Comparative Study of the Characteristics of Green and Brown Coconut Fibers for the Development of Green Composites

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The growing consumption of tender coconut water in Brazil has resulted in a generation of green husk, which in turn has led to pollution, as it takes eight to ten years to degrade. With the objective of finding applications for these fibers, the characterization of their chemical composition, tensile properties, and structural properties is presented in this paper. The density of the green fibers was 1200 kg/m^3, and the diameter ranged between 272 µm and 513 µm. The length of the ultimate fibers was 940 µm, while the cell wall thickness and size of lumen were approximately 3.6 µm and 11.8 µm, respectively. The crystallinity index, ultimate tensile strength, Young's modulus, and elongation of the lignocellulosic fiber were 48%, 114 MPa to 159 MPa, 1.20 GPa to 1.96 GPa, and 41% to 44%, respectively. These results were compared with previously published results of both green and brown coir fibers with the purpose of exploring the addition of value to this abundant agro-industrial residue.

Keywords: Green coir fibers; Ultimate fibers; Morphology; Chemical composition; Physical properties; Tensile properties

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INTRODUCTION

Cocos nucifera L., commonly known as the coconut tree or coconut palm, is one of many species grown in approximately 10 million ha of land throughout tropical and subtropical countries (FAO 2016). This tree has various uses and is one of the most economically important crops (Calado *et al.* 2000). The coconut fruit has different colors (green, yellow, or brown) depending on its cultivation (Lorenzi *et al.* 2006).

In Brazil, coconut palm is cultivated across almost the entire national territory (in tropical areas), except for the states of Rio Grande do Sul and Santa Catarina because of their climatic limitations during part of the year (Cuenca 2007). Currently, the country is the fourth highest producer of coconuts in the world (2 metric tons (MT), 89 MT, and 286 MT; about 5.66% of total world production), behind Indonesia, the Philippines, and India (Esmeraldo *et al.* 2010; Top 5 of Anything 2016). The benefits of coconut-based natural foods with excellent nutritional values, such as coconut water, have steadily promoted an

emphasis on exploring dwarf varieties of coconut trees. The use of coconut water from green coconuts (tender coconuts) is prevalent in countries like India. Accordingly, the market for coconut water from green coconut has been growing in many countries in the last few years, particularly in Brazil, which has led to an increase in the consumption of green coconuts. This has increased the generation of byproducts, such as the husk, which corresponds to approximately 85% of the fruit weight (Rosa *et al.* 2001a; Cuenca 2007; Ishizaki *et al.* 2008). The husks are generally discarded on embankments and left in the open environment as waste, where it takes eight to ten years to degrade. This leads to the generation and accumulation of waste (Carrijo *et al.* 2002; Ferreira *et al.* 2006; Corradini *et al.* 2009). As a result, contamination of the soil has become a problem. Therefore, there is a need for finding ways to use the husks from green coconuts, particularly for value addition. This is because these fibers, which are obtained from the mesocarp of coconuts (Corradini *et al.* 2009; Esmeraldo *et al.* 2010), are cheap, recyclable, abundant, and non-poisonous (Satyanarayana *et al.* 2007). Additionally, coir fiber is a versatile material, with applications in various engineering sectors, including as a sustainable construction material (Ali 2010). The objective of this study was to characterize the green coconut fibers from Brazil with respect to their physical, structural, and chemical aspects (crystallinity and morphology), as well as their tensile properties.

The global annual production of coir fibers in 2014 was reported to be 350 x 10^3 MT (Coir Board 2014), despite coir industries all over the world processing only a fraction of the available coconut husks. There are two types of coir fibers produced globally, which are multi-cellular in nature. The first is produced from green coconuts, which are eight to nine months in age, mostly by a biological maceration process, and these fibers are called green coir or green coconut fibers. These are smooth and mostly white in color and possess a moisture content of 85% (Rosa *et al.* 2001b; Rajkumar *et al.* 2009; Ali 2010; da Costa *et al.* 2013). This moisture content usually discourages their use in some applications that use husks and fibers from dry coconut (Rosa *et al*. 2001b). This is because the surface of green coconut exhibits different properties. These differences include: a smooth surface, a high moisture content (85%), and hardness; the rigidity is provided by the lignin, which gives a good resistance along with the preservation against the attacks by bacteria because the lignin has a high natural preservation towards other natural fibers (da Costa *et al.* 2013; Leite *et al*. 2010; Rosa *et al.* 2001a,b) from those of ripe coconuts.

The second variety is produced from the husks of mature coconuts by retting or biotechnological methods. These fibers are brown in color, thick, and possess a high resistance to abrasion, the environment, and rot (Mogre 2012; da Costa *et al.* 2013).

There have been several papers published dealing with the availability, processing or extraction, surface treatments, and characterization of coir fibers. There are also many papers and reviews dealing with coir fiber-based composites with polymers and ceramics or cement as matrices. In view of the very large numbers of publications related to both varieties of lignocellulosic fibers, only those related to green and brown fibers were mentioned here (Karnani *et al.* 1997; Calado *et al.* 2000; Rout *et al.* 2000; Rowell *et al.* 2000; Silva *et al.* 2000; Bismarck *et al.* 2001; Rosa *et al.* 2001a; Carrijo *et al.* 2002; Ferreira *et al.* 2002; Razera and Frollini 2002; van Dam *et al.* 2004; Ramakrishna and Sundararajan 2005a; Ramakrishna and Sundararajan 2005b; Ferreira *et al.* 2006; Khalil *et al.* 2006; Lorenzi *et al.* 2006; van Dam *et al.* 2006; Wiedman 2006; Cuenca 2007; Munawar *et al.* 2007; Satyanarayana *et al.* 2007; Tomczak *et al.* 2007; Bessadok *et al.* 2008; Ishizaki *et al.* 2008; John and Anandjiwala 2008; Monteiro *et al.* 2008;

Satyanarayana *et al.* 2008; Bodîrlău and Teacă 2009; Corradini *et al.* 2009; Guimarães *et al.* 2009; Ishizaki *et al.* 2009; Mahato *et al.* 2009; Mothé and de Miranda 2009; Oksman *et al.* 2009; Rajkumar *et al.* 2009; Brígida *et al.* 2010; Carvalho *et al.* 2010; Esmeraldo *et al.* 2010; Samarawickrama 2010; Wu 2010; Hussain *et al.* 2011; Israel *et al.* 2011; Lomelí-Ramírez 2011; Sen and Reddy 2011; Thomason *et al.* 2011; Yogaratnam 2011; Fiorelli *et al.* 2012; Mogre 2012; da Costa *et al.* 2013; Everitt *et al.* 2013; Reddy 2013; Reddy and Hussain 2013; de Oliveira and Marques 2014; Dhandhania and Sawant 2014; Kadam *et al.* 2014; Lomelí-Ramírez *et al.* 2014; Mathura *et al.* 2014).

After studying the above-mentioned published reports, some important conclusions were drawn. These included: (i) there is variation in the properties between the two types of lignocellulosic coir fibers; (ii) variation in many of the properties are dictated by the age of the coconuts from which the fibers are extracted and locality (geography) in which the coconut trees are grown; (iii) variation of the properties are caused by the method of extraction of the lignocellulosic fiber; and (iv) variation of the properties are caused by the method of determination and experimental conditions used. These conclusions will be highlighted later in the paper.

The applications of these two varieties of lignocellulosic coir fibers, particularly in conventional applications, are generally based on their characteristics. For example, green lignocellulosic fibers have been mostly used in making yarn, rope, and fishing nets, while brown fibers are used in mattresses, brushes, agriculture, automobiles, composites, *etc*. (Ferreira *et al.* 2006; Sen and Reddy 2011).

Considering the fact that a very few characteristics as mentioned in the previous page are known [smooth surface, high moisture content (85%), hardness and the rigidity] (Rosa *et al.* 2001b), green lignocellulosic coir fibers are not favored for applications as much as the brown variety, despite their potential (Ferreira *et al.* 2006; da Costa *et al.* 2013). Hence, their characteristics may also be different because the morphology, chemical constituents, physical properties, mechanical properties, and other properties of coconuts and their fibers vary with their genetic variety, maturity, collection time, growth area, soil type, *etc*. (van Dam *et al.* 2006). A complete characterization of the fibers from green coconut is thus needed to provide a basis for exploring wider applications of green coconut fibers in value-added products, which is relevant to countries such as Brazil, India, *etc*.

Accordingly, the present authors systematically characterized green coconut lignocellulosic fibers from Brazil, including extraction of ultimate fiber, measurement of their dimensions, and determination of their possible use in the development of green composites. Except for two studies on the extraction and measurement of the dimensions of ultimate fibers from coir fiber (Khalil *et al*. 2006; John and Anandjiwala 2008), there is no mention of ultimate fibers in any of the published reports. Their potential in the development of green composites has been studied by the authors of this paper (Lomelí-Ramírez *et al.* 2011; Lomelí-Ramírez *et al.* 2014). Ultimate fiber refers to individual microfibers that have some useful characteristics, such as the dimensions (length and diameter), resistance to tension, and other properties, required for various industrial applications.

EXPERIMENTAL

Materials

The green coconut fibers used in this study were obtained from the Cooperative of Improvement of Green Coconut Husk (COOBCOCO) located in Fortaleza, Ceará, Brazil.

Methods

Measurement of the dimensions of the fibers and cells

The fibers were first dried for 48 h at 70 °C in a hot air oven. They were cut into 10-mm long strips using a knife and sieved in a Wiley mill to get fibers of different mesh sizes. The fibers were not of uniform diameter along the length of the fiber, which could be observed visually. Fifteen samples for each group of fibers (fine and thick) were examined with a minimum of three measurements of each region of the fiber along its length (the two extremities and middle of the fiber). The measurement data was used to determine the average diameter of the fibers.

For measurement of the dimensions of the fiber cells (also called ultimate fibers), the as-received fibers were first softened by a modified Franklin's technique following the method in Monteiro *et al*. (2008). Briefly, the method is as follows: the fibers were soaked in a solution of hydrogen peroxide and acetic acid (1:1) and heated in a hot air oven at 60 °C for 24 h.

The cells obtained were then washed using distilled water and colored with safranin stain. This color has been stated in the literature to be the standard color used, particularly for lignocellulosic fibers. The dimensions of these cells were measured following the IAWA standard (Wheeler *et al*. 1989). This standard is a valuable guide and reference to identify wood (lignocellulosic materials in general) and anatomy of descriptive wood/lignocellulosic materials in general as per the authors of this standard, *viz*., Wheeler *et al*. (1989).

Measurements of both the diameter of the fiber and dimensions of the obtained cells were made with an optical microscope (CX410, Olympus, Tokyo, Japan). This was done on a micrometric scale and increasing the ocular magnification with lenses from 2.5x to 100x. An Olympus Camedia digital camera coupled with C3000 attached to a camera and suitable software was also used.

The longitudinal and cross-sectional features of the fibers were also analyzed with scanning electron microscopy (SEM) (JSM-6360 LV, JEOL, Tokyo, Japan) using an accelerating (operating) voltage of 15 kV. The fibers were first coated with a thin layer of gold and kept on a metallic support.

Measurement of density

The density of the coconut fibers from green coconut was determined using a pycnometer (laboratory bottle to measure specific gravity) following the methods used by Monteiro *et al*. (2008).

Chemical composition of the fibers

A chemical analysis of the fibers was conducted because of the importance of knowing the fiber constituents for finding potential uses and determining the possibility of using these fibers in the preparation of green composites with cassava starch. A certain amount of fibers was prepared for the chemical composition analysis by milling them in a Wiley mill. Fibers with a mesh size of 60 were chosen per TAPPI T257 cm-02 (2002).

Various constituents of the coir fibers were determined using the following standards: NBR 11941 (2003) for the moisture content, TAPPI T222 om-02 (2002) for the lignin content, NBR 14577 (2009) for water soluble materials, NBR 14660 (2010) for the total extractives content, NBR 14853 (2010) for the alcohol-toluene solubility, NBR 7990 (2001) for the solubility in NaOH to 1%, and TAPPI T413 om-02 (2002) for the ash content. The amount of cellulose and hemicellulose were calculated using the amount of all of the other constituents mentioned above. Three repetitions were done for the determination of each constituent.

Fourier transform infrared (FTIR) analysis

The Fourier transform infrared (FTIR) spectrum of the fiber was obtained using Bio-Rad equipment (Excalibur model, Hercules, CA, USA) with 64 scans and a resolution of 4 cm^{-1} over the wave number range of 4000 cm^{-1} to 700 cm^{-1}. The fibers were mixed with a KBr tablet at a ratio of 1:3. Three repetitions were done, and the average values were considered.

X-ray diffraction (XRD) analysis

To determine the crystallinity index of the fibers, an X-ray diffraction (XRD) analysis was performed at least three times using a Shimadzu X-ray diffractometer (XRD 7000, Kyoto, Japan) with CuK α radiation (wavelength = 0.15418 nm). The operating conditions used were as follows: 40 kV/20 mA and an angular scanning speed of 1°/min in the 2θ range of 2.5° to 60°. Then, the crystallinity index (C) of the fibers was determined using Eq. 1 (Tanobe 2003),

$$\%C = 1 - \frac{I_1}{I_2} \quad (1)$$

where I_1 and I_2 are the intensities of the amorphous and crystalline areas of the fiber, respectively. Origin software (v. 5.0, OriginLab Corporation, Northampton, MA, USA) was used for the calculations of deconvolusions indicating amorphous and crystalline areas in the obtained spectrum.

Tensile properties determination

Stress-strain curves of the green coconut fibers were obtained by testing two sizes of the fibers (fine and thick) in a universal testing Instron machine (model 4467, Norwood, MA, USA) with a gauge length of 20 mm and strain rate (testing speed) of 5 mm/min. A minimum of five samples for each fiber size (Thick fiber = 2.02 ± 0.25 μm; fine fiber = 0.39 ± 0.05 μm) were tested and the average values of the ultimate tensile strength (UTS), Young's modulus (YM), and elongation were obtained from the curves.

RESULTS AND DISCUSSION

The results of the present study were compared with previously reported results of both green and brown coir fibers. The variations, if any, were discussed based on two factors (Silva *et al*. 2000; Satyanarayana and Wypych 2007; Satyanarayana *et al*. 2008; Oksman *et al*. 2009; Thomason *et al*. 2011). The first factor was inherent to the fibers, while the second was with respect to the testing conditions and techniques used by

various researchers. The first factor included the maturity of the coconuts from which the fibers were produced (age), location, fiber extraction techniques, chemical composition of the fiber (particularly the cellulose and lignin contents), morphology, and dimensions of the cells (ultimate fiber). The second factor included the temperature, humidity, environment (for example, the use of inert gas in thermal property analyses), and equations used to calculate a certain property (for example, crystallinity).

Physical Properties

Dimensions and morphology

The diameter and length of natural fibers, such as green coconut fibers, are relevant parameters to their applications, especially in the development of polymer-based composites, where they are used as reinforcement. Figure 1a shows a photograph of green coconut fibers with different diameters and lengths, while Fig. 1b is a photograph of a single fiber showing the non-uniformity along the length of the fiber. The diameter of the fiber tapered from one end to the other, as can be seen from the values listed in Table 1. This is understandable because the green coconut fibers can be divided into fine, medium, and thick and the most resistant fibers are those with higher linear density (da Costa *et al.* 2013), which may be the ones within the range of 300 and 500 microns. Similar reports were published from different regions and countries. For example, reported values for green coir fibers include 55.6 μm to 197.6 μm (Rahman and Khan 2007), 69 μm to 495 μm (Brígida *et al*. 2010), 170 μm to 240 μm (Hasan *et al*. 2012), 200 μm (Leite *et al.* 2010), and 600 μm (Reddy 2013), while a value of 110 μm to 460 μm (Mathura *et al.* 2014) was reported for brown lignocellulosic coir fibers.

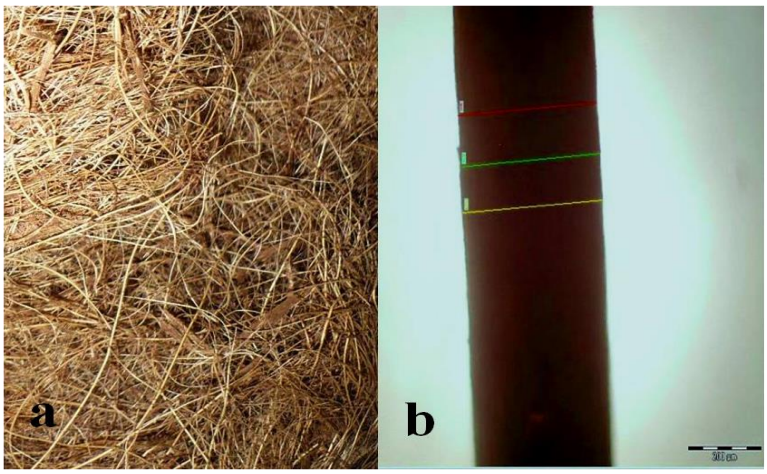

Fig. 1. Photographs of lignocellulosic fibers from green coconut: (a) bunch of fibers with different diameters; and (b) single fiber for measuring the diameter

Table 1. Diameter of the Fibers from Green Coconut

Location in the Fiber	Fine or Thin Fiber (μm)	Thick Fiber (μm)
One Extreme	321.18 ± 1.41	513.71 ± 2.53
Middle	305.90 ± 1.45	498.06 ± 4.65
Other Extreme	272.63 ± 2.58	470.84 ± 5.95

Figures 2 and 3 show SEM images of the green lignocellulosic coconut fibers obtained from the SEM study, both in the transverse and longitudinal directions respectively. While Fig. 2a shows the cross-sectional view of the fiber, Fig. 2b is the enlarged view of the center of Fig. 2a. Figure 2c is a higher magnification of part of Fig. 2b. As shown, the fiber was multi-cellular, containing cells with a polygonal shape joined at the surface, and it had a large empty space called a 'lacuna' at the center of individual cells. The cells (micro fibrils) were of different sizes, but had thick walls. There was a hole at the center of each of these microfibers called a lumen, which also varied in size. Such observations have also been reported for coir fibers from other regions (Khalil *et al.* 2006; Tomczak *et al.* 2007; John and Anandjiwala 2008).

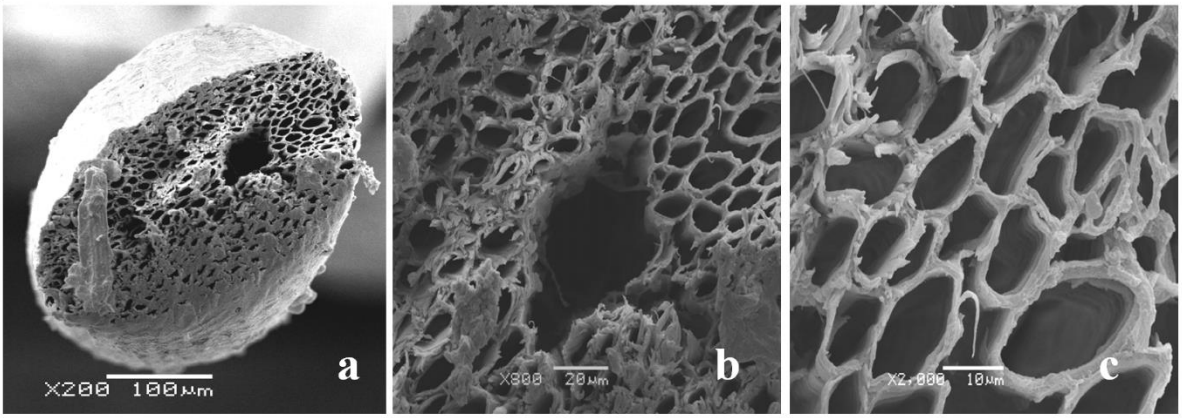

Fig. 2. SEM photographs: (a) Transverse section of the green coconut fiber; (b) Enlarged view of (a) showing the cellular wall of the ultimate fibers; and (c) Higher magnification of (b)

Transmission electron microscopy studies have shown that the cell walls of coconut fibers are composed of a primary cell wall, which is supported by three secondary walls (Khalil *et al.* 2006). Figure 3 shows the SEM photographs of the longitudinal sections of the green coconut fiber. Figure 3a is an image of the surface of the fiber, which showed a typical rough surface with some openings. Carvalho *et al*. (2010) have referred to these structures as globular protuberances and tyloses (outgrowths on parenchyma cells of xylem vessels), where wax is deposited. These protuberances or re-entrances are located on the fiber surface and are placed at regular intervals. They were also reported by other researchers for similar fibers (Calado *et al.* 2000; Rout *et al.* 2000; Monteiro *et al.* 2008; Corradini *et al.* 2009; Rosa *et al.* 2009; Brígida *et al.* 2010; Carvalho *et al.* 2010). These protrusions have been reported to be clusters of calcium oxalate crystals.

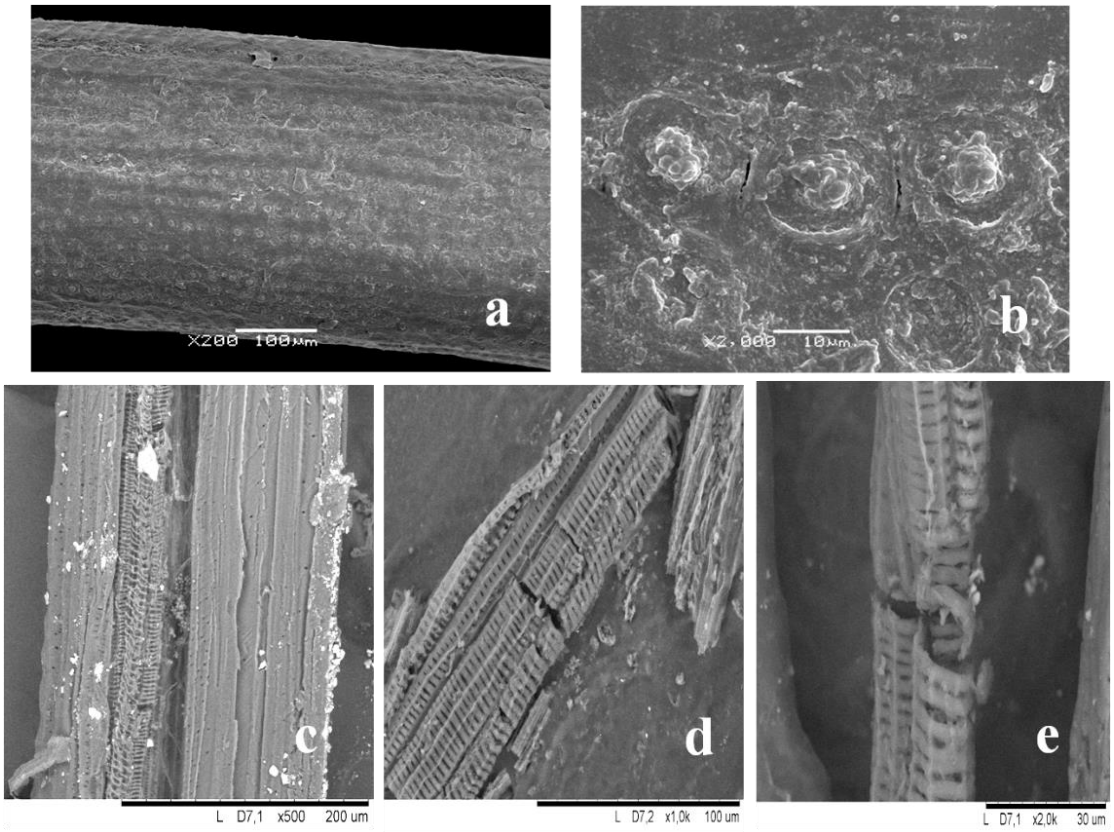

Fig. 3. SEM photographs showing the longitudinal view of the lignocellulosic fiber from green coconuts: (a) Surface of the fiber; (b) High magnification image of the longitudinal surface showing round clusters of protrusions made of calcium oxalate crystals; (c-e) High magnification photographs of the surface: (c) Cracks on the surface showing the spiral nature of the cellulose micro fibrils; (d) Various cellulose micro fibrils; and (e) Photograph of one single cellulose micro fibril

Figure 3b is an enlarged photograph of the surface, which showed globular-type protrusions adhering to the surface and revealed more details of such protrusions occurring at regular intervals. These rough surfaces were found to be lying along the length of the fiber when the coir fiber was used in composites (Corradini *et al.* 2009). According to Corradini *et al.* (2009), these could act as anchor areas, whereby the interface between the fiber and matrix could improve, leading to an increased mechanical resistance. They further showed that the coconut fiber was covered with a layer of oils, waxes, and extractives, which are part of the natural constitution of the lignocellulosic fibers. Monteiro *et al.* (2008) have reported the presence of calcium and silicon associated with the protrusions on the surface of coir fiber. The above-mentioned differences may have been because of the different types of soil used for the cultivation of coconut plants.

The high magnification photographs of the fiber surface show some cracks along the surface (Fig. 3c), a number of cellulose micro fibrils with a spiral nature (Fig. 3d), and a single cellulose micro fibril (Fig. 3e).

Further studies were conducted to understand the finer structure of ultimate fiber (micro fibrils) from green coconut fibers. Figure 4 shows macro photographs of ultimate fibers obtained from these fibers. While Fig. 4a shows the longitudinal morphology of

these ultimate fibers, Figs. 4b and 4c show the detailed structure (spiral of cellulose fibrils) of each individual ultimate fiber.

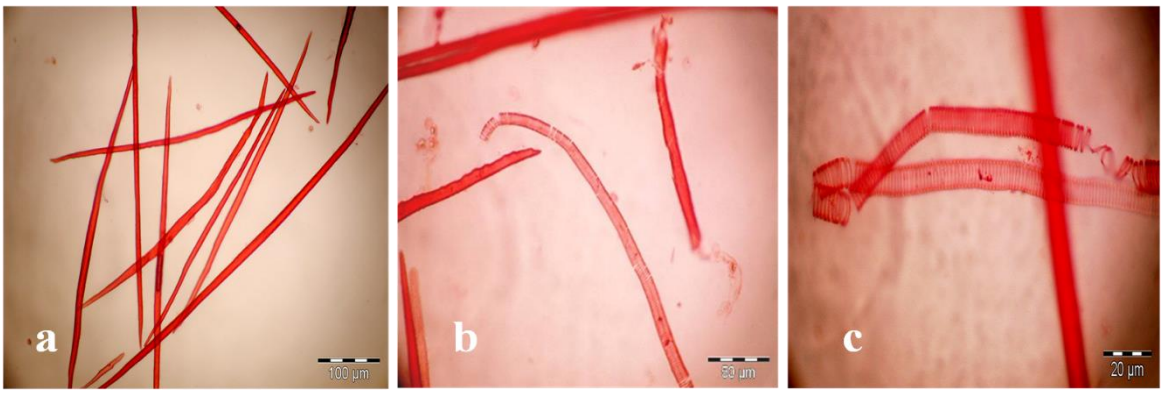

Fig. 4. Macro photographs of the individual ultimate fibers obtained from the green coconut lignocellulosic fibers: (a) Group of ultimate fibers; (b) Higher magnification of (a); (c) Individual ultimate fiber showing the spiral structure of the cellulose

These observations were in agreement with the results of earlier SEM studies, which also showed cellulose microfibers having a spiral structure (John and Anandjiwala 2008; Bakri and Eichhorn 2010). Some researchers have also reported such structures for coir fibers with different origins and found that the presence of these ultimate fibers have an orientation of 45°, which affects the tensile properties, such as the elongation and YM (van Dam *et al.* 2006; John and Anandjiwala 2008; Mahato *et al.* 2009).

Measurements of the dimensions of the ultimate fibers (length of the cells, cell wall thickness, and diameter of lumen) were made from the above photographs, and the values are given in Table 2. It was observed that the length varied with the fine cell walls and diameter of the lumen. The values were in agreement with previously reported values (van Dam *et al.* 2006).

Table 2. Dimensions of the Individual Ultimate Fibers

	Length (µm)	Cell wall thickness (µm)	Diameter of lumen (µm)
Average	942.5	3.5	11.7
Standard deviation	145.9	0.4	2.2
Maximum value	1177.3	4.5	16.3
Minimum value	681.8	2.6	8.1

It was also observed that the two values listed in Table 2 contrasted with previously reported values for green coconut fiber (Munawar *et al.* 2007) and the length and diameter of individual cells from brown coir fiber (approximately 1 mm and 10 µm to 20 µm, respectively) (Dixit and Verma 2012). Such differences in the values of these lignocellulosic fiber dimensions depend on the species, maturity, and location, as well as the conditions of fiber extraction (Reddy and Yang 2005). Reddy and Yang (2005) have also asserted that usually the individual cells (ultimate fibers) are too small to be used for textile applications, but they can be used in the manufacture of paper or other similar applications.

Density of the green coconut fibers

The true density of the fibers used in this study was found to be 1200 kg/m^3, which was in the range of values (1177 kg/m^3 to 1310 kg/m^3) reported in other studies (Ferreira *et al.* 2006; van Dam *et al.* 2006; Wu 2010; Sen and Reddy 2011). However, there are also reports of very low density values (0.825 kg/m^3 to 0.87 kg/m^3 (Munawar *et al.* 2007; Brígida *et al.* 2010). Again, all of these differences depend on the species, whether the fiber is from green or mature coconut (brown fiber), and the method used to determine the density. It has been generally noted that the density value of lignocellulosic fibers, such as that of coir fiber, is 40% to 55% lower than that of glass fibers (2500 kg/m^3); therefore, these lighter fibers can substitute glass fibers as reinforcement in polymer-based composites (Satyanarayana and Wypych 2007).

Chemical and Structural Aspects

Chemical composition

The chemical composition of fibers is essential and relevant for finding their uses, particularly when using them as reinforcement for several types of natural and synthetic polymeric matrices. For instance, in the case of their use as reinforcement, the interface between the matrix and filler/reinforcement leads to chemical compatibility between the polymer matrix and fiber because the interface plays an import role in dictating good adhesion and consequently good mechanical properties. However, it is important to note that the complete chemical characterization of a specific fiber may be difficult because the composition of lignocellulosic fibers is affected by a number of factors (nature, collection location, age, genetic variety, species, type of soil, growth conditions, maturity of the coconut at time of harvesting, *etc*.) (Satyanarayana *et al.* 1986; Asasutjarit *et al.* 2007; Satyanarayana *et al.* 2008; Guimarães *et al.* 2009).

Table 3 lists the average values of the chemical composition of the fibers from green coconut used in this study, along with those reported in the literature for fibers from both green and brown coconuts in other regions and countries. It should be noted that the holocellulose contents were derived by taking the total composition as 100%, while that of the lignin, ash, and extracts from cold and hot water, NaOH, and ethanol-toluene were determined in the present study. It is also interesting to note that except for one earlier study on brown coir fibers (Khan and Alam 2012), no other study has reported the hemicellulose content, and hence those values are not shown in the table.

Results in Table 3 indicate that the lignin content obtained in the present study was in the range of 32% to 33%, which was similar to values reported by some researchers (Ramakrishna and Sundararajan 2005a; Reddy and Yang 2005; Khalil *et al.* 2006; van Dam *et al.* 2006; Reddy 2013), but was also lower than some values reported by others (Satyanarayana *et al.*1986; Reddy and Yang 2005; Salazar and Leão 2006; Asasutjarit *et al.* 2007; Satyanarayana *et al.* 2007; Guimarães *et al.* 2009; Ali 2010; Brígida *et al.* 2010; Khan and Alam 2012). Similarly, the lignin content obtained in the present study was higher than that obtained for green coconut fibers from Brazil and other countries that have a predominantly tropical climate, such as Jamaica and Sri Lanka (Satyanarayana *et al.* 2007; Corradini *et al.* 2009). It may be relevant to note that the lignin content of approximately 48% was reported for brown fibers from Brazil (da Costa *et al.* 2013). This higher lignin content in coir fiber is advantageous for application in the footwear industry because lignin provides good resistance and protection against microorganisms.

Table 3. Comparison of the Composition of Green and Brown Coir Fibers

Ash (%)	Cellulose (%)	Lignin (%)	Other (%) [b]	Fiber	Reference
1.25	29.96 [a]	35.46	33.33	Green Fiber	Present Study
2.6	35.1	33.6	2.3	Green Fiber	Van Dam *et al*. (2006)
NA	43.4 - 53	38.3 - 40.77	3.5	Brown Fiber	Wiedman *et al*. (2000)
2.4	44.2	32.8	6.4	Brown Fiber	Khalli *et al*. (2006)
1.34	31.83	45.47	2.33	Brown Fiber	Salazar and Leão (2006)
NA	37.11	44.06	NA	Brown Fiber	Khan and Alam (2012)
NA	41.55	45.95	31.10	Brown Fiber	Brígida *et al*. (2010)
NA	20.50	33.20	NA	Brown Fiber	Ramakrishna and Sundararajan (2005a)
NA	20 - 48	35 - 60	15 - 28	Brown Fiber	Agopyan *et al*. (2005)
NA	41 - 45	36 - 43	0.15 - 0.25	Brown Fiber	Corradini *et al*. (2006)
NA	32.1	68.9	16.8	Brown Fiber	Asasutjarit *et al*. (2007)
0.61	29.23 - 36.51	23.81 - 33.51	15 - 28	Brown Fiber	Reddy (2013)

NA: not available; a: Holocellulose; b: Includes extracts from cold and hot water, NaOH, and ethanol-toluene

The values obtained for the ash content in the present study were lower and the total extracts content was between the values reported in the literature (2.3% to 6.4%). However, the extracts content was almost twice the value reported by Reddy (2013) (0.61%). This was a reflection of the soil characteristics.

All of the variations in the chemical composition of green coconut fibers were because of the various factors mentioned earlier, such as the method of chemical analysis, experimental conditions, *etc*. (Rowell *et al.* 2000; van Dam *et al.* 2006; Satyanarayana and Wypych 2007; Bessadok *et al.* 2008).

FTIR analysis

FTIR spectroscopy is one of the techniques used most often for the chemical characterization of natural fibers because it helps to identify functional groups and molecules that are present in a sample. Figure 5 shows the FTIR spectra of the green coconut fibers.

The absorption bands in the spectrum were typical of lignocellulosic material reported in the literature. The observed bands were attributed mainly to cellulose and lignin. The peaks in the region of 3413 cm^{-1} to 3444 cm^{-1} represented hydrogen bonded stretching bands of OH groups in cellulose on the 2, 3, and 6 carbons of glucose (Satyanarayana *et al.* 2007). Such bands also appeared around 1060 cm^{-1}. De Oliveira and Marques (2014) have reported similar FTIR spectra for green coir fibers with an intense absorption band in the 3500 cm^{-1} to 3200 cm^{-1} region (stretching of –OH bond), followed by bands at 2900 cm^{-1} to 2750 cm^{-1} (stretching of saturated hydrocarbon of CH), at 1740 cm^{-1} (stretching of C=O bonds related to carboxylic ester or acetyl groups (hemicelluloses)), 1240 cm^{-1} (absorption of angular strain of CH (cellulose and lignin)), and 730 cm^{-1} (aromatic H in lignin). These results confirmed that the major constituents of this fiber were cellulose, hemicellulose, and lignin.

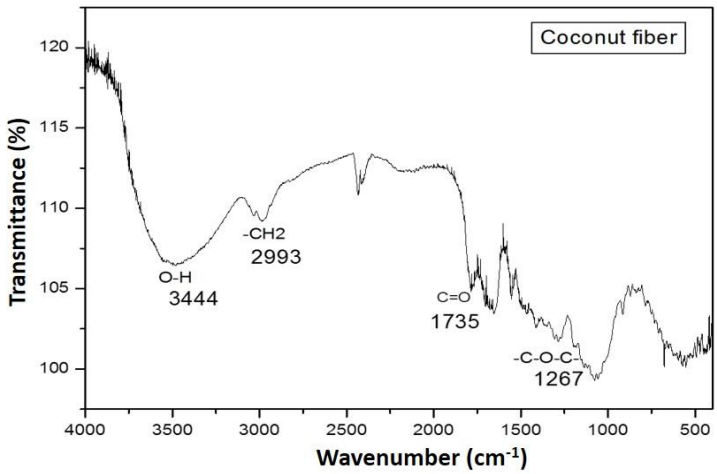

Fig. 5. FTIR spectra of the lignocellulosic fiber from green coconut

The absorption peaks at 3413 cm^{-1} to 3444 cm^{-1} were attributed to cellulose and lignin, including C-O-C and C-O stretching (primary and secondary hydroxide groups), and bonds belonging to the glucoside linkage and lignin that contains aromatic compounds. The broad band in the region of 3413 cm^{-1} to 3444 cm^{-1} may also have indicated the presence of moisture in the coconut fibers. While Satyanarayana *et al*. (2007) attributed the bands in the region of 1800 cm^{-1} to 400 cm^{-1} to structural cellulose (for brown coir fibers), the bands in the region of 1735 cm^{-1} observed in this study were attributed to unconjugated C=O stretching, mainly because of the presence of hemicellulose groups, which was in agreement with the conclusions drawn by Mahato *et al*. (2009) and Brígida *et al*. (2010). The bands near 1650 cm^{-1} were assigned to conjugated carbonyl groups present in lignin, and the band at 1508 cm^{-1} corresponded to aromatic rings of lignin (Mahato *et al.* 2009). Additionally, the presence of bands near 1600 cm^{-1} were attributed to molecular vibration of absorbed water, while bands in the region of 2993 cm^{-1} to 2375 cm^{-1} were attributed to C-H symmetric and asymmetric stretching. Finally, bands around 1267 cm^{-1} were related to axial asymmetric deformation of glycosidic bonds in polymeric chains (Guimarães *et al*. 2009).

XRD analysis

Figure 6 shows the XRD curve of green coconut fibers. The curve was characteristic of native cellulose or type I cellulose found in most lignocellulosic fibers and commonly seen in lignocellulosic materials (Satyanarayana *et al.* 1986; Guimarães *et al*. 2009). The lines shown in red, green, and pink, which were traced using the Origin software (v. 5.0) during the calculation of the deconvolutions, were indicative of amorphous and crystalline areas. The red and green curves corresponded to the crystalline areas, while the curve in pink corresponded to the amorphous area. The observed peaks were produced by specific portions of the crystalline regions and corresponded to the peaks centered at the diffraction angles (2θ or Bragg angle) of 16.19°, 22.14°, and 34.73°. These results agreed with the results of Carvalho *et al.* (2010), who reported diffraction peaks of 16.15° and 22.01° for untreated coconut fiber. In fact, more realistically it can be said that the angles in the regions of 13 ^{o} to 15 ^{o} and 18 ^{o} to 19 ^{o} at which the peaks related to type I cellulose are related to the amorphous part of this cellulose.

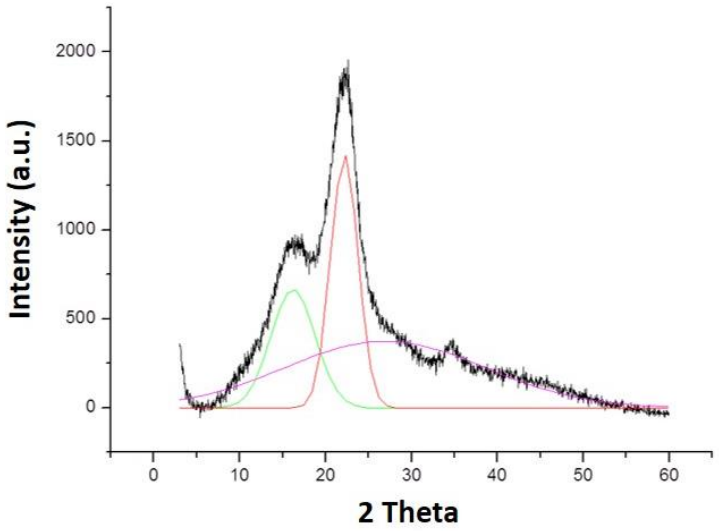

Fig. 6. Diffractogram of the lignocellulosic fiber from green coconut

Peaks at 2θ values of 14.6°, 16.2°, and 22.4° are cellulose, and correspond to the inter planar spacing of the three main planes (110), (110), and (200) of the monoclinic unit cell of cellulose (de Oliveira and Marques 2014). These values were determined in the present study to be 5.99 Å, 5.40 Å, and 3.95 Å, respectively.

To determine the crystallinity index of the fibers, peaks corresponding to the crystalline (highly ordered) and most diffused amorphous (disordered) areas had to be considered (Frost *et al*. 2009). A clear transition occurs from an orderly arrangement of the cellulose chains to a more disordered or amorphous state (Tanobe 2003). Thus, the maximum peaks that were observed in the present study were produced by specific planes of the crystalline areas and these corresponded to the peaks centered in the diffraction angles (2θ) of 16.19°, 22.14°, and 34.73°.

The crystallinity index calculated using Eq. 1 (Tanobe 2003) was 47.82%. This value clearly differed from the values reported for Brazilian green lignocellulosic coir fiber by de Oliveira and Marques (2014) (44%) and of brown fibers reported by Tomczak *et al.* (2007) (51%, and 57%). The above differences in the crystallinity values could be due to the equation used in these studies. It may be noted that such variations in the crystallinity index of coconut fibers as reported by different researchers depend on the conditions used to obtain the spectra and the appropriate equation. For instance, Carvalho *et al*. (2010) obtained a crystallinity index of 29.93% for green coconut fiber by using another equation. These values are different from those reported for brown fibers from the Caribbean (21.59%) (Mathura *et al*. 2014), India (15% to 45%), and other countries (Rowell *et al*. 2000; Satyanarayana *et al*. 2008).

Tensile properties

Figure 7 shows a typical load-displacement curve for green lignocellulosic coconut fibers. The curve shows the typical nature of some natural fibers and reveals a two-phase phenomenon.

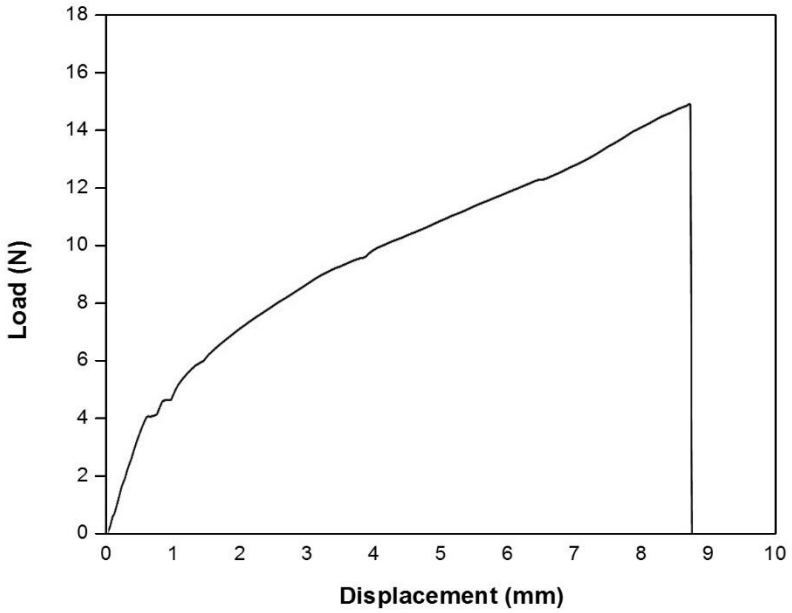

Fig. 7. Typical load-deformation curve of the lignocellulosic fibers from green coconut

The curve was almost similar to those reported for Indian brown coir fiber obtained by a retting process (Satyanarayana and Wypych 2007; Satyanarayana *et al.* 2007; Satyanarayana *et al.* 2008). It was also similar to coir fibers from different countries, which were used to prepare composites (Bakri and Eichhorn 2010; Esmeraldo *et al.* 2010; Ali 2011). This curve suggested a relatively high elongation and was attributed to the resulting stress relaxation of already existing residual stresses during the fiber loading or self alignment of ultimate fibers or micro fibrils during deformation (Everitt *et al.* 2013).

From the above curve, the UTS, YM, and elongation of the tested fibers were calculated. The YM values were obtained from the initial linear part of the curve, while the UTS values were evaluated from the high plastic region. These values are given in Table 4. They were compared with values from published reports on both brown and green lignocellulosic coir fibers.

Table 4. Comparison of Dimensions and Tensile Properties of Green and Brown Coir Fibers

Diameter (mm)	Length (mm)	YM (GPa)	UTS (MPa)	Elongation (%)	Fiber Type	Reference
0.39 ± 0.05 (Thick) 0.25 ± 0.02 (Thin)	- -	1.167 ± 0.272 1.959 ± 0.199	113.98 ± 12.65 158.54 ± 26.41	43.50 ± 7.73 41.28 ± 4.60	Green	Present Study
0.225	5 - 25	1.26 - 2.73	118.3 - 142.6	-	Brown	Tomczak *et al.* (2007)
0.21	-	2.8	107	37.7	Brown	Agopyan *et al.* (2005)
0.10 - 0.40	60 - 50	-	15 - 327	17.6 - 75	Brown	Ramakrishna and Sundarajan (2005a); Ramakrishna and Sundarajan (2005b)
0.27 ± 0.073	50 ± 10	2.0 ± 0.3	142 ± 36	24 ± 10	Brown r	Li *et al.* (2006); Li *et al.* (2007)
0.11 - 0.53	-	2.50 - 4.50	108 - 252	13.7 - 41	Green	Toledo Filho *et al.* (2005)
0.12 ± 0.005	-	3.7 ± 0.6	158	-	Brown	Munawar *et al.* (2007)
0.1 - 0.4	-	16 - 26	174	10 - 25	Green r	Reis (2006)
	-	8	95 - 118	-	Brown	Sen and Reddy (2011)
		0.8 - 1.6	82 - 129	-	Green	Carrijo *et al.* (2002)
			75 - 140	20 - 40	Green	van Dam *et al.* (2006)
0.11 - 0.46	20 - 50	1.74 - 2.78	132 - 139	34.18 - 67.77	Brown	Mathura *et al.* (2014)
-	-	-	204.39	3.17	Brown	Khan and Alam (2012)

Table 4 shows that the fibers with a smaller diameter (0.25 mm) had higher UTS and YM values compared with those of the larger diameter fibers (0.39 mm). Furthermore, it was also observed that these properties were lower than those of other lignocellulosic fibers, such as jute, pineapple, sisal, *etc*. (Munawar *et al.* 2007; Santos *et al.* 2009). This was understandable because the tensile properties depend on the dimensions of the fibers, chemical composition, micro fibril angle, crystallinity, *etc*. Also, it has been reported that despite the similarity of the cellulose structure in all lignocellulosic fibers, there is a variation in the degree of polymerization. This in turn is related to the mechanical properties of the fibers (Khan and Alam 2012).

Furthermore, the values obtained in the present study agreed with those reported by Munawar *et al*. (2007) and Tomczak *et al*. (2007) for green coconut fibers. These researchers observed a decreasing UTS value with an increasing coconut fiber diameter. However, the values were higher than those obtained for coir fibers from Brazil reported by Corradini *et al*. (2009) (UTS: 82 MPa to 129 MPa; YM: 801 MPa to 1,600 MPa), lignocellulosic coir fibers from Brazil reported by Satyanarayana *et al.* (2007) (YM: 2.5 GPa to 4.5 GPa and UTS: 13.7 MPa to 41 MPa), and coconuts from the Philippines reported by van Dam *et al*. (2006) (TS: 75 MPa to 140 MPa; elongation: 20% to 40%). Further, the reported values were also in agreement with the conclusions of Santos *et al.* (2009) and Munawar *et al*. (2007), who stated that coconut fibers exhibited lower tensile properties than other lignocellulosic fibers, such as sisal, jute, pineapple, *etc*. In the case of the elongation (strain at failure), although a slight variation was seen between various researchers, it was considered roughly constant at approximately 42%, which was again slightly lower than that observed in the case of Indian brown coir fibers (~45%) (Satyanarayana *et al.* 2007).

The variations in the characteristics are dependent on the conditions and methods used for determining, including the clamping length, area, elongation, and modulus (Everitt *et al.* 2013). Among various factors, the variation in the diameter of the fiber (standard deviation) was reported to affect the tensile strength. There are reports stating a well-known inverse relationship between the clamping length and strength of the fiber. This is attributed to the weakening of the fiber due to an increase in existing defects because the weakest point of the fiber is the most probable point of breakage (Nechwatal *et al.* 2003; Defoirdt *et al.* 2010; Osorio *et al.* 2011).

Similarly, an inverse relationship exists between the length and strain to failure of the fiber, which has been attributed to the same factors mentioned above. Another factor, which is reported to be the reason for the variation of tensile properties during single fiber testing, is the measurement of the area under the stress-strain curve. Variation in elongation values is due to slippage of the fiber from the fixture (bonding by adhesive), which is difficult to control directly (Defoirdt *et al.* 2010).

Finally, lignocellulosic fibers exhibit high specific tensile properties compared with many inorganic fibers (Yao *et al.* 2008). In the case of coconut fiber, despite the limitation of lower tensile properties compared with other natural fibers (with the exception being the high breaking strain), it is considered to be an attractive reinforcing fiber for various polymers. This is because of some of its unique characteristics, which include a high durability to microbial attacks and high resistance to applied loads, result in a very high elongation. Elongation is an important factor in applications that involve repeated tension efforts, flexing, and relaxation (Khalil *et al.* 2006). Accordingly, the authors of this study have used the fibers of green coconuts from Brazil in the preparation of green composites (Lomelí-Ramírez *et al.* 2011, 2014).

CONCLUSIONS

1. The diameter of the green lignocellulosic coconut fiber was larger at one end, and decreased along the length of the fiber to the other end. The fiber, being multi-cellular, has a cell length of 942.5 μm ± 45.9 μm, cell wall thickness of 3.57 μm ± 0.48 μm, and lumen size of 11.75 μm ± 0.20 μm.
2. The true density of the fibers found in this study was 1200 kg/m^3, which was in the range of values reported by other studies.
3. The obtained lignin content of 32% to 33% and total extracts content of 5.82% for the fibers in this study were in the content ranges reported for green coconut fibers in the literature. However, the ash content was lower than that reported by other studies.
4. XRD spectrum obtained for the green coir fiber indicated that these fibers contain type I cellulose of the amorphous type, which gives rise to XRD results lying in the region of 18-19^o and 13-15^o. The crystallinity index of the fiber was found to be approximately 48%, which was different from the values reported in the literature for Brazilian coconut fibers.
5. Green coconut fibers with a smaller diameter (0.25 mm) showed higher values for the UTS (158.54 MPa ± 6.41 MPa) and YM (1959.45 MPa ± 98.87 MPa) when compared with those of the larger diameter fibers (0.39 mm) (UTS: 113.98 MPa ± 2.65 MPa; YM: 1166.86 MPa ± 271.76 MPa). In contrast, the elongation did not show any remarkable variation (41.28% ± 4.60% to 43.50% ± 7.73%) between the two fiber types.
6. Green coconut fibers, despite their limitation of lower tensile properties compared with other natural fibers (except for the high breaking strain) are considered to be an attractive reinforcing fiber for various polymers because of some of its unique characteristics as mentioned earlier in the Introduction (See pages 2&3). The present authors have themselves used these fibers from Brazil in the preparation of green polymer composites, which has been reported in other studies.

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Conflicts of Interest

There are no conflicts of interest for any of the authors of this paper.

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