



Short Bamboo Fibers Coated by Lignin during Super-Heated Steam Treatment and Bio-composites using Same

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Short Bamboo Fibers Prepared by Super-Heated Steam Treatment for Antistatic Bio-composites

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In order to apply short bamboo fiber (sBF) as a functional reinforcement of bio-composites having specific electrical properties, lignin-surfaced sBF was prepared via super-heated steam (SHS) treatment of bamboo. The sBF was easily isolated from the intrinsic fibrovascular bundle structure of bamboo after SHS treatment and pulverization. The isolated sBF was surfaced by brown-colored hydrophobic compounds, which were lignin-derived compounds generated during the SHS treatment. The functional bio-composites were prepared from the SHS-treated sBF and polypropylene and showed specific antistatic properties. Surface electrical resistance values of the composites decreased significantly with increase in the aspect ratio (AR) value of sBF. It is considered that the lignin-derived surfacing of sBF functions as an electron carrier in the composite. In particular, the longer sBF acts as an effective bridge for transporting electrons over long distances along conductive paths. From a cross-sectional microscopic image of the bio-composite, orientated sBFs were observed in its surface layer, supporting the suggestion of conductive path formation. Further, it was confirmed that the reinforcing effect of the presence of sBF was increased with increasing AR value.

Keywords: Short bamboo fiber; Super-heated steam; Lignin-derived compound; Aspect ratio; Antistatic bio-composite; Surface electrical resistance; Fiber reinforcement

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INTRODUCTION

Bamboo is an abundant biomass available in many countries. The total bamboo forest area worldwide is 22 million hectares, which generates over 30 million tons of bamboo fiber (BF) per year (Han *et al.* 2008). Bamboo takes only six to eight months to fully mature; therefore, bamboo plantations regenerate about twenty times more rapidly than normal woods. BF is a typical ligno-cellulosic fiber possessing a much lower density than glass or steel fibers, a high tensile modulus, and low elongation at break (Shin *et al.* 1989). Because of its useful physical and mechanical properties, BF has attracted worldwide attention as a potential reinforcement for polymer composites. It is anticipated that its low water absorption, based on an intrinsic fibrovascular bundle structure, is a property that will make it desirable for blending with hydrophobic plastics.

Much research has been ongoing concerning the effect of its alkali treatment (Rao *et al.* 2011), size (Takagi *et al.* 2004), and content (Bonse *et al.* 2010) on bio-composites with various plastics. Bonse *et al.* (2010) reported that a compatibilizer: maleic anhydride-grafted polypropylene (PP) had a significant positive effect on the mechanical properties of polypropylene/bamboo fiber composites. Phuong and Gilbert investigated

the effect of alkaline and acetylation treatments of BF surfaces. The alkali treatment of BF improved the mechanical properties of bio-composites such as tensile strength as well as the Charpy impact strength. Acetylated BF also showed better mechanical properties due to the grafting of acetyl groups onto the cellulose fiber surface, improving the compatibility between BF and polypropylene (Phuong and Gilbert 2010). Kushwaha and Kumar investigated the chemical surface modification of woven bamboo mat with maleic anhydride, permanganate, benzoyl chloride, benzyl chloride, and pre-impregnation, resulting in increases in tensile strength and modulus of composites when used with epoxy and polyester (Kushwaha and Kumar 2011). Moreover, they reported that silane treatment significantly reduced the water absorption properties of mercerized bamboo mat-reinforced polyester composites compared to untreated bamboo (Kushwaha and Kumar 2010).

Short fiber has been attracting much interest because it is easily processed in melt-blending with plastics and can be molded into many intricately shaped products. Many of the short fiber reinforced bio-composites are prepared by injection molding (Nayak *et al.* 2009), allowing products to be manufactured with high precision and at high speed. In order to prepare short bamboo fiber (sBF) effectively, physical and chemical treatments of bamboo have been conducted. Okubo *et al.* (2004) prepared sBF by the mesh filtration of mechanically pulverized bamboo chips. Tokoro *et al.* (2008) prepared three kinds of sBF: short bundle, alkali-treated filament, and steam exploded filament using a cutting machine, in a 1.5N NaOH solution at 70 °C, and over-heated steam at 175 °C and 0.7 to 0.8 MPa, respectively.

Chattopadhyay *et al.* (2011) treated BF with NaOH at 160 °C followed by grinding and sieving to obtain sBF. Although mechanical pulverization is the simplest method, blades suffer severe abrasion in contact with the hard epidermis of bamboo. There are some disadvantages also associated with other means of sBF production. Steam explosion is a popular method for isolating sBF, but it is difficult to operate on a large scale because a high-pressure resistant reactor is required. Chemical treatment is a desirable method for isolating the prescribed forms of cellulose fiber, but this has the disadvantage of creating a large amount of liquid waste that must then be processed.

In comparison with the above sBF preparation methods, super-heated steam (SHS) treatment is a superior method for preparing sBF, because SHS is a powerful and clean reagent for biomass conversion to organic materials and tar-free vinegar at normal pressure (Bahrin *et al.* 2012; Yamashiro and Nishida, 2015). Recently, SHS treatment has been attracting the attention of researchers not only as a powerful method of producing sBF, but also as a process for effecting changes in the surface properties of sBF during the SHS treatment. In this paper, in order for sBF to operate as a functional filler of bio-composites so as to be utilized in various industrial fields, changes in chemical properties of the sBF surface and some specific properties of the bio-composites are investigated.

EXPERIMENTAL

Materials

Moso bamboo (*Phyllostachys heterocycla f. pubescens*) samples (diameter: 10 to 20 cm at chest height, height: *ca.* 20 m) were collected at Yame city in Japan. Polypropylene (PP) (Japan Polypropylene Corporation, Japan, extrusion grade

NOVATEC-PP FY6, density $0.90 \text{ g}\cdot\text{cm}^{-3}$, melt flow index $2.5 \text{ g}\cdot 10 \text{ min}^{-1}$) was used as a matrix resin.

SHS Treatment and Preparation of sBFs

The Moso bamboo sample was cut to 40 cm in length, treated by SHS at 150 to 230 °C for 3 h under thermostating within ± 5 °C at a constant steam flow rate of $6 \text{ kg}\cdot\text{h}^{-1}$ in a SHS oven: model NHL-1 (Naomoto Corp., Japan) inner dimensions: W $590 \times$ D $385 \times$ H 555 mm^3 with an internal fan for agitating the steam. After the SHS-treatment, the bamboo was converted to a mixture of sBF and fine bamboo powder (fBP) with pulverizers: RUB Master SRM-15 (Sanken Engineering Co., Ltd., Japan) and a Jiyu mill type M-2 (Nara Machinery Co. Ltd., Japan). The obtained mixture was then sieved under vibration with a sieve shaker model MVS-1 (AS ONE Corp., Japan) using two kinds of sieves with aperture sizes: 63 and 106 μm to differentiate material into fBP ($< 63 \mu\text{m}$ in particle size) and sBF (63-106 μm in diameter) as the main portions. The fibers having longer than 106 μm in length also passed through the sieve, leaving a large diameter size residue ($> 106 \mu\text{m}$). Moisture contents of obtained products were measured on a moisture-balance MOC-120H (Shimadzu Corporation, Japan) at 105 °C.

Melt-blending with Polypropylene and Mechanical Properties of Bio-composites

In order to evaluate the contribution of sBF to the mechanical properties of bio-composites, two kinds of sBFs were melt-blended with polypropylene (PP) in prescribed weight ratios of PP:sBF = 10:90~50:50 (wt/wt).

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, 200, and 210 °C for four zones from hopper to die with a screw rotational speed of 15 rpm. Extruded sBF/PP composite strands were cut into pellets with a pelletizer.

Casting sheet samples ($100 \times 100 \text{ mm}^2$, thickness 0.6 mm) were prepared from sBF/PP pellets by heat-pressing with a compact heating press IMC-180C (Imoto Machinery Co., Ltd., Japan) at 190 °C/12 MPa for 5 min.

Injection molding of sBF/PP pellets was carried out with a simple injection molding machine IMC-18D1 (Imoto Machinery, Japan) at 190 °C/12 MPa for 30 s, resulting in the preparation of specimens ($20 \times 5 \times 2 \text{ mm}^3$) for flexural strength tests.

Characterization

Morphology of sBF was observed on a digital microscope VH-5000 (KEYENCE, Japan) equipped with a high resolution zoom lens VH-Z500 to obtain an averaged aspect ratio value from length and diameter values of more than 100 pieces of sBF. Micromorphology of sBF was scrutinized with a 3D laser scanning confocal microscope model VK-X 100/105 (KEYENCE, Japan) under prescribed conditions of laser: red semiconductor laser, $\lambda=658 \text{ nm}$, 0.95 mW, and pulse width of 1 ns. Micromorphology of the cross-sectional surface of a sBF/PP bio-composite molding was observed on a scanning electron microscope (SEM) (Hitachi Ltd., Japan, model S-3000N) at an accelerating voltage of 15 kV with an electron conductive layer of platinum.

The chemical structure of the surface layer of SHS-treated sBF was analyzed with a microscopic Fourier transform infrared (FT-IR) spectroscope Nicolet iN10 MX

(Thermo Fisher Scientific, Japan) equipped with an image-mapping system in reflection absorption mode and in a range of 675 to 4000 cm^{-1} .

Surface electrical resistance values of the sBF/PP bio-composite sheet samples were measured on a Hiresta UP (Mitsubishi Chemical Analytech Co., Ltd., Japan) equipped with a probe UR-100 (measurement range: 10^{10} - 10^{16} Ω , main electrode: $\phi 50$ mm, inner diameter of guard: $\phi 53.2$ mm). After charging at an applied voltage of 1000 V for 60 s, the sample was measured to obtain surface resistance values at 23 °C. Measurements were taken fivefold to obtain average values.

Flexural strength tests of the bio-composite specimens were conducted on a compact tensile and compression tester IMC-18E0 (Imoto Machinery Co., Ltd., Japan) at a bending rate of 10 $\text{mm}\cdot\text{min}^{-1}$.

RESULTS AND DISCUSSION

The Fibrovascular Bundle Structure of Bamboo

The tissue structure of bamboo is characterized by its intrinsic fibrovascular bundle structure (Fig. SM-1 in Supplementary Materials). A major ingredient of the fibrovascular bundle structure is a multi-laminated and linearly grown cellulose crystalline structure, which is entwined by hemicellulose and lignin ingredients (Fuentes *et al.* 2011), as shown in Fig. 1. These integrated structures give the bamboo great rigidity allowing it to grow to heights of greater than 20 m despite its hollow structure.

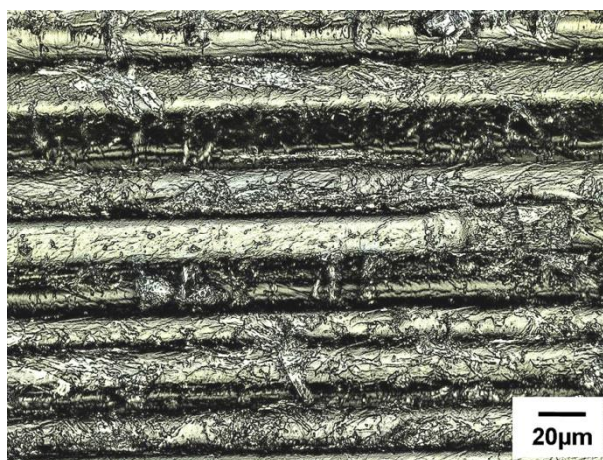


Fig. 1. 3D laser scanning confocal microscopic image of a cellulose fibrovascular bundle structure entwined by hemicellulose and lignin ingredients. Magnification: $\times 1000$. Bar 20 μm

Super-heated Steam Treatment of Bamboo

The simplest procedure thus far developed for extracting sBF from the fibrovascular bundle structure is by SHS treatment of the bamboo by preferentially degrading the hemicellulose ingredient surrounding cellulose bundles and removing it by SHS flow to recover specific bamboo vinegar (Yamashiro *et al.* 2015). The bamboo vinegar also contained phenolic degradation products as minor components derived from lignin ingredient, which was also partly degraded in the process and discharged by the SHS flow.

After pulverization and sieving of the SHS treated bamboo, isolated sBF was found to be dark brown in appearance (Fig. SM-2). The water content of this sBF, in a range of 3 to 5 wt%, was remarkably lower than the 51 wt% (Kushwaha and Kumar, 2010) of raw bamboo. Interestingly, the hydrophobic property of sBF was retained for a long time (more than 1 year) without change under normal atmospheric conditions including rainy season in Japan, suggesting that a hydrophobic layer covered the sBF surface during the SHS treatment. In Fig. 2, a 3D laser scanning confocal microscope image of a cellulosic fibrovascular bundle structure and parenchymal cells after SHS treatment at 210 °C for 3 h is shown. As reported previously (Yamashiro *et al.* 2015), the SHS treatment at 210 °C was favorable due to the lowest temperature, at which almost all of the hemicelluloses could have degraded, while no crystalline cellulose would have broken down. The hemicellulose and lignin ingredients previously surrounding the bundle structure had either disappeared or been covered over as a glossy surfacing. In order to confirm the presence of the surfacing, a cellulosic filter paper was put just above the bamboo tips and treated by SHS at 210 °C for 3 h, resulting in the cellulosic filter paper changing in color to pale brown and thereafter showing an ability to hold a water droplet in the typical manner of a hydrophobic surface (Fig. SM-3). This hydrophobicity disappeared after washing with toluene as a hydrophobic solvent, resulted in lightening in color and water absorption. Thus, it is assumed that this brown colored, glossy, and hydrophobic layer is generated from lignin ingredient during the SHS treatment.



Fig. 2. 3D laser scanning confocal microscope image of a cellulosic fibrovascular bundle structure and parenchymal cells after SHS treatment at 210 °C for 3 h. Magnification: $\times 1000$. Bar 20 μm

Pulverization of SHS-treated Bamboo and Classification

The SHS-treated bamboo was easily pulverized to obtain sBF and fBP, derived from fibrovascular bundles and parenchymatous ground tissue, respectively (Yamashiro and Nishida, 2015). When the sBF was used as a reinforcement fiber agent in injection molding, it was first made to pass through a screen located at the end of the barrel of an extruder, and then the narrow spaces of the sprue, runner, and gate of metal molds. Therefore, the dimensions of sBF were constrained to being less than the aperture sizes. Since after sieving almost all the sBFs were less than 750 μm in length and less than 50 μm in diameter, the dimensions of selected sBF were judged to be suitable for use in injection molding.

The two classifications of sBF (63-106 μm) and fBP ($< 63\mu\text{m}$) after the SHS treatment at 210 $^{\circ}\text{C}$ for 3 h showed significantly different aspect ratio (AR) values: 12.3 and 2.8, respectively. Further, when the SHS temperature was varied in a range of 150 to 230 $^{\circ}\text{C}$, sBF classified by sieving showed specific changes in AR values: 13.0 (SHS treatment temperature: 150 $^{\circ}\text{C}$), 14.1 (170 $^{\circ}\text{C}$), 15.1 (190 $^{\circ}\text{C}$), 12.3 (210 $^{\circ}\text{C}$), and 8.0 (230 $^{\circ}\text{C}$) as shown in Fig. 3. These changes in AR value are considered to be due to the SHS temperature, which as it increases facilitates the isolation of fibrovascular bundle structure, but also causes decomposition of the fiber structure because of damage to the cellulose crystalline. These isolation promotion and the decomposition effects of BF must be responsible for the result in Fig. 3. Thus, the highest AR value at 190 $^{\circ}\text{C}$ is the balanced result between the both effects. Although the sBF samples were prepared at different SHS temperatures, their water content was almost identical in a narrow range of around 6 wt %.

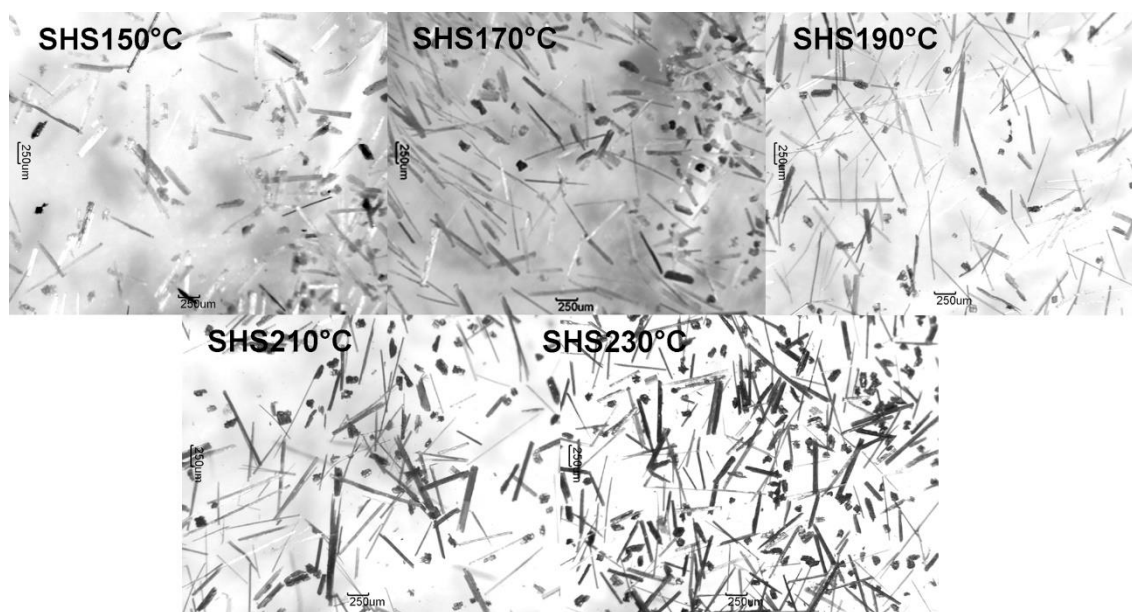


Fig. 3. Classified sBFs (63-106 μm) prepared at different temperatures. AR value (SHS treatment temperature): 13.0 (150 $^{\circ}\text{C}$), 14.1 (170 $^{\circ}\text{C}$), 15.1 (190 $^{\circ}\text{C}$), 12.3 (210 $^{\circ}\text{C}$), and 8.0 (230 $^{\circ}\text{C}$)

Characterization of Surfacing Layer Formed by SHS Treatment

Characterization of the surfacing layer formed by SHS treatment was carried out by microscopic FT-IR analysis. The microscopic FT-IR reflection spectra before and after the SHS treatment at 210 $^{\circ}\text{C}$ for 3h are depicted in Fig. 4. These are integrated spectra produced from 20 measurements for each sample. The raw sBF surface before the SHS treatment showed characteristic absorption peaks at 1396 ($\delta_{\text{C-H}}$), 1134 ($\nu_{\text{as,C-O}}$), 1080 ($\nu_{\text{C-O}}$), 1057 ($\nu_{\text{C-O}}$), and 879 cm^{-1} ($\nu_{\text{glucose ring}}$), which are derived from cellulose, whereas the SHS treated sBF surface showed typical absorption peaks derived from lignin at 1720 ($\nu_{\text{C=O}}$), 1643 ($\nu_{\text{aromatic ring}}$), 1543 ($\nu_{\text{aromatic ring}}$), 1504 ($\nu_{\text{C=C}}$), 1450 ($\delta_{\text{C-H}}$), 1288 ($\nu_{\text{C-O,guaiacyl ring}}$), 1126, 1003, 957 ($\delta_{\text{C-H,out of plane}}$), 756, and 694 cm^{-1} ($\delta_{\text{C-H,aromatic}}$) (Yang *et al.* 2007; Pandey 1999). These spectra clearly show that the cellulose-rich sBF was surfaced by lignin-derived aromatic compounds: *e.g.*, phenolic, guaiacyl, and syringyl compounds.

In Fig. SM-4, results of the principal component analysis are illustrated. The principal component analysis also suggested lignin-derived compounds as the first

principal component, indicating that the surfacing layer consists of lignin-derived compounds formed during the SHS treatment.

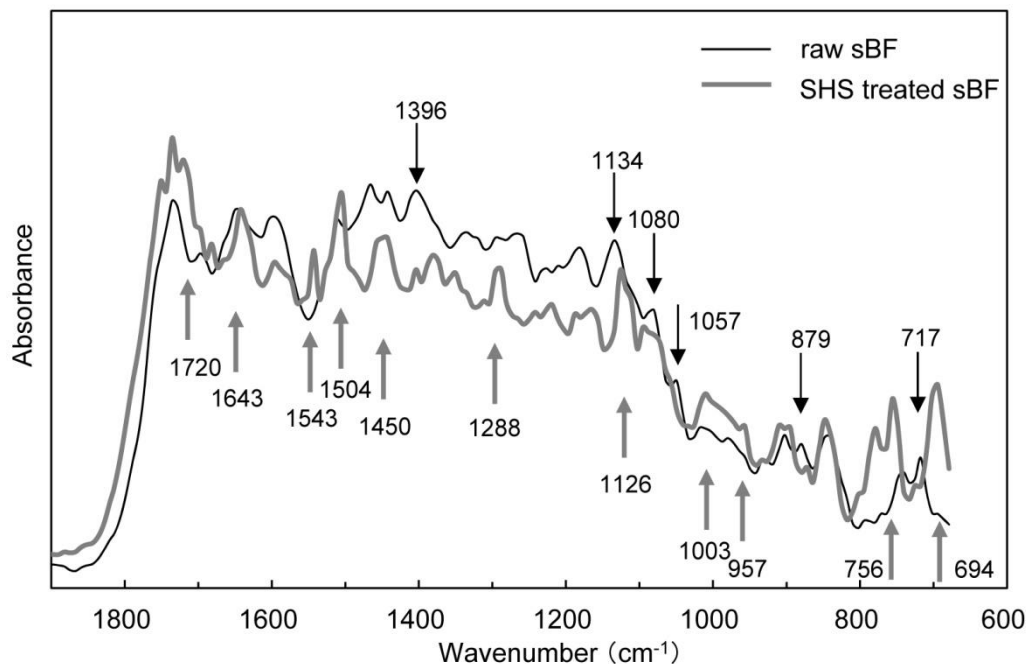


Fig. 4. Integrated microscopic FT-IR reflection spectra of raw and SHS-treated sBF samples at 210 °C for 3 h. Number of measurements: 20 times for each sample.

Antistatic Properties of sBF/PP Bio-composites

The antistatic property of sBF/PP bio-composites was evaluated from the measurements of surface and volume electrical resistance values of their sheet samples. In Fig. SM-5, the effects of size, AR, and content of sBF on the electric resistance of samples are shown in a composition range of PP:sBF = 10:0 ~ 5:5 (wt/wt). The surface electric resistance value of bio-composites was decreased by the addition of 10% sBF from the previous value of 10^{16} Ω for neat PP to around 10^{13} Ω . This value was then gradually decreased from 10^{13} to 10^{12} Ω with increase in the content of sBF (63-106 μm , AR 12.3), whereas the surface electric resistance value of PP/fBP (< 63 μm , AR 2.8) bio-composites remained at a constant value of around 10^{13} Ω , even with increase in the fBP content. On the other hand, the volume electrical resistance scarcely reduced from the value of 10^{16} Ω for neat PP in the composition range of PP:sBF = 10:0 ~ 5:5 (wt/wt).

In order to confirm the effect of AR value on the surface electrical resistance, 5 kinds of sBF/PP (63-106 μm) (50:50 wt/wt) bio-composites were prepared. In Fig. 5, changes in the surface electrical resistance of the bio-composites are shown. Interestingly, a relationship of inverse proportion was found between the surface electrical resistance value and the AR value of sBF (63 to 106 μm). Any influence of water content could be ignored because water content was constant at around 7 wt% in the sBFs used. These results suggest that the layer of surfacing of the sBF functions as an electron carrier in the bio-composites as a result of the lignin-derived materials layer (Volpati *et al.* 2011); and the larger the AR value, allowing electrons to be transported more easily over long distances along conductive paths formed by sBF bridges (Shen, 2011).

From a cross-sectional SEM image of sBF/PP (63 to 106 μm) (50:50 wt/wt) bio-composite (Fig. SM-6), it was observed that sBFs concentrated in the surface layer of bio-composites, and were frequently found to be contacting each other to form conductive paths. Contrastively, only a small amount of sBF was observed in the moldings central area explaining the relatively small decrease in volume electrical resistance.

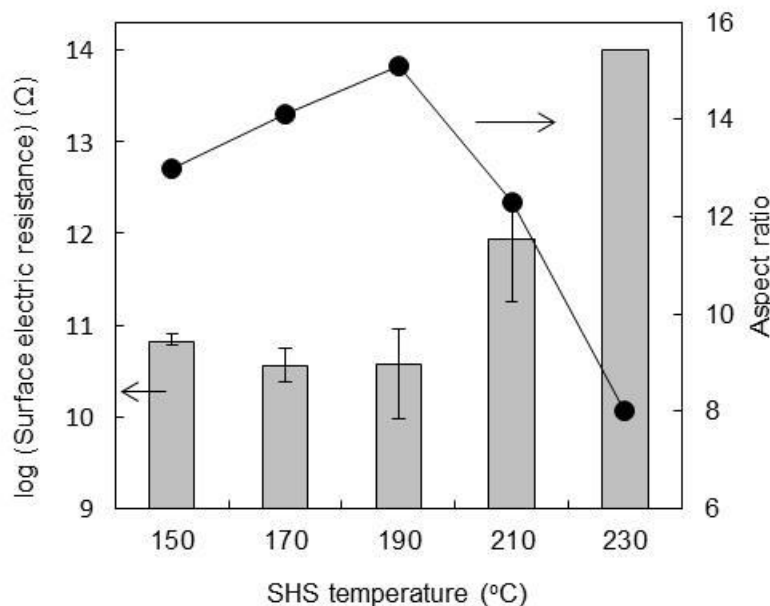


Fig. 5. Effects of aspect ratio (AR) value of sBF (63-106 μm) on surface electric resistance of sBF/PP (50:50 wt/wt). Samples: sBF (AR 13.0), (AR 14.1), (AR 15.1), (AR 12.3), and (AR 8.0) were prepared by SHS treatment at 150, 170, 190, 210, and 230 $^{\circ}\text{C}$, respectively, for 3h and followed by pulverization and sieving.

Mechanical Properties of sBF/PP Composites

As referred to in a previous report (Yamashiro and Nishida 2015), three kinds of sBF and fBP: 150 to 250 μm (AR 2.85), 63 to 150 μm (AR 9.94), and < 63 μm (AR 3.24) after SHS treatment at 210 $^{\circ}\text{C}$ for 3h were melt-blended with PP to prepare sBF/PP and PP/fBP (70:30 wt/wt) bio-composites. These bio-composites showed significantly improved mechanical properties of flexural strength: 79 MPa (AR 9.94) > 68 MPa (AR 3.24) > 55 MPa (AR 2.85) \approx 54 MPa (PP), and 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), respectively. The superiority of sBF (AR 9.94) must be due to its high AR value, which is the key factor in the fiber-reinforcement of bio-composites. Therefore, it was confirmed that for larger AR values, not only was the antistatic property boosted, but there was also a strengthening of the reinforcement effect of SHS-treated sBF on the mechanical properties of bio-composites.

CONCLUSIONS

1. Bamboo consists of an intrinsic cellulosic fibrovascular bundle structure with entwined hemicellulose and lignin ingredients. The SHS treatment facilitated the easy conversion of bamboo to fine sBF and fBP, changing both its color to brown and its

chemical properties at the surface to become more electrically conductive. The sBF was surfaced by glossy hydrophobic compounds, which were lignin-derived compounds generated during the SHS treatment.

2. Bio-composites prepared from SHS-treated sBF and PP showed interesting antistatic properties, indicating that the surfacing of the sBF functions as an electron carrier in the composite. The larger the AR value of sBF, the more easily the electrons were transported over long distances along conductive paths formed by sBF bridges. From the cross-sectional SEM image of sBF/PP bio-composite, concentrated sBFs in the surface layer of the bio-composite were observed, forming conductive paths through increased inter-fiber contact. Further, it was confirmed that the reinforcement effect increased with increase in the AR value of sBF.

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APPENDIX

Short Bamboo Fibers Prepared by Super-Heated Steam Treatment for Antistatic Bio-composites

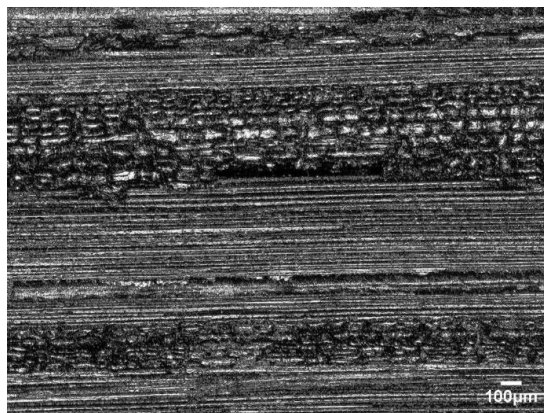


Fig. SM-1. Digital microscope image of fibrovascular bundle structure of *Phyllostachys heterocycla f. pubescens* (Moso bamboo). Magnification: $\times 100$. Bar 100 μm .

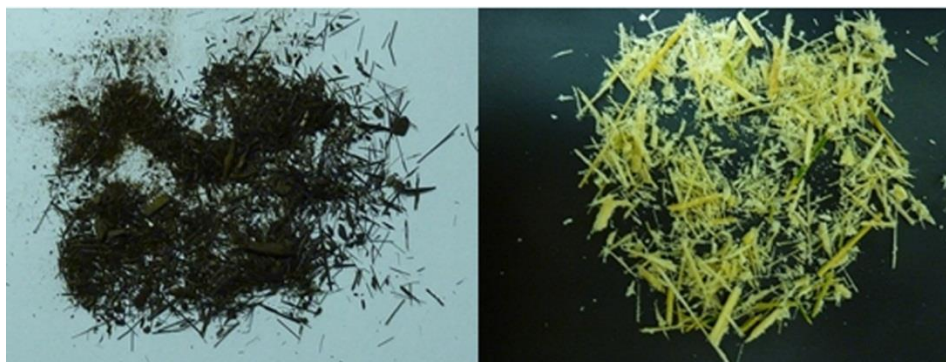


Fig. SM-2. Isolated sBF changed color to dark brown (left) from pale yellow of raw bamboo powder (right).

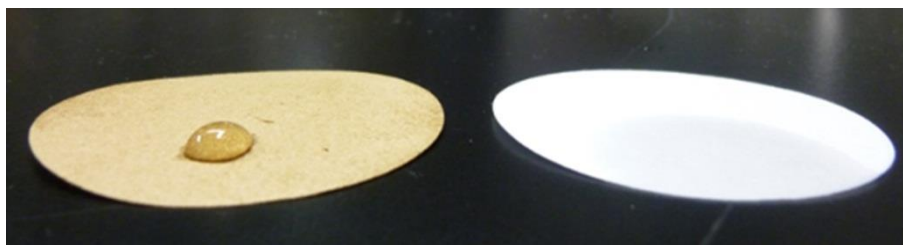


Fig. SM-3. Effect of SHS treatment of bamboo at 210 $^{\circ}\text{C}$ for 3 h. Cellulosic filter paper (left) put just on the upper side of bamboo in a SHS reactor and original filter paper (right). The SHS-treated paper retained water droplet by showing xx degree of contact angle on the surface. On the other hand, the original filter paper smoothly absorbed water droplet.

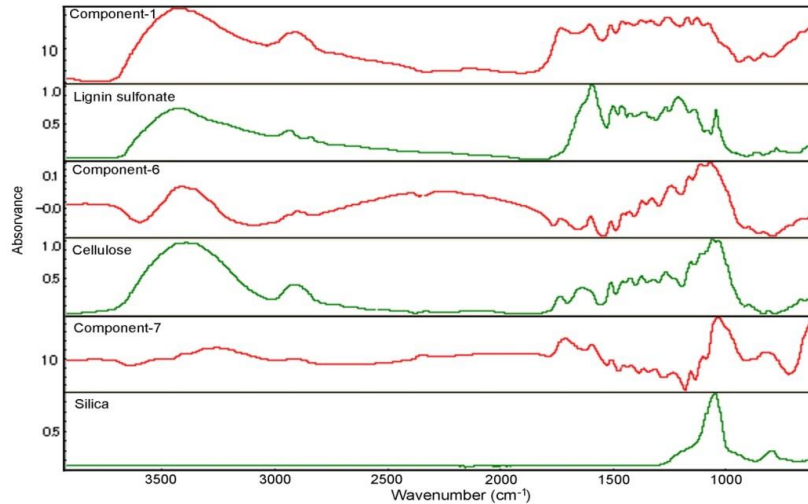


Fig. SM-4. Principle component analysis of microscopic reflection mapping image of SHS-treated sBF at 210 °C for 3 h.

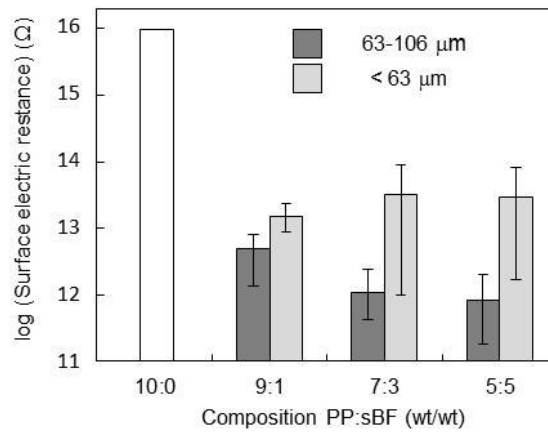


Fig. SM-5. Effects of size, AR, and content of sBF on surface electric resistance of sBF/PP. Samples sBF (63-106 μm, AR 12.3) and fBP (< 63 μm, AR 2.8) were prepared by SHS treatment at 210 °C for 3h and followed by pulverization and sieving.

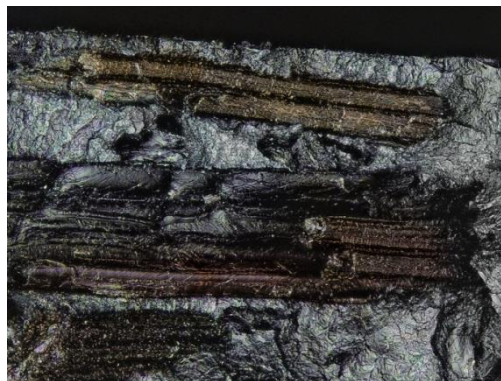


Fig. SM-6. Cross-section SEM image of sBF/PP (63-106 μm) (50:50 wt/wt) bio-composite

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