow spaces of sprue, runner, and gate of metal molds. Therefore, the size of sBF is restricted. Main fractions in length were 200-250, 50-100, and <50 μm for the sieved components: 150-250 μm , 63-150 μm , and 0-63 μm , respectively, and in diameters 180-190, 10-20, and <10 μm , resulting in different aspect ratio (AR) values of 2.85, 9.94, and 3.24, respectively. These distribution histograms show that the sieved component 150-250 μm includes a large amount of the bundle structure of elementary

fibers, whereas the component 63-150 μm mainly consists of cellulose elementary fibers [27]. The component 0-63 μm comprises grinded particles derived from parenchymatous ground tissue.



Figure 8 : sBF including fine particles obtained by grinding from SHS treated bamboo.

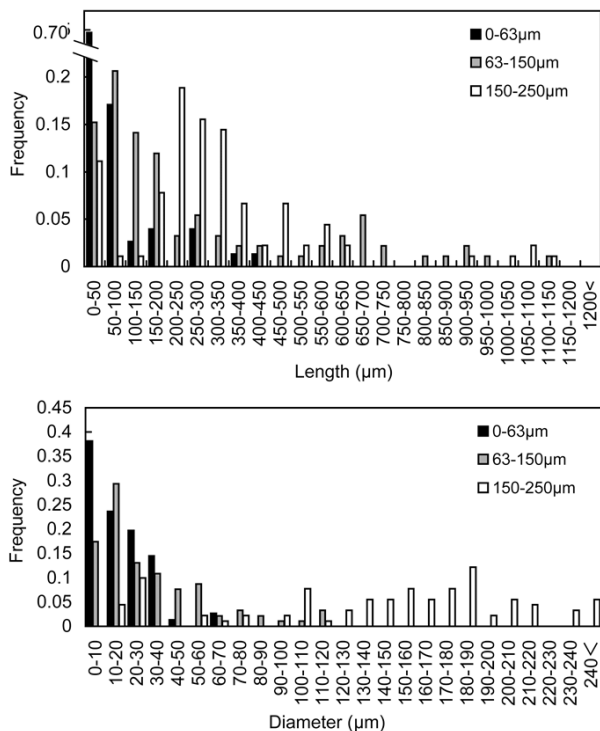


Figure 9 : Length and diameter distribution histograms of sBF components prepared by grinding and sieving after SHS treatment at 210 °C for 3h.

3.5 Mechanical properties of PP/sBF composites: Effects of sBF morphology

The three kinds of sBF as sieved components: 150-250 μm , 63-150 μm , and 0-63 μm were melt-blended with PP to prepare composite pellets. The weight ratio, sBF:PP = 30:70 (wt/wt), was employed, because of showing the highest mechanical properties in proportion to the sBF content without any compatibilizer in a preliminary examination. The composite pellets were injection-molded to produce specimens for flexural tests. In Figure 10, strain-stress curves of the specimens and original PP as a reference were illustrated. Obviously, flexural modulus and strength values significantly increased in comparison with neat PP. A close look at Figure 10 shows in order of flexural strength: 79 MPa (63-150 μm , AR 9.94) > 68 MPa (0-63 μm , AR 3.24) > 55 MPa (150-250 μm , AR 2.85) \approx 54 MPa (PP), and in order of flexural modulus: 3.2 GPa (AR 9.94) > 2.5 GPa (AR 3.24) \approx 2.5 GPa (AR 2.85) > 1.1 GPa (PP). The superiority of sBF (63-150 μm , AR 9.94) must be due to its high aspect ratio, which is a key factor in fiber-reinforcement; the higher the AR value, the more effectively the reinforcement effect appears on the biocomposites. Moreover, it has been suggesting that the high AR value effects in dimension stability and electrical property of composites. These effects will be reported elsewhere in detail in future.

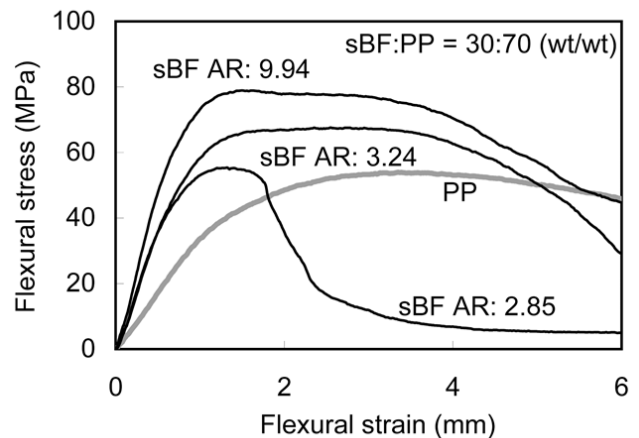


Figure 10 : Stress-strain curves in flexural tests of sBF:PP = 30:70 (wt/wt). sBF: 150-250 μm (AR 2.85), 63-150 μm (AR 9.94), and 0-63 μm (AR 3.24).

4. Conclusion

Moso bamboo was treated by SHS, resulting in the preferential decomposition of hemicellulose without any decrease in the molecular weight of cellulose crystalline. The SHS treated bamboo was easily ground to a fine powder capable of passing through 250 μm aperture, and classified by sieving to obtain various sBF having different aspect ratios. Inorganic

component analysis revealed that potassium element evenly presented as a main component, and silicon element was unevenly distributed at the surface layer. Composition of these inorganic elements was maintained even after SHS treatment. Blends of sBF having the highest aspect ratio of 9.94 with PP (30:70 (wt/wt)) showed excellent melt-processability to produce high-performance composites by injection molding, resulting in flexural strength and modulus values of 79 MPa and 3.2 GPa, respectively, which are significantly higher than those of original PP of 54 MPa and 1.1 GPa, respectively.

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