Microstructural, Thermal, and Tensile Characterization of Banana Pseudo-stem Fibers Obtained with Mechanical, Chemical, and Enzyme Extraction

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Banana pseudo-stem fibers (BPSFs) have desirable tensile properties. In this study, BPSFs were extracted using mechanical, chemical, and enzymatic methods. The aim was to evaluate the effect of these three extraction methods on the tensile, thermal, and morphological properties of BPSFs. Microstructural analysis showed the presence of structural and arch fibers in banana pseudo-stem (BPS). The average tensile strength and elongation for mechanically, chemically, and enzyme-extracted BPSFs were 210, 333, and 235 MPa, and 0.8%, 1.6%, and 1.4%, respectively. Young's modulus was enhanced by 19.1% in the mechanically extracted BPSFs compared with that of chemically extracted BPSFs. The morphology of BPSFs was correlated with their tensile properties *via* scanning electron microscopy (SEM) image analysis. Fourier transform infra-red (FTIR) and X-ray diffraction (XRD) analyses of fibers showed that chemically extracted BPSFs contained less hemicellulose and lignin with a crystallinity index of 61.2%. Chemically extracted BPSFs exhibited enhanced thermal properties over mechanically extracted BPSFs. Mechanically extracted BPSFs demonstrated similar thermal and tensile properties to chemically and enzyme-extracted BPSFs. Thus, mechanically extracted BPSFs could act as highly suitable reinforcing agents in bio-based composite material preparation. Given that mechanical methods need no chemicals and they are environmentally friendly, such techniques have potential applications.

Keywords: Banana pseudo-stem fibers; Tensile properties; FTIR; XRD; Thermogravimetric analysis

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INTRODUCTION

Natural materials have attracted great interest for manufacturing because of ecological concerns (Baley *et al.* 2005). In recent years, natural fibers have been demonstrated to be a feasible replacement for non-renewable, abrasive, and expensive synthetic fibers (Bourmaud *et al.* 2010; Osorio *et al.* 2012). Natural fibers have many advantages, such as biodegradability, renewability, wide availability, low density, low cost, high tensile strength, and high stiffness, which are beneficial for developing lightweight, environmentally friendly structural composites (Reddy *et al.* 2013). Therefore, great efforts have been made to use natural fibers to reinforce polymer composites for applications in the aircraft, automobile, electronics, and medical industries (Sreenivasan *et al.* 2011b; Khan *et al.* 2013).

Demand for natural materials is increasing with the development of densely populated regions. The current production level does not meet today's demand. However, some tropical fibers, such as bananas (*Musa sapientum*), are available in large quantities (Defoirdt *et al.* 2010). Bananas are produced in high quantities in the tropical and sub-tropical regions. At present, bananas are the fourth most widespread fruit crop in the world. After harvesting, a large amount of pseudo-stem residue is left behind in plantation soil to be used as organic material. A few tons per hectare of banana pseudo-stem (BPS) are estimated to be produced annually. These crops have been utilized as a fiber source in the pulping industry, and their decomposition generates energy (Aziz *et al.* 2011). Fibers from the banana plant are comparable in physical strength and cellulose content with fibers obtained from other fibrous commodity by-products, and they have been extensively characterized from their fruit stalk, pseudo-stem, and leaves (Padam *et al.* 2012). Fibers from bananas are an environmentally friendly alternative to glass or other reinforcing fibers utilized in engineering composites.

Raw banana pseudo-stem fibers (BPSFs) are generally composed, by mass, of 59% to 64% cellulose, 10.2% to 18.6% hemicelluloses, 17.5% to 4.9% lignin, 9.1% to 10.4% moisture content, and 2.1% pectin (Deepa *et al.* 2011; Jayaprabha *et al.* 2011). As a natural fiber, BPSFs have specific characteristics, such as antimicrobial properties, high absorbency, hygroscopicity, and good thermal stability. However, high quantities of non-cellulosic components in BPSFs, such as hemicelluloses, lignin, and pectin, and impurities negatively influence the fibers' properties. Various methods, including chemical, mechanical, enzymatic, and combined treatments, have been reported to extract fibers from BPS. Each method presents different advantages or drawbacks according to the amount of fiber produced or the quality and properties of fiber obtained (Ganan *et al.* 2004). Chemical treatment is usually conducted by immersing samples into alkaline solution, based on the mechanism that cellulose and non-cellulosic substances have different stabilities in the solution. Chemical treatment requires a relatively short process time, but it consumes a large number of chemicals that pollute the environment. With biological methods, enzymes or bacteria are used to degrade one or more of the non-cellulosic substances. Biological methods are environmentally friendly, but they require a longer processing time, which significantly affects the fibers' quality. Moreover, the cost of enzymes or bacteria is high. Numerous studies have been conducted on the physical properties of natural fibers extracted by chemical and enzyme-based methods (Jayaprabha *et al.* 2014; Lazim *et al.* 2014). Mechanical separation of fibers from BPS is implemented by scraping, hacking, steam explosion, and ultrasonic vibration. Mechanical treatments are considered to be inefficient in the modification or processing of fiber surface properties and removal of non-cellulosic substances (Ganan *et al.* 2004; Rahman and Itoh 2011). On the other hand, these mechanical methods are environmentally friendly and economical because they don't require chemical usage.

The present study aimed to compare the microstructural, tensile, and thermal properties of BPSFs extracted by mechanical, chemical, and enzyme-based methods. After different treatments, changes in structural composition were verified using Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), and thermogravimetric analysis (TGA). Scanning electron microscopy (SEM) was used to observe the surface morphology and mode of failure of fibers.

EXPERIMENTAL

Materials

BPSs were harvested in Sanmenpo, Haikou, China. BPSFs were extracted from the sheet of the stem. Sodium hydroxide, sulfuric acid, hydrogen peroxide, protease, pectinase, and sodium citrate were used. Protease (Novozyme) and pectinase (Novozyme) were purchased from Novozymes Biotechnology Co., Ltd. (China).

Methods

Mechanical extraction

In this process, fresh BPS sheaths were fed into a fiber-extracting machine called a mechanical decorticator. A schematic of the mechanical decorticator is shown in Fig. 1. The machine consists of a pair of feed rollers and two scraper rollers. The sheaths were fed into the scraper roller with blunt knives through the feed rollers. The pulps were separated by the scraper rollers, leaving only the fibers. Decorticated fibers were dried to obtain 18% product for 24 h in the sunlight.

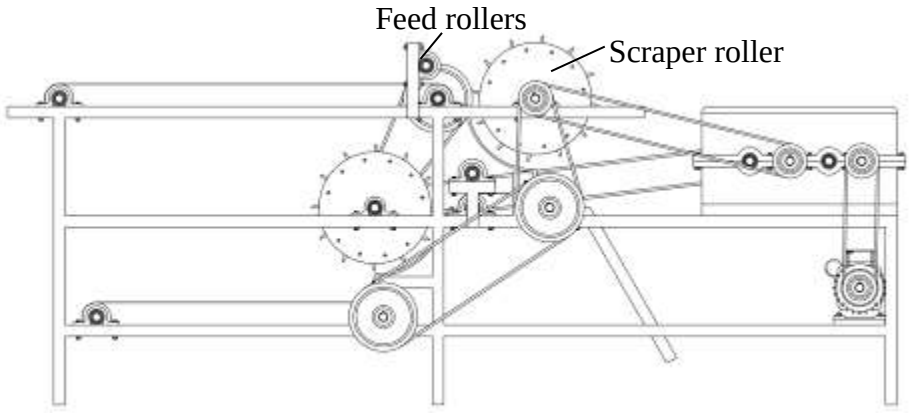

Fig. 1. Schematic of a mechanical decorticator

Chemical extraction

BPS samples were treated with sulfuric acid solution followed by alkali solution to increase the relative amount of cellulose by removing some of amorphous cellulose, hemicelluloses, lignin, and other amorphous materials from the fiber surface. Several studies have reported that alkalization leads to the removal of hydrogen bonds (Fiber-OH+ NaOH→Fiber-ONa+H_2O) in the network structure (Arifuzzaman Khan *et al.* 2013; Hossain *et al.* 2014). In the present study, dried BPS (10 g) was first treated with 200 mL of 2 g/L H_2SO_4 aqueous solution at 55 °C for 2 h to remove external wax, followed by washing with distilled water to remove residual H_2SO_4. The treated banana BPS was immersed in 200 mL of a liquid mixture composed of 7 g/L H_2O_2, 3 wt% Na_2SiO_3, and 2 wt% sodium polyphosphate at 95 °C for 1.5 h to remove hemicelluloses and lignin. Subsequently, the sample was immersed in 200 mL of 9 g/L NaOH solution at boiling temperature for 3 h for further removal of hemicelluloses and lignins and then washed with 2 g/L H_2SO_4 to neutralize the residual alkali in the sample. Finally, the sample was washed with DI water and dried to obtain 32% product in a hot air oven at 105 °C for 24 h.

Enzymatic extraction

The dried BPS sheath was cut into pieces approximately 15 cm long. To remove lignin, 12 g of the BPS sheath was treated with 360 mL of 0.4 wt% trisodium citrate solution at 85 °C for 1 h and then washed with DI water three times. This process was repeated twice. Next, 4.5 g of lignin-free BPS was mixed with 60 mL of 4 g/L protease aqueous solution to decompose protein at 55 °C for 3 h, in which 250 mL of 0.1 wt% trisodium citrate was used as a buffering solution. The sample was washed with DI water and separated by centrifugation. To further remove pectin, the obtained sample was mixed with 60 mL of 2 g/L pectinase enzyme buffered with 350 mL of 4 g/L citric acid-trisodium citrate for 1 h at 55 °C, followed by washing with DI water five times. Finally, the sample was bleached at 70 °C for 1 h with 150 mL of aqueous solution composed of 0.55 wt% H_2O_2 and 0.2 wt% NaOH, washed with DI water, and dried to obtain 34% product in a hot air oven at 105 °C for 24 h.

Characterization and measurement of tensile behavior

Healthy BPS was collected for microstructural analysis. The BPS was cut into 30 μm-thick pieces and observed on an ECLIPSE TS/TS100-F optical microscope (Nikon, Japan) with a maximum magnification of 400×. Morphologies of BPSFs obtained by different methods were examined using a Hitachi S-3000N (Japan) SEM. FTIR spectra of the samples were obtained using a Bruker Spectrum TENSOR 27 (Germany) FTIR spectrometer with a scan rate of 32 scans per minute and a resolution of 4 cm^{-1} in the wavenumber region from 4000 cm^{-} to 500 cm^{-1}. XRD spectra were carried out in a Bruker AXS D8 ADVANCE diffractometer (Germany) with a Co tube operating at 40 kV and 40 mA. All samples were scanned with 2θ ranging from 5° to 70°. TGA was performed using the SDT Q600 (TA Instruments, USA), which was carried out in a nitrogen atmosphere. In each case, 2 mg of fibers was placed in platinum pans and heated from 30 °C to 500 °C, with a heating rate of 10 °C min^{-1}.

The longitudinal tensile properties of BPSFs were measured as per ASTM D3822-01 (2001) standards using a Universal Testing Machine WDW-1 (Jinan Yinuo Century Experiment Instrument Co., Ltd., Shandong, China) under a controlled temperature (35 °C) and relative humidity (65%). In each case, the gauge length was 40 mm. The fiber was mounted on the opposite sides of a paper frame to generate a good grip and keep it straight. A constant crosshead displacement rate was 2 mm/min up to rupture. Sixteen specimens of each sample category were tested to provide statistically significant results as other researchers have worked with 10 samples (Hossain *et al.* 2014). Samples that broke near the edge of the clamps were excluded from the analysis.

RESULTS AND DISCUSSION

Transverse and longitudinal microstructures of BPS are shown in Fig. 2. Transverse sections of the BPS sheath displayed dermal, ground, and vascular tissues, as shown in Fig. 2a. The dermal tissue consisted of a well-defined epidermis with radially oblong, fairly wide cells with thick cuticles. The ground tissue was homogeneous and parenchymatous. The cells were circular, polygonal, thin walled, and compact. Fiber bundles were observed in the ground tissue. Fiber bundles and bundle cap fibers might be observed within the BPS sheath. The fiber bundles provide stiffness for the BPS sheath and are found in the periphery of the stem. The fiber bundles are of great importance

commercially because they almost never split during extraction (Sreenivasan *et al.* 2011b). The bundle cap fibers were found at one end of the vascular tissues. A longitudinal view of the BPS sheath is shown in Fig. 2b, which shows that the bundles were narrow with a segmented structure (Li *et al.* 2010).

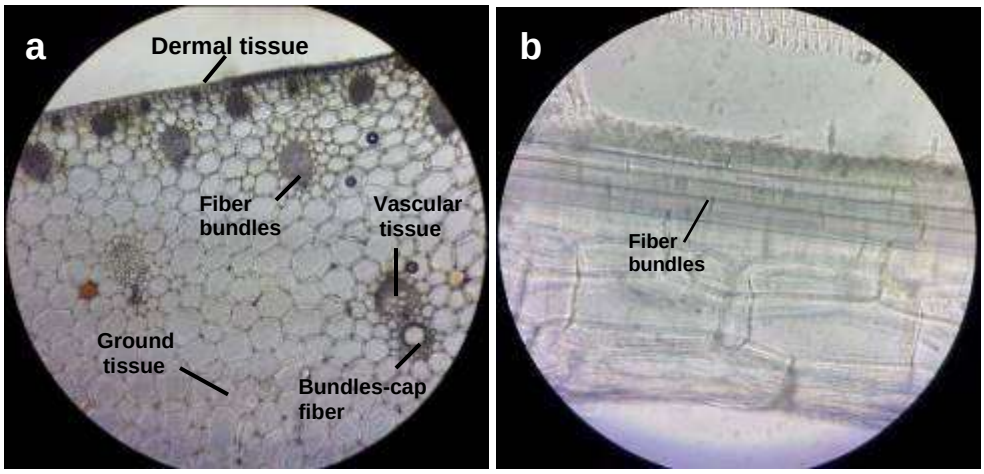

Fig. 2. (a) Transverse section of a banana pseudo-stem sheath showing fiber bundles and vascular bundles (polarized light macrograph; 100×). (b) Longitudinal view of banana pseudo-stem sheath (polarized light macrograph; 400×)

Digital photographs of BPSFs extracted using the three methods are shown in Fig. 3. Mechanically obtained fibers were light brown and had a diameter of 0.056 to 0.143 mm (Fig. 3a). Many short fibers were observed as a result of mechanical scraping damage, and numerous non-cellulosic matter were present on the fiber surface. Chemically extracted BPSFs were white and clean (Fig. 3b), and they consisted of a large number of fine microfibrils with a diameter of 0.05 to 0.09 mm. The enzyme-extracted BPSFs were light yellow and had a diameter of 0.14 to 0.18 mm (Fig. 3c). In addition, chemically extracted BPSFs were much softer than mechanically and enzyme-extracted ones.

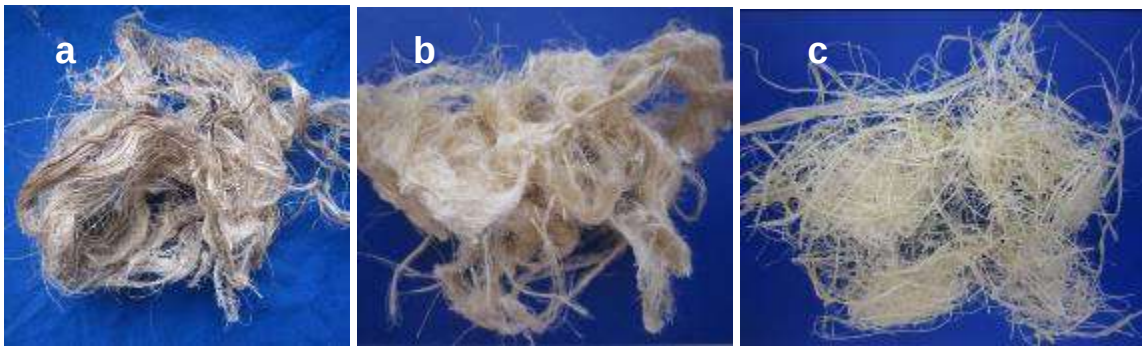

Fig. 3. Photographs of BPSFs: (a) mechanical extraction, (b) chemical extraction, and (c) enzymatic extraction

The morphology of BPSFs is important for predicting the fiber interaction with the polymer matrix in composites (Reddy *et al.* 2013). Longitudinal morphologies of mechanically extracted, chemically extracted, and enzyme-extracted BPSFs were investigated using SEM micrographs (Fig. 4). Mechanically extracted fibers consisted of

waxy substances, hemicelluloses, cellulose, and lignin (Fig. 4a). A small number of surface cracks could also be observed in Fig. 4a. Comparatively, fibers extracted by chemical and enzyme-based methods had a cleaner, rougher, and fibrillated fiber surface because of dissolution of the waxy substances, hemicelluloses, and lignin using alkali chemical treatment. Impurity-free fibers exhibited noticeable separation, as shown in Figs. 4b and 4c. Table 1 shows that the tensile strength of the chemically extracted BPSFs and enzyme-extracted BPSFs was greater than that of the mechanically extracted ones. This finding might be because of the increase in the degree of orderly arrangement of the cellulose and lignin structure present in the fiber (Rout *et al*. 2001). The presence of hemicelluloses and other waxy substances reduced the strength of the fiber because of its brittle and amorphous nature.

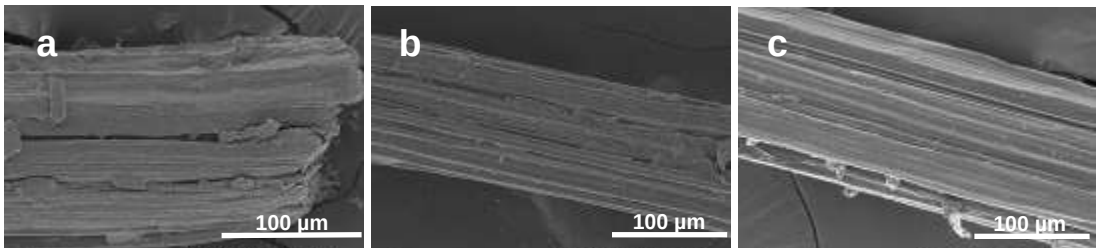

Fig. 4. Longitudinal-view SEM images of BPSFs: (a) mechanical extraction, (b) chemical extraction, and (c) enzymatic extraction.

The fracture morphology was studied in an effort to determine the mode of failure. Tensile fractures can be segregated into brittle and ductile fractures. The principal difference between brittle and ductile fractures is the plastic deformation that takes place in ductile materials before fracture occurs, whereas brittle materials show no or little plastic deformation (Hossain *et al.* 2014). The fracture surface of broken fibers was observed under SEM. Representative SEM images of the BPSF cross-section are presented in Fig. 5. Cross-sections were obtained by axial forces and used to study internal structure behavior. Such images might provide a better understanding of brittle fracture by analyzing each fiber behavior (Osorio *et al.* 2012). In terms of mechanical extraction, tuberous fibers were observed with an inner diameter of about 30 µm, as shown in Fig. 5a.

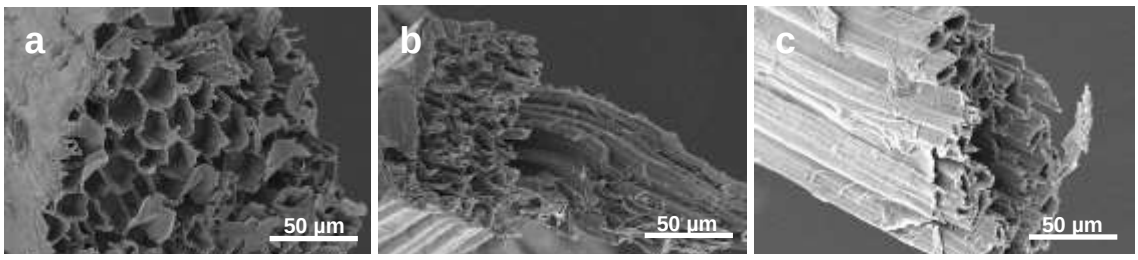

Fig. 5. Cross-section SEM images of BPSFs: (a) mechanical extraction, (b) chemical extraction, and (c) enzymatic extraction.

Based on the SEM micrographs, the fracture surface was smooth at the fracture zone. No tuberous structure was found for chemically extracted fibers in Fig. 5b. However, parts of specimens were found in which fibers were visibly pulled out in the

proximity of the interface of fibers. These findings revealed the onset of ductile fracture as these surfaces were relatively rough compared with the mechanically extracted ones, indicating a higher energy absorption ability of the fiber, which enhanced tensile properties. Enzyme-extracted fibers displayed a tuberous structure, as shown in Fig. 5c. These fibers should also be brittle based on the fracture on the cross-section.

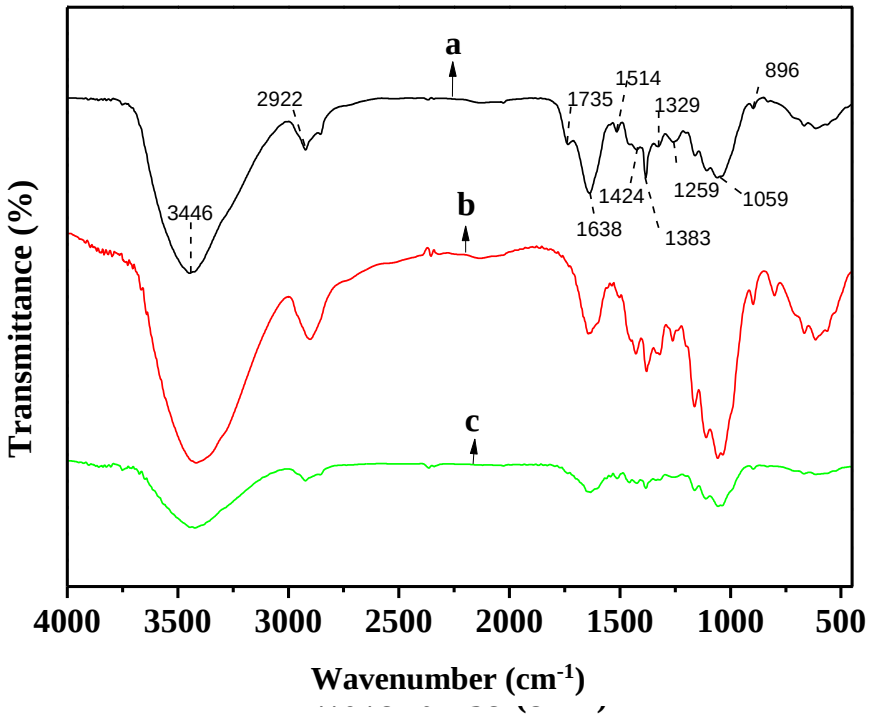

Fig. 6. FTIR spectra of BPSFs: (a) mechanical extraction, (b) chemical extraction, and (c) enzymatic extraction

The FTIR spectra of BPSFs are presented in Fig. 6. The broad band at 3446 cm^{-1} corresponded to the O-H stretching vibration of cellulose (Reddy *et al.* 2013). The band at 2921 cm^{-1} corresponded to the C-H stretching vibration. The band at 1735 cm^{-1} was associated with the absorption of C=O stretching of ester and carboxyl groups in hemicelluloses in mechanically extracted BPSFs. However, the band disappeared in the spectra for chemically and enzyme-extracted BPSFs, because of the removal of hemicelluloses (Sreenivasan *et al.* 2011b; Reddy *et al.* 2013; Buana *et al.* 2013). The FTIR spectra confirmed that chemical and enzymatic treatments could effectively reduce the hemicellulose and lignin contents. The bands at 1638, 1514, 1424, and 1383 cm^{-1} corresponded to the aromatic skeletal vibrations, and they were associated with C-O stretching in lignin. The band at 1259 cm^{-1} was attributed to C-O stretching in hemicelluloses. The strong band at 1059 cm^{-1} represented C-O stretching in cellulose and hemicelluloses. The small band at 896 cm^{-1} was attributed to the β-glucosidic linkages between the sugar units in hemicelluloses and cellulose (Reddy *et al.* 2013).

The XRD patterns of samples are displayed in Fig. 7. Two main peaks at 16° and 22.5° were assigned to the (2 0 0) and (1 1 0) crystallographic planes, respectively, contributed by both the amorphous and crystalline fractions (Sreenivasan *et al.* 2011a). The crystallinity index (CI) was calculated using Eq. 1 (Sreenivasan *et al.* 2011b),

$$CI = \frac{H_{22.5} - H_{18.5}}{H_{22.5}} \quad (1)$$

where $H_{22.5}$ is the peak height at $2\theta = 22.5°$, which is caused by the contribution of both the amorphous and crystalline fractions. $H_{18.5}$ is the diffracted intensity at $2\theta = 18.5°$, and it is attributed to the amorphous fraction. The *CI* values were 56.6%, 61.2%, and 60.2% for mechanically, chemically, and enzyme-extracted BPSFs, respectively. The higher *CI* values of chemically and enzyme-extracted BPSFs possibly resulted from the removal of amorphous hemicelluloses and enhancement of crystalline fraction.

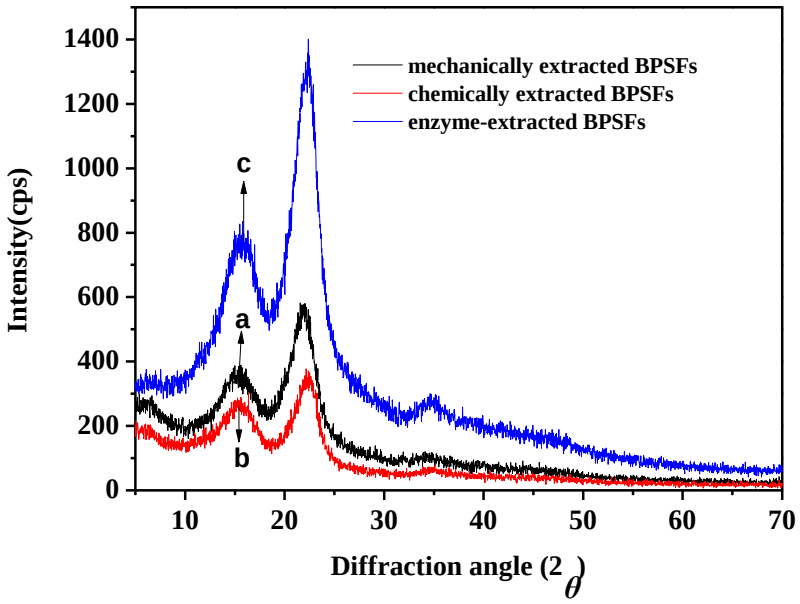

Fig. 7. XRD patterns of BPSFs: (a) mechanical extraction, (b) chemical extraction, and (c) enzymatic extraction

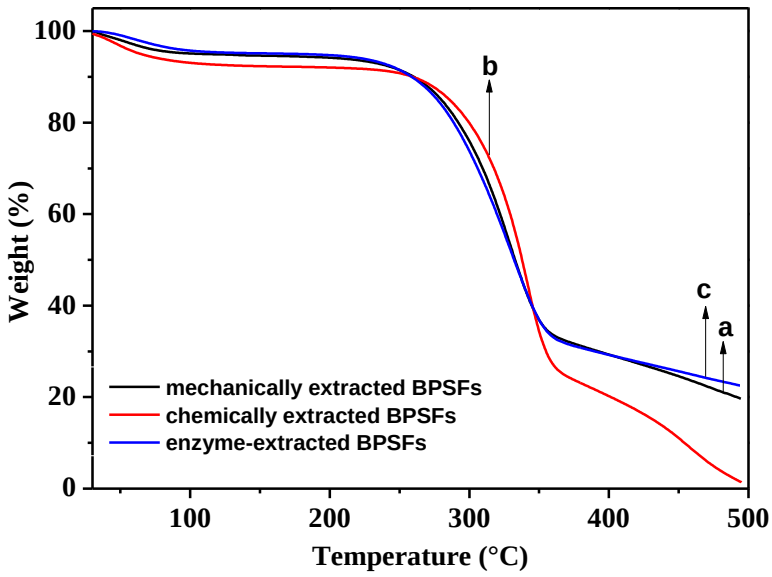

Fig. 8. TGA curves of BPSFs: (a) mechanical extraction, (b) chemical extraction, and (c) enzymatic extraction

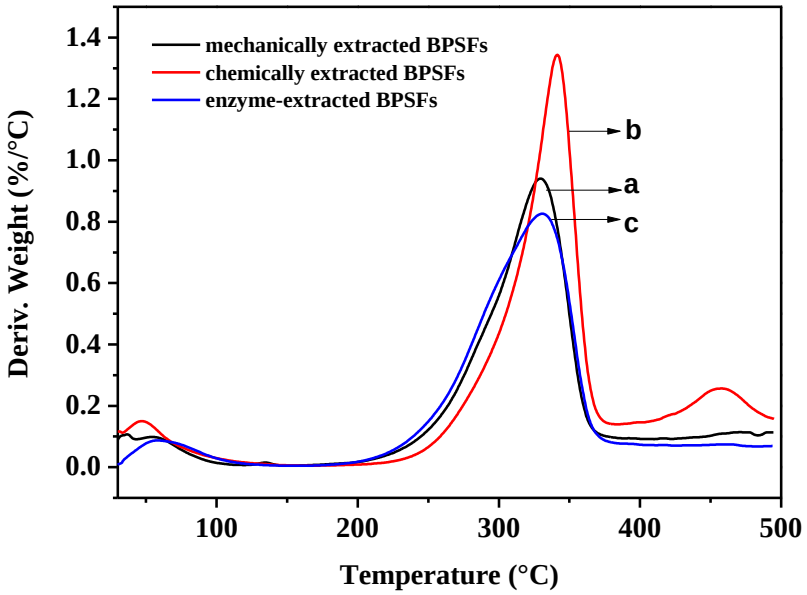

Fig. 9. DTG curves of BPSFs: (a) mechanical extraction, (b) chemical extraction, and (c) enzymatic extraction

The TG analyses of fibers are shown in Fig. 8. The fibers had three main weight loss regions for three samples. The initial small weight loss, which was caused by the loss of free water adsorbed in the fibers, occurred in a range of 45 °C to 100 °C. The weight loss percentage was about 5% to 8%. The second weight loss in the temperature range of 250 °C to 370 °C was 65% to 71%, and it was mainly contributed to depolymerization of hemicelluloses and thermal decomposition of α-cellulose (Deepa *et al.* 2011; Buana *et al.* 2013). The third weight loss region was caused by the degradation of non-cellulosic substances, such as lignin. Given that lignin is the most difficult constituent to decompose, its decomposition usually covers the whole temperature range of 200 °C to 500 °C (Reddy *et al.* 2013). However, the decomposition temperature of chemically extracted fibers was about 10 °C higher than those obtained by the other two methods (Fig. 9). The relative amount of cellulose increased and the thermal properties were enhanced as the chemically extracted fibers were mainly composed of cellulose (Hossain *et al.* 2014).

The strength, modulus, and elongation of BPSFs are summarized in Table 1. The tensile strength of chemically obtained BPSFs was higher than that of those extracted using the other two methods. The enhancement in tensile strength might be attributed to the lack of ductility of the fibers, which resulted from relatively complete removal of hemicelluloses and lignin. Another possible reason for the high tensile strength of chemically obtained BSFs is the non-tuberous structure of the fibers, where the fibrils rearranged themselves in a more compact manner, resulting in close fiber packing (Reddy *et al.* 2013). The tensile properties of BPSFs decreased as a consequence of enzymatic extraction, relative to the important changes registered by the fibers. The enzyme attacks the fibers, resulting in the disappearance of the fiber cell walls and loss of their compactness, which leads to a decrease in the tensile properties of fibers (Ganan *et al.* 2004; Dai *et al*. 2011). Moreover, the corresponding elongation of BPSFs also displayed the same trend. Greater elongation of chemically obtained BPSFs was also observed in the SEM image (Fig. 5b). Mechanically and enzyme-extracted BPSFs showed a tendency toward dominantly brittle fracture, and they were characterized by a linear relationship

between deformation and stress until failure, without noticeable plastic deformation. However, chemically extracted BPSFs experienced a combination of ductile and brittle deformation (Buana *et al.* 2013). Mechanically extracted BPSFs had the highest Young's modulus. SEM features were satisfactorily correlated with the tensile properties of the BPSFs extracted using different methods.

The high cellulose content of BPSFs could be obtained by mechanical, chemical, and enzyme-based methods. Chemically extracted BPSFs exhibited relatively better thermal and tensile properties. The thermal and tensile properties of BPSFs extracted by mechanical methods were slightly lower than the chemically extracted ones. So, at least in terms of chemical use, they can be regarded as being environmentally friendly. Thus, the mechanical methods have potential application value. When the composite industry looks for light reinforcements for their products, they have the option to choose BPSFs. The potential functions of the tested BPSFs that are commonly available depend on their tensile properties. Mechanically extracted BPSFs should be reinforcing fibers in lightweight high-performance composites because of their higher tensile strength (Defoirdt *et al*. 2010).

Table 1. Tensile Properties of BPSFs Obtained Using Mechanical, Chemical, and Enzymatic Extraction Methods (mean±standard deviation)

BPSFs	Diameter (mm)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation (%)
Mechanical extraction	0.095±0.028	210±86	26.86±11.84	0.8±0.3
Chemical extraction	0.075±0.015	333±92	22.56±7.75	1.6±0.5
Enzymatic extraction	0.125±0.013	235±94	16.36±5.51	1.4±0.5

CONCLUSIONS

1. In this research, banana pseudo-stem fibers (BPSFs) were extracted using mechanical, chemical, and enzymatic methods. The polarized light micrographs revealed that the banana pseudo-stem sheath contained structural and arch fibers.
2. The FTIR spectra indicated that hemicelluloses and lignin were greatly reduced after chemical or enzymatic treatment.
3. Compared with mechanically extracted fibers, chemically, and enzyme-extracted BPSFs demonstrated a finer fiber bundle with rougher surface. Among the fibers, the chemically extracted ones exhibited a combination of ductile and brittle failure, whereas the fibers obtained by the other two methods underwent brittle failure. Moreover, thermogravimetric tests suggested that chemically extracted fibers had a higher thermal stability.
4. The tensile behavior of mechanically extracted, chemically extracted, and enzyme-treated BPSFs was investigated. Their tensile properties were correlated with their morphology.

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