

# Characterization of banana, sugarcane bagasse and sponge gourd fibers of Brazil

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## ABSTRACT

In recent times, increasing attention has been paid to the use of renewable resources particularly of plant origin keeping in view the ecological concerns, renewability and many governments passing laws for the use of such materials. On the other hand, despite abundant availability of lignocellulosic materials in Brazil, very few attempts have been made about their utilization, probably due to lack of sufficient structure/property data. Systematic studies to know their properties and morphology may bridge this gap while leading to value addition to these natural materials. Chemical composition, X-ray powder diffraction, and morphological studies and thermal behavior aspects in respect of banana, sugarcane bagasse sponge gourd fibers of Brazilian origin are presented. Chemical compositions of the three fibers are found to be different than those reported earlier. X-ray diffraction patterns of these three fibers exhibit mainly cellulose type I structure with the crystallinity indices of 39%, 48% and 50% respectively for these fibers. Morphological studies of the fibers revealed different sizes and arrangement of cells. Thermal stability of all the fibers is found to be around 200 °C. Decomposition of both cellulose and hemicelluloses in the fibers takes place at 300 °C and above, while the degradation of fibers takes place above 400 °C. These data may help finding new uses for these fibers.

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## 1. Introduction

It is well known that plant fibers also known as lignocellulosic fibers (LC) have been one of the attractive fillers for different types of polymers including rubbers as well as for ceramic matrices due to some of their unique characteristics unparalleled with any other reinforcing/filler materials. They include biodegradability contributing to a healthy ecosystem, low cost and higher stiffness to those of glass fibers, which would be attractive for the applications requiring stiffness dominant ones such as the automotive applications (Rijswijk and Brouwer, 2002). Additional motivation for their use in composites to receive greater attention in recent times is the increasing ecological considerations with many governments such as European Union passing laws for the use of about 95% recyclable materials with about 85% renewable materials in them in all new automobiles to achieve the “end of life” required by 2015 (Netravali and Chabba, 2003; Peijs, 2003). In addition, use of their composites have established comparable performance with those of glass fiber composites with possibility for their use as structural components as well (Bledzki and Gassan, 1999; Burgueno et al., 2004; Corbiere-Nicollier et al., 2001; Dweib et al., 2004; Holbery and Houston,

2006; Joshi et al., 2004; Marsh, 2003; Mehta et al., 2005; Mohanty et al., 2002; Netravali and Chabba, 2003; Peijs, 2003; Rijswijk and Brouwer, 2002; Schloesser, 2004; Schuh and Gayer, 2000; Suddell et al., 2002; Wambua et al., 2003). When such materials are used in composites, developing countries, which produce these, become part of global composite industry as developer and manufacturer leading to increased revenues and creation of jobs (Rijswijk and Brouwer, 2002).

In view of the above, many attempts have been made to characterize the lignocellulosic fibers either individually (Chand et al., 1984; Kulkarni et al., 1981; Mukherjee and Satyanarayana, 1984; Satyanarayana et al., 1986), or as part of their composites research (Jacob et al., 2004). Many of these can be found in various periodic reviews published (Bledzki and Gassan, 1999; Eichhorn et al., 2001; Jacob et al., 2004; John and Thomas, 2008; Satyanarayana et al., 2007) or as chapters in some books (Rowell et al., 2000; Satyanarayana, 2007; Satyanarayana and Wypych, 2007; Satyanarayana et al., 2009). While a large amount of data on their structure and properties is available on a variety of lignocellulosic fibers including various Brazilian fibers, comparative studies of Brazilian fibers and their polymeric composites have been reported only recently (Satyanarayana, 2007; Satyanarayana et al., 2007, 2009). But, the search is on for finding new resources of such fibers (Beakou et al., 2008; D'Almeida et al., 2006; Elenga et al., 2009; Ghali et al., 2006).

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**Table 1a**

Reported chemical composition of banana, bagasse and sponge gourd fibers of Brazil.

Constituent (%)	Moisture	Ash	Holocellulose	Cellulose	Hemi-celluloses	Klasson lignin	References
Banana	10–12	1.2	66–73	60–65	6–8	5–10	Nolasco et al. (1998)
Sugarcane bagasse	–	1–4	50–84	32–55	27–32	19–25	Almeida et al. (2000), Anselmo and Badr (2004), Hoareau et al. (2004)
Sponge gourd	–	0.4–3	82	62	20	11.2	Almeida et al. (2000), Tanobe et al. (2005)

**Table 1b**

Reported tensile properties of banana, bagasse and sponge gourd fibers of Brazil.

Property/reinforcement	Young's Modulus (GPa)	Tensile strength (MPa)	Elongation (%)	Crystallinity (%)	Reference
Banana	27–32	700–800, 711–789	2.5–3.7	–	Nolasco et al. (1998), Satyanarayana and Wypych (2007)
Sugarcane bagasse	27.1	222	1.1	47	Anselmo and Badr (2004), Satyanarayana and Wypych (2007)
Sponge gourd <sup>a</sup>	–	–	–	59.1	Tanobe et al. (2005)

<sup>a</sup> Since this material is available in the mat form, no tensile property data of fiber is available.

In the case of three fibers chosen in this study, there were only a couple of papers till recently reporting on the properties and utilization of Brazilian banana fiber (Nolasco et al., 1998; Savastano et al., 2000). On the other hand, though a number of papers have been published on sugarcane bagasse and sponge gourd fibers of Brazil origin, most of the publications have reported their characterization as part of composite development (Almeida et al., 2000; Anselmo and Badr, 2004; Boynard and Almeida, 1999, 2000; Boynard et al., 2003; Hoareau et al.,

2004; Satyanarayana, 2007; Satyanarayana et al., 2007, 2009; Tanobe et al., 2003, 2004, 2005; Trindade et al., 2004) and thus not exclusively as part of any systematic studies on Brazilian fibers. Reported chemical composition and strength properties on banana and sugarcane bagasse fibers of Brazil are listed in Tables 1a and 1b.

Taking the above into account, the authors have taken up systematic studies on various lignocellulosic fibers of Brazil including development of composites using these fibers (Guimarães et al.,



**Fig. 1.** Photographs of sources of (a) banana, (b) sugarcane bagasse and (c) sponge gourd fibers.



2006a,b; Pinto, 2007; Satyanarayana et al., 2007, 2009; Tomczak et al., 2007a,b).

This paper presents chemical composition, X-ray diffraction, morphological and thermal behavior aspects in respect of three different types of lignocellulosic fibers of Brazil namely, fiber from pseudostem or bark (banana), fruit fiber (sponge gourd) and fiber from grass/agro waste (sugarcane bagasse). It is hoped that such attempts would lead to the better utilization of these LC fibers through composite technology leading to a whole spectrum of opportunities and challenges for many of the developing countries such as Brazil in particular and the world in general keeping in view one of the long-term objectives of such attempts being to fulfil the socio-economic responsibilities of the State.

## 2. Experimental aspects

### 2.1. Materials

Three types of fibers, namely banana fibers (*Musa sapientum*) obtained from the pseudostem of the plant, sugarcane (*Saccharum officinarum*) bagasse fibers obtained from a near by alcohol plant production in the city of Curitiba, capital of Paraná (Brazil) and Brazil sponge gourd (*Luffa cylindrica*) fibers obtained from the local market were used in this study. Banana fibers were extracted from the pseudostem (average diameter of 150 mm) of the banana plant. The leaf sheaths from the pseudostem were cut in parts of about 300 mm length using long knife and then dried outdoors exposing the fibers to the sun for about a week. They were then dried in a hot air oven at 65 °C until constant mass was observed. They were then further cut in a knife mill (Marconi – MA280) to get fibers of 3–5 mm length. The banana sheaths used to produce fibers used in this investigation are shown in Fig. 1(a). The banana fibers were cleaned with 5% sodium hypochlorite before they were maintained at 100 °C for 1 h in an autoclave followed by drying at 65 °C for 72 h to maintain constant moisture content.

Fig. 1(b) shows the source of sugarcane bagasse fiber (here after called as bagasse) used in the present study. No fiber preparation was required in this case since the bagasse fiber received was in the fiber form (not extracted from the stem/stalk as described for banana fiber), except cutting them to random size using a suitable cutter and conditioning them by keeping the fibers for 12 h at 65 °C to maintain constant moisture content.

Fig. 1(c) shows the source of sponge gourd (*L. cylindrica*) fibers, here after referred in the paper as “sponge gourd”. Details of obtaining these fibers from the fruit were described elsewhere (Tanobe et al., 2005) and hence not described here since the fibers were obtained from the market. These fibers are multi-directionally arranged and entangled around the beehive-like structure of the sponge gourd fruit (15–25 cm long) nucleus. These fibers were cleaned with 5% sodium hypochlorite before they were maintained at 65 °C for 72 h to maintain constant moisture content. The chemical treatments followed by thermal treatments and drying of both banana and sponge gourd fibers was necessitated for their use in the preparation of their composites in subsequent studies since such treatments made the surface of the fibers brighter in addition to making them more amenable for adhesion with the polymer matrix.

### 2.2. Methods

Detailed chemical composition of all the fibers was determined as explained below using oven dried fibers: Moisture content of all the three fibers was determined according to ABNT NBR 9656 (Associação Brasileira de Normas Técnicas, the Brazilian association for technical standards), which consists of determining the percentage difference between the initial weight of the sample (1.0 g) and after 4 h drying at 105 °C. Ash content was measured by considering the percent difference between the initial weight of dried fiber of the sample and that after calcination for 4 h at 800 °C. Klason lignin content was evaluated following the TAPPI T13M-54 method, which is based on isolation of lignin after polysaccharide hydrolysis by concentrated sulfuric acid (72%). The samples (1.0 g, previously dried) were macerated using 72% H<sub>2</sub>SO<sub>4</sub> (15 mL) at room temperature and kept under these conditions for 24 h. After that, the material impregnated with sulfuric acid was transferred to an appropriate vessel, and 560 mL of distilled water was added. The system was then kept under reflux for 4 h. The remaining solid (insoluble lignin and ash) was filtered (sintered glass funnel, number 4, previously weighed), dried (105 °C, 24 h), and then weighed. The acid-insoluble lignin content was calculated by considering the weight difference between the initial sample (fibers) and that of the filtered solid and subtracting the ash content from the weight of this last sample. The Klason soluble lignin was determined following the same method described above. Absorbance values were measured at 280 (A<sub>280</sub>) and 215 (A<sub>215</sub>) nm. The concentration [C (g/L)] of Klason soluble lignin was determined using Eq (1), based on Lambert Beer law (Kerr and Goring, 1975):

$$C(\text{g/L}) = \frac{4.53 \times A_{215} - A_{280}}{300} \quad (1)$$

**Table 2**

Chemical composition of banana, bagasse and sponge gourd fibers.

Component (%)	Banana	Bagasse	Sponge gourd
Moisture	8.57 ± 0.19	9.21 ± 0.01	4.79 ± 0.02
Ash	4.14 ± 0.92	2.75 ± 0.19	0.66 ± 0.03
Holocellulose	50.92 ± 0.34	71.39 ± 0.60	84.03 ± 0.03
Cellulose	50.15 ± 1.09	54.87 ± 0.53	66.59 ± 0.61
Hemicelluloses	0.77 ± 0.58	16.52 ± 0.56	17.44 ± 0.21
Klason lignin	17.44 ± 0.19	23.33 ± 0.02	15.46 ± 0.02
Crystallinity (%)	39	48	50

The holocellulose content was determined according to TAPPI T19m-54, which consists of a selective degradation of the lignin polymer. Sodium hypochlorite (2.5 g) and glacial acetic acid (1 mL) were added to an aqueous suspension (120 mL put in an Erlenmeyer flask) of previously dried and milled LC fibers (3 g). The mixture was covered with an inverted Erlenmeyer flask and kept at 70 °C, under magnetic stirring for 1 h. This sequence was repeated twice. Then, after 3 h (3 × 1 h), the system was cooled to nearly 5 °C, and the holocellulose was filtered and washed exhaustively with water and methanol and then dried at 40 °C, in a vacuum oven, until constant weight was observed. For the determination of α-cellulose content, sodium hydroxide solution (10 mL, 17.5%) was added to cellulose (1.0 g) at room temperature. Then, the mixture was grounded for 8 min, and sodium hydroxide (10 mL, 17.5%) was added to the mixture, which was kept at rest for 20 min. If the alkaline solution is kept at room temperature longer than 20 min, as usually described for wood, cellulose can also degrade, leading to erroneous results for hemicellulose content. Then, water (40 mL) was added, and the solid residue was filtered and washed exhaustively with aqueous acetic acid and water. This remaining solid, considered α-cellulose, was dried at 40 °C, in a vacuum oven, until constant weight was obtained. An average of three repetitions was taken for all mentioned analyses. The hemicellulose content was obtained by subtracting the α-cellulose part from the holocellulose content.

Fourier transform infra-red spectroscopy (FTIR) studies on banana, bagasse and sponge gourd fibers were carried out dispersing the powdered samples of the fibers on KBr pellets and using an Excalibur Bio-Rad spectrophotometer, Model FTS 3500 GX with a resolution of 2 cm<sup>-1</sup> and accumulation of 32 scans.

The crystallinity index, *I<sub>cr</sub>*, was determined by X-ray powder diffraction (XRPD) using a URD-6, VEB CARL ZEISS-JENA diffractometer operating at 40 kV, 20 mA and CuK<sub>α</sub> radiation (λ = 1.5406 Å). The crystallinity index was calculated using the Buschle-Diller and Zeronian equation (Buschle-Diller and Zeronian, 1992) (Eq. (2)):

$$I_{cr} = 1 - \frac{I_{min}}{I_{max}} \quad (2)$$

where *I<sub>min</sub>* is the intensity at the minimum of the crystalline peak (18° < 2θ < 19°) and *I<sub>max</sub>* is the intensity at its maximum (22° < 2θ < 23°).

Morphology of all the fibers was examined using an optical and a scanning electron microscope. For optical microscopic study, the specimens were prepared using a mixture of polyester resin with 2% catalyst and 2% initiator poured into a mold. A bunch of fibers was kept vertically in the resin and allowed the resin to set. After curing, the samples were ground successively using silicon carbide papers of grit size ranging from 220 to 600 meshes. Then the specimens were polished on a silvyl cloth mounted on disc polisher using diamond paste (6, 3 and 0.25 μm size). Then, the polished specimens were observed in reflected light using a LEICA DMRX optical microscope.

For scanning electron microscopic study, the fiber samples were mounted on brass studs and after giving gold coating, they were observed under a JEOL scanning electron microscope model JSM 6360 LV at 15 keV and current of 0.65 A.

Thermogravimetric (TGA) studies of all the three fibers were carried out using a Shimadzu model TGA-50TA. Samples of approximately 8 mg were placed in appropriate pans and heated from 25 to 800 °C at 10 °C min<sup>-1</sup>, under dynamic flow of air (20 mL min<sup>-1</sup>). Also, differential scanning calorimetric (DSC) analyses of the fibers were carried out using a Shimadzu DSC model 50. Samples of approximately 6.0 mg were placed in appropriate sealed pans and heated from 25 to 500 °C, at 10 °C min<sup>-1</sup>, under dynamic flow of air (20 mL min<sup>-1</sup>).

## 3. Results and discussions

### 3.1. Chemical composition of fibers

Table 2 lists the chemical composition of Brazilian fibers used in this study. It can be seen that all the values of present study are different from those reported earlier (Table 1a). An analysis of the data reveals that in the case of banana fibers, moisture content is lower in the present case, while ash content and Klason lignin content are higher than the reported values. Lower moisture content may be due to the test procedure used in the present study as explained

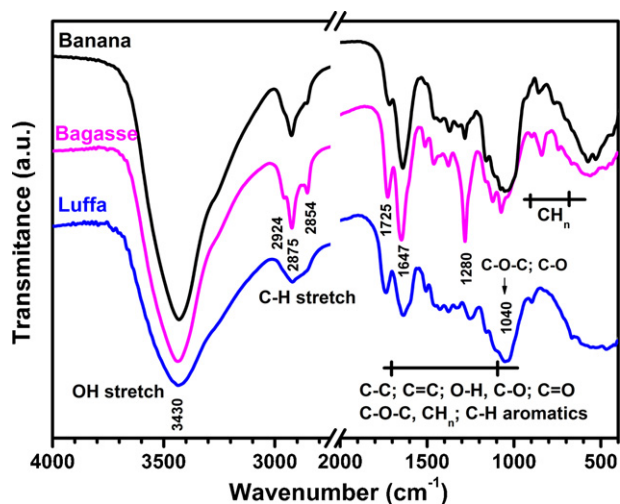


Fig. 2. FTIR spectra of banana, bagasse and sponge gourd fibers.

above (Section 2.2) and the ambient temperature of testing. On the other hand, values of holocellulose, cellulose and hemicellulose are lower than the reported values elsewhere.

In the case of bagasse fibers, it may be noted that the sum of the errors is about  $\pm 2.0$  and while the ash content is low, other constituents are within the range of reported values by others.

In the case of sponge gourd fibers, it can be seen that except for hemicellulose content, all other values are slightly higher than those reported for this fiber by others although the sum of errors being within  $\pm 1.0$ . Also, the cellulose content is similar to that reported for sisal, jute, hemp and abaca (Manila) while the ash content is similar to that reported for agave, bagasse and abaca (Nolasco et al., 1998).

The above observations and the discrepancies between the earlier reported values and the present study is understood as due to the fact that the chemical composition of lignocellulosic fibers depends on various factors such as species, variety, type of soil used, weather conditions, part from which the fibers are extracted, age of the plant, etc. (Rowell et al., 2000; Satyanarayana and Wypych, 2007). Also, it may be noted that the chemical composition of lignocellulosic fibers reported include cellulose, lignin, hemicellulose and ash content, which are normally do not add up to 100% since most of these values are not normalized with respect to all the constituents. Accordingly, total percentage of constituents given for banana fiber in the present study adds up to 80% only, similar to

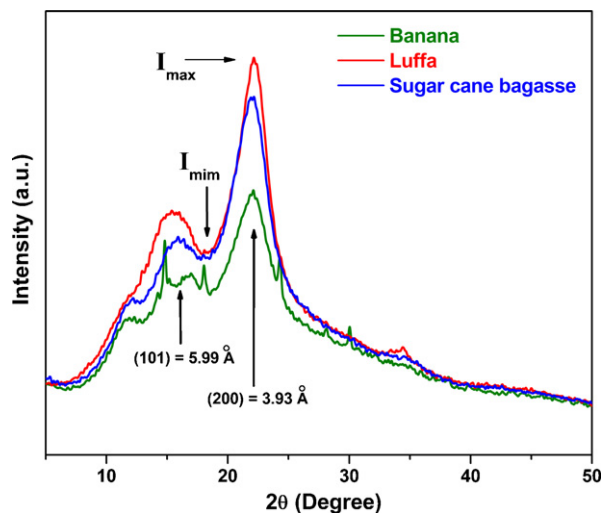


Fig. 3. X-ray diffraction patterns of fibers of banana, bagasse and sponge gourd.

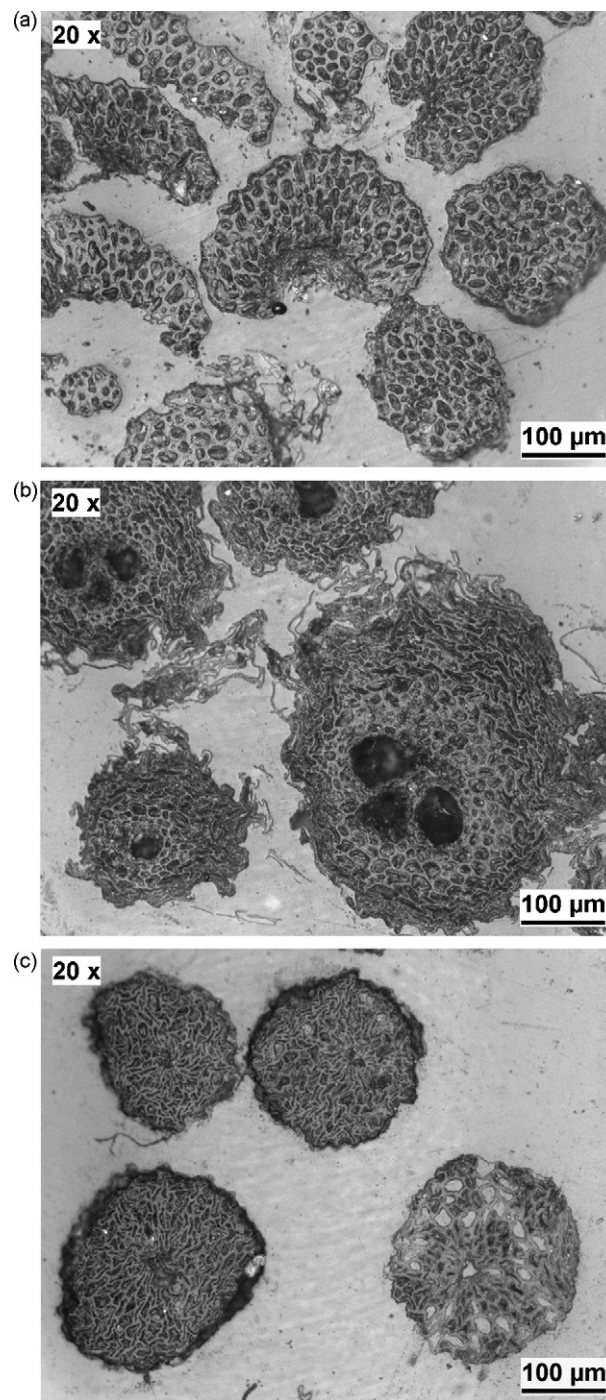
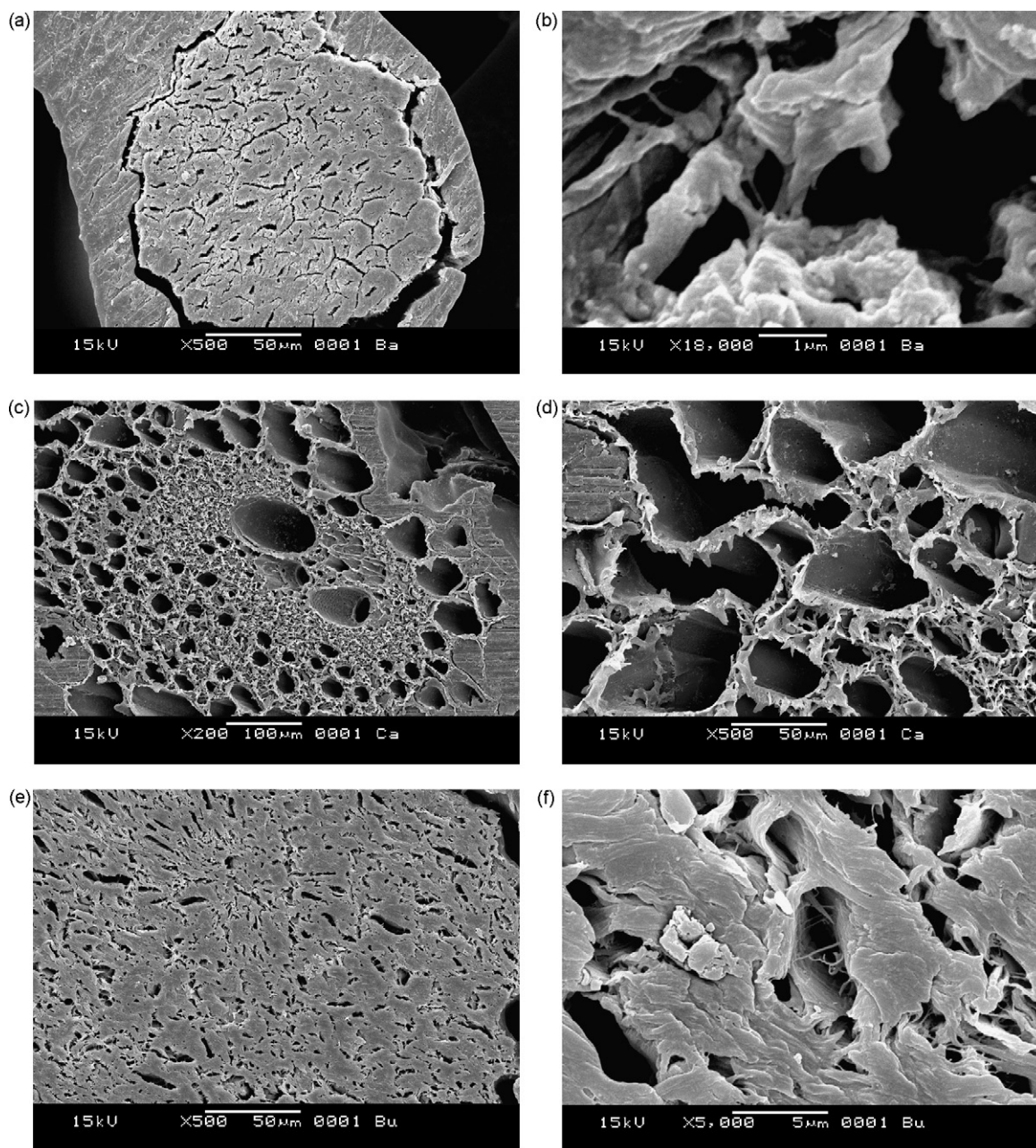


Fig. 4. Optical micrographs of cross-sections of fibers: (a) banana, (b) bagasse and (c) sponge gourd (All figures are 20 $\times$ , while magnification bars with scale in  $\mu\text{m}$  are given on the photographs).

80–90% reported (Table 1a) for other fibers by various researchers (Rowell et al., 2000; Satyanarayana and Wypych, 2007; Tanobe et al., 2005). This may be due to the experimental conditions including the methodology used in the present study. It is also relevant here to point out (Browning, 1967) that in practice, the sum of all the chemical constituents of lignocellulosic materials adding “exactly to 100% is considered as fortuitous” while such totals reported “in the range of 95–102% is not uncommon”. When the total does not conform to 100%, it could be due to one of the following reasons (Browning, 1967): (i) some loss of protein or polysaccharides during the isolation of cellulose and/or loss of sugars resulted by the





**Fig. 5.** Scanning electron micrographs of cross-sections of fibers (a and b) banana [a: 500 $\times$ ; b: 18,000 $\times$ ]; (c and d) bagasse [c: 200 $\times$ ; d: 500 $\times$ ]; and (e and f) sponge gourd [e: 500 $\times$ ; f: 5000 $\times$ ] (magnification bars with scale in  $\mu\text{m}$  are given on the photographs).

reverse reaction during hydrolysis; (ii) overlapping of some of the constituents such as residuals of lignin in celluloses or ash; (iii) nitrogenous or other materials in the extracts; (iv) presence of some impurities in protein or degradation products of polysaccharide in lignin; and (v) the presence of not quantified waxes/oils and other chemical components.

In the case of banana fiber, as the fibers were extracted in the laboratory itself, mass loss was also determined after drying it in the sun as well as drying in an oven at constant temperature of 85 °C. It was found that after 220 min of drying of this fiber, there was a 3% loss in water content.

### 3.2. Fourier transform infra-red (FTIR) spectroscopic studies

Fig. 2 shows the FTIR spectra of all the three fibers. As the main components of the fibers are cellulose based materials

(Tables 1a and 2), the observed FTIR spectra can be attributed mainly to these components. Identification of these spectral lines reveals lines representing the hydrogen bonded stretching bands of OH groups in the region of 3400  $\text{cm}^{-1}$  and in the region of 1030–1150  $\text{cm}^{-1}$ , the absorptions can be attributed mainly to the carbohydrates (cellulose and lignin), including C–O–C and C–O stretch (primary and secondary hydroxide groups) and bonds belonging to the glucoside linkage and possibly to lignin since lignin has hydroxyphenyl, guaiacyl and syringyl groups, which are aromatic compounds. The bands in the region of 1725  $\text{cm}^{-1}$  can be attributed to unconjugated C=O stretching (vibration of aliphatic carboxylic acids and ketones, mainly due to hemicellulose groups), and that near 1650  $\text{cm}^{-1}$  can be assigned to conjugated carbonyl present in typical lignin groups, although this can also be assigned to proteins, the amount of protein is negligible and normally, they produce a

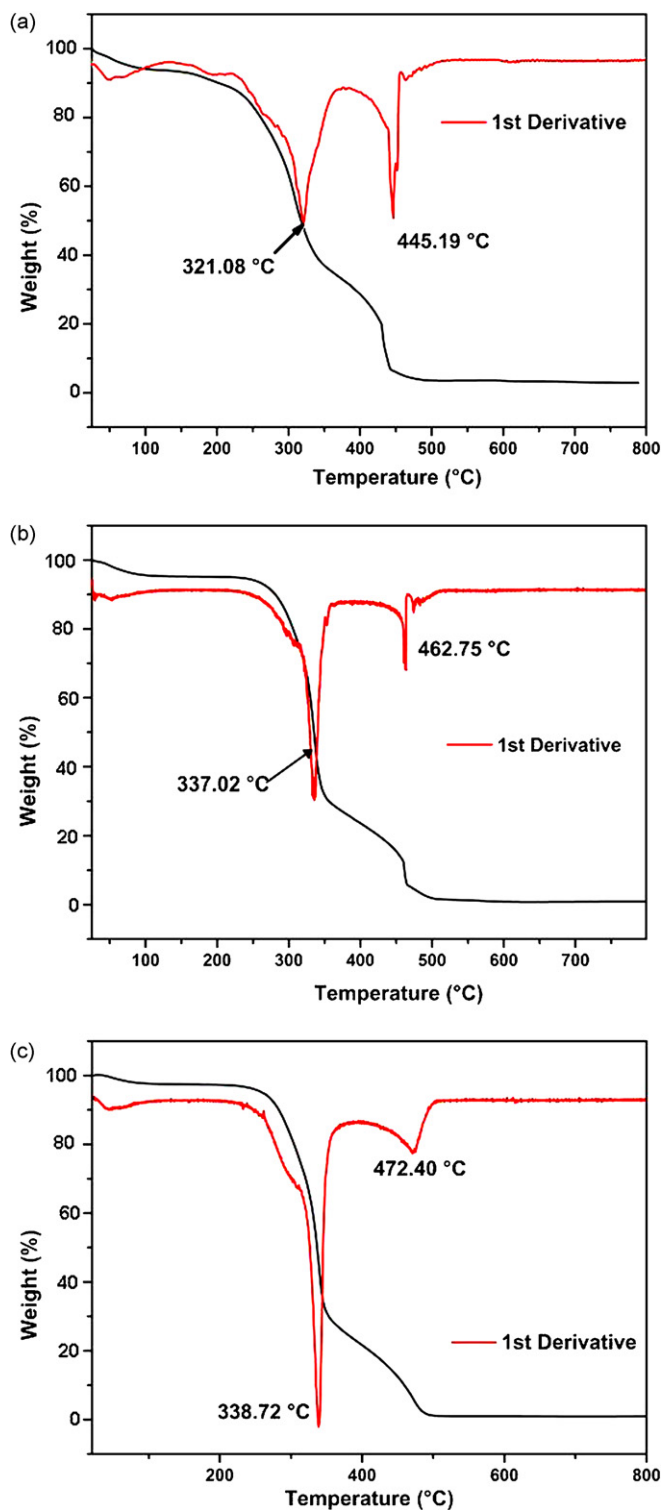


Fig. 6. TGA/DTG curves: (a) banana fiber, (b) bagasse fiber and (c) sponge gourd fiber. Dynamic flow of air,  $10^{\circ}\text{C min}^{-1}$ ,  $20\text{ mL min}^{-1}$ .

broad band if there is no presence of water due to amino groups.

In addition, the presence of moisture may be contributing to the deformation of water molecules near  $1650\text{ cm}^{-1}$  and also the contribution for the intensity of the broad band in the region of  $3400\text{ cm}^{-1}$ .  $\text{C}=\text{C}$  of aromatic skeletal vibrations in lignin appears in the region of  $1500\text{--}1600\text{ cm}^{-1}$  and  $\text{C-H}$  symmetric and asymmetric stretching bands in the region of  $2900\text{ cm}^{-1}$ . The spectrum

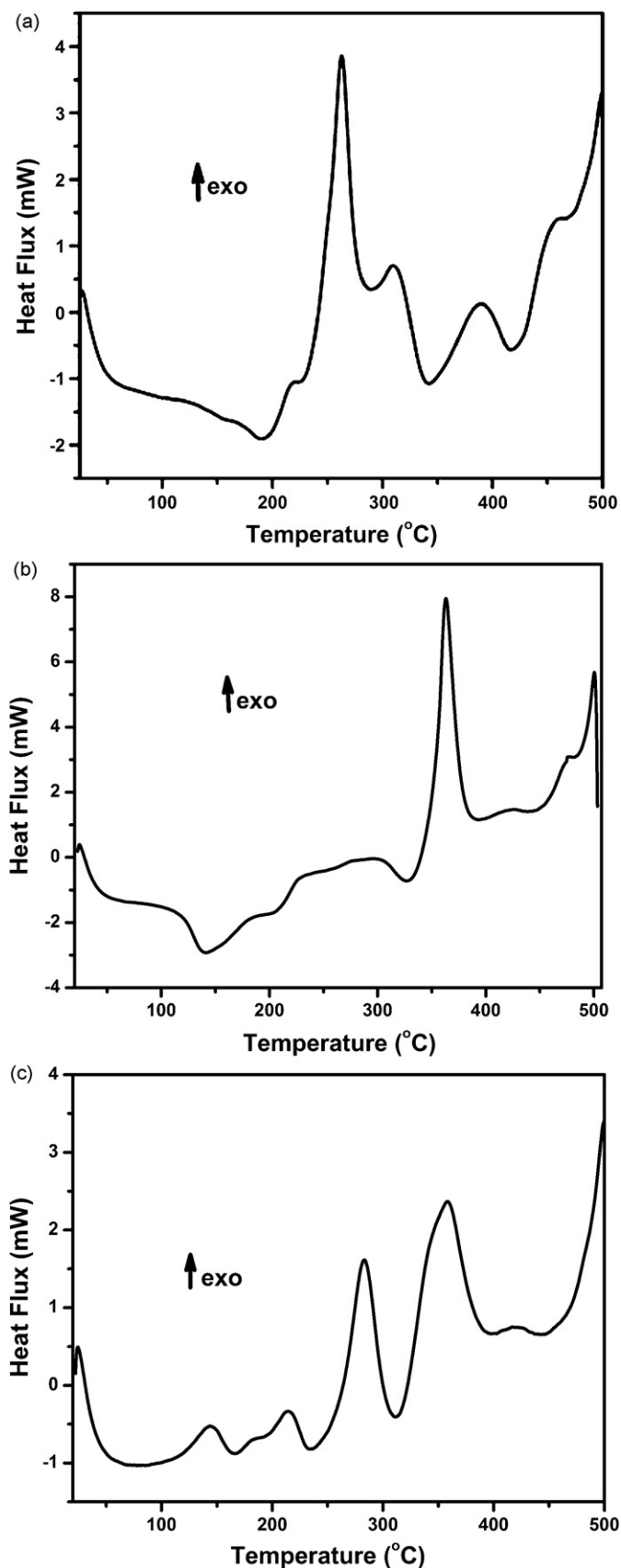


Fig. 7. DSC curves: (a) banana fiber, (b) bagasse fibers and (c) sponge gourd fibers. Dynamic flow of air,  $10^{\circ}\text{C min}^{-1}$ ,  $20\text{ mL min}^{-1}$ .

for banana fiber compares well with the earlier reported values for fibers from elsewhere (Bilba and Ouensanga, 1996; Bilba et al., 2007). This also applies to the spectrum of sponge gourd fibers (Tanobe et al., 2005).

### 3.3. X-ray powder diffraction (XRPD) studies

Fig. 3 shows the X-ray powder diffraction patterns of the three fibers studied in this work. They are similar to those reported for other lignocellulosic fibers exhibiting mainly the Cellulose I structure, common structure observed in plants, showing X-ray diffraction indexed peaks at  $d=5.99$ ,  $5.33$  and  $3.93$  Å. Banana fibers have also shown narrow diffractions peaks, attributed to an unknown contamination, probably from an inorganic substance. Also, the values of crystallinity calculated using these curves are found to be 39%, 48% and 50% for banana, bagasse and sponge gourd fibers respectively. It can be seen from Table 1 that crystallinity of banana fiber is reported for the first time. Also, the crystallinity value for bagasse is almost identical to that reported elsewhere (Hoareau et al., 2004; Trindade et al., 2004), while that for sponge gourd is slightly lower than reported earlier (Tanobe et al., 2005). This small difference could be due to the methodology used in these studies including the measurement of area in the X-ray diffraction patterns.

### 3.4. Morphology studies of fibers

The dimensions (diameter only since length is not much of consequence for the characterization of this study) of these fibers used in the present study are  $320\text{ }\mu\text{m}$  for banana fiber, about  $66.9\text{--}312\text{ }\mu\text{m}$  for bagasse and about  $233\text{ }\mu\text{m}$  for sponge gourd fiber. These mean values are arrived based on 100 fiber observations on each fiber taking into account variation in fiber sizes ( $<1.5\text{ mm}$  to  $>3.00\text{ mm}$ ) and the fraction of each (up to  $1.5\text{ mm}$  –57% for banana, 7% for sugarcane bagasse and 24% for sponge gourd fibers, and  $>1.5\text{--}3\text{ mm}$  at 37%, 84% and 67% respectively for these fibers and  $>3\text{ mm}$  at 6%, 9% and 9% respectively).

Very few studies report on the morphology for the fibers under study (Tanobe et al., 2005; Satyanarayana et al., 2007, 2009; Satyanarayana and Wypych, 2007). Figs. 4(a)–(c) and 5(a)–(f) show the cross-section of all the three fibers studied by optical microscope and scanning electron microscope respectively.

It can be seen that, as in the case of other natural fibers (Satyanarayana and Wypych, 2007), the three fibers studied here show variations in their structure, namely, different sizes, shape and arrangement of their cells as well as nature of lumen.

Cells are non-spherical rather irregular in shape and cell walls are thick in banana and sponge gourd fibers, while they are almost spherical, compactly arranged and thin walled in the case of bagasse fiber. In fact, this is reported to be the reason for providing more compression during the bagasse fiber composite preparation by compression molding, resulting in less interference of the fibers within the matrix unlike kenaf fibers (Shibata et al., 2005). The central lumen is elliptical (or nearly spherical) and of higher diameter in the case of bagasse fiber compared to other two fibers, which show very narrow and elongated lumen. At higher magnifications, all the fibers show helical winding of microfibrils attach to each other by a binding material (lignin). The interior of these microfibrils shows longitudinal array in some cases. Thickness of this is highest in sponge gourd fiber followed by that in banana fiber. Also, the lacuna is present in bagasse fibers while it seems to be absent in other two fibers.

### 3.5. Thermal behavior studies of fibers

Fig. 6(a)–(c) shows the TGA and DTG (1st derivative) curves, while Fig. 7(a)–(c) the DSC curves of the three fibers respectively. It can be seen that the curves in Fig. 6(a)–(c) show similar behavior with three main mass loss steps.

The first one up to  $100^\circ\text{C}$  representing the mass loss in all the fibers could be related to water loss associated with moisture present in the fibers. Although the fibers were dried before the analysis, total elimination of water was difficult due to the hydrophilic nature of the fibers, which is present even as structurally bound water molecules. Then, between  $100$  and  $200^\circ\text{C}$ , the fibers present thermal stability (see TGA and DTG curves, Fig. 6). One may consider  $200^\circ\text{C}$  for banana ( $250^\circ\text{C}$  for bagasse and sponge gourd) as the maximum temperature up to which these fibers can be used since above this temperature, mass loss is high. From  $300^\circ\text{C}$  and above, all the samples show considerable mass loss due to the decomposition of both cellulose and hemicellulose in the fibers. Further, above  $400^\circ\text{C}$ , degradation of fibers can be seen as a result of the break of bonds of the *protolignin* (lignin present in the fibers) (Paiva et al., 2004; Rohella et al., 1996). In both steps the mass loss is attributed to the oxidation (burning) of the fragments in the presence of air, as confirmed by the DSC curves (Fig. 7). After  $500^\circ\text{C}$  only the ashes can be seen, which are 4.14%, 2.75% and 0.66% for banana, bagasse and sponge gourd fibers respectively.

The DTG results (1st derivative, Fig. 6) seem to confirm that banana fibers have a lower ratio of holocellulose/lignin when compared to the other fibers, which is also in agreement with the results of fiber composition (see Table 1). The ratio between the intensity of the peaks related to the decomposition of cellulose–hemicellulose (holocellulose)/lignin is lower for banana fibers compared to those of bagasse and sponge gourd fibers.

From DSC studies of the fibers (Fig. 7), it can be seen that the peak of all the three fiber samples around  $300^\circ\text{C}$  can be related to the decomposition of cellulose, which is in agreement with the mass loss observed in this range in the thermogravimetric analyses of all samples. During the thermal degradation of cellulose, depolymerization occurs and formation of 1.6 anhydro-glucose takes place whose decomposition involves the formation of volatiles, as indicated by the endothermic peaks. The peak between  $400$  and  $500^\circ\text{C}$  is related mainly to the breaking of the chemical bonds of the *protolignin* present in the fiber. Within this temperature interval, a mass loss is also observed in the TGA curves of the fibers (Paiva and Frollini, 2006). Considering the DSC and TGA (mainly the 1st derivative) curves, it can be observed that banana fibers present a slight decrease in temperature related to the beginning of the thermal degradation (corresponding to the degradation of cellulose and hemicelluloses) compared to those of sugarcane and sponge gourd fibers. The banana fibers show lower degree of crystallinity than the other fibers in agreement with X-ray diffraction results presented above. The higher proportion of chains with weaker intermolecular interactions (non-crystalline region) of banana fibers can lead to a downshift in the degradation temperatures.

## 4. Conclusions

Chemical compositions of three Brazilian fibers (banana, sugarcane bagasse and sponge gourd) studied present study are different from those reported elsewhere due to dependence of chemical composition of lignocellulosic fibers on locality and species.

Banana fibers showed lower moisture content, holocellulose, cellulose and hemicellulose contents, but higher ash and Klason lignin contents than the reported values elsewhere. Bagasse fibers showed lower ash content with all other contents mentioned are



within the range reported values by others. In the case of sponge gourd fibers, except for hemicellulose content, all other values were found to be slightly higher than those reported for this fiber by others.

X-ray diffraction patterns of these three fibers are similar to those reported for other lignocellulosic fibers exhibiting mainly the Cellulose I structure with the crystallinity values of 39%, 48% and 50% for banana, bagasse and sponge gourd fibers respectively.

Morphological studies of the fibers revealed different sizes and arrangement of cells, with cells being non-spherical and thick cell walls in both the fibers of banana and in sponge gourd, while they are almost spherical, compactly arranged and thin walled in the case of bagasse fiber. Also, the central lumen is almost spherical and bigger in the case of bagasse fiber compared to other two fibers.

Thermal stability of all the fibers is found to be around 200 °C. Decomposition of both cellulose and hemicellulose in the fibers takes place at 300 °C and above, while the degradation of fibers takes place above 400 °C, as a result of the break of bonds of the *protolignin*.

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