

Physicochemical Characterization of Natural Fibers Obtained from Seed Pods of *Ceiba aesculifolia*

Ulises Carranza-Nuñez,^a Salomon Ramiro Vasquez-Garcia,^{a,*} Nelly Flores-Ramirez,^b Hamdy Ahmed Abdel-Gawwad,^c José Luis Rico,^a Arlette Arizbe Santiago,^d Joel Vargas,^e and José Cruz-de-León^b

Characterization of *Ceiba aesculifolia* (CA) fibers by various techniques is herein reported. The seed pods were collected, and the fibers surrounding the seeds were characterized or treated in an oven at 100 °C prior to characterization by scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and thermogravimetric analysis coupled with a differential scanning calorimeter (TGA-DSC). The SEM micrographs showed that the natural material is comprised of tubes of external diameter of approximately 27 µm and a mean wall thickness of about 0.62 µm. The results also indicated that the tubes begin to decompose at approximately 220 °C.

Keywords: *Ceiba aesculifolia*; SEM; FTIR; DRX; TGA-DSC

Contact information: a: Department of Chemical Engineering, Universidad Michoacana de San Nicolás de Hidalgo, Morelia, 58030, México; b: Department of Wood Engineering and Technology, Universidad Michoacana de San Nicolás de Hidalgo, Morelia, 58030, México; c: Raw Building Materials Technology and Processing Research Institute, Housing and Building National Research Center (HBRC), Cairo, Egypt; d: Escuela Nacional de Estudios Superiores, Unidad Morelia, Universidad Nacional Autónoma de México, Morelia, 58190, México; e: Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México, Morelia, 58190, México; *Corresponding author: salomon_vg@yahoo.com

INTRODUCTION

Natural fibers obtained from plants and animals are used in many applications. In nature, vegetal fibers commonly contribute to the stability of the plant, or they can serve as a protection in the case of animal fibers. The world natural fiber production was approximately 33 million tons in 2013. Among this 78.8% was cotton linter, 10% of jute, 3.6% of clean wool, 2.7% of coir, and 4.8% of other fibers such as hemp, kapok, abaca, flax, silk, ramie, and sisal (Townsend and Sette 2016). This information clearly shows the importance of vegetal fibers. These fibers may present interesting properties, for instance, high tensile and low bending and torsional rigidities. In addition, vegetal fibers with low density, are unexpansive, biodegradable, non-toxic, and easy to handle (Wambua *et al.* 2003; Cheung *et al.* 2009; Dittenber and GangaRao 2012). Due to these properties, vegetal fibers are used in many applications including housing, clothing, and packing. One recent and growing application of vegetal fibers is in the production of composites, for instance as a reinforcement of synthetic polymeric materials (Wambua *et al.* 2003; Cheung *et al.* 2009; Zakikhani *et al.* 2014; Djafari Petroudy 2017). Synthetic fibers present excellent mechanical properties that can be tuned according to the final application. However, these materials frequently possess ecological problems during disposal, due to their high stability under atmospheric and environmental conditions. Characterization of several vegetal fibers has been reported in the last decades (Zeriahene *et al.* 1998; Fiore *et al.* 2014; Reddy *et al.*

2014; Belouadah *et al.* 2015; Porras *et al.* 2015; Mohamad *et al.* 2017), in addition to the well-documented cotton. *Ceiba aesculifolia* (CA) is a neotropical species that belongs to the Males order, and to the Bombacaceae family; it is distributed from Mexico to Costa Rica (Cascante-Marín 1997; Herrerías-Diego *et al.* 2008). This tree gives beautiful flowers and can reach 15 m high. The thin trunk is protected with strong thorns. The ellipsoid fruits have seed pods of about 15 cm long and 8 cm wide that keep the seeds covered with abundant soft and white fibers (Carranza-González and Blanco-García 2000; Avendaño *et al.* 2006). These fibers are commonly used to make pillows, and the bark, stem, and branches are used as firewood (Carranza-González and Blanco-García 2000). The number of pods that a *Ceiba aesculifolia* can produce every season vary according to the age of the tree and the environmental conditions. A tree can give tens or hundreds of pods. The relative abundance of this tree in our region, the basic usage of its constituents and the scarce publications related to this tree motivated the present research interest. Furthermore, the white fibers of this tree resemble those of cotton buds, and their characterization was performed for the first time in the present study. The results will undoubtedly set the basis to allow envisage new possible applications of these fibers.

EXPERIMENTAL

Fibers from *Ceiba aesculifolia*

Figure 1 shows an open brown CA pod, which contains a husk, fibers, and seeds. As can be observed, the fibers resemble those of the raw cotton. Their color is bright white and the texture is smooth.

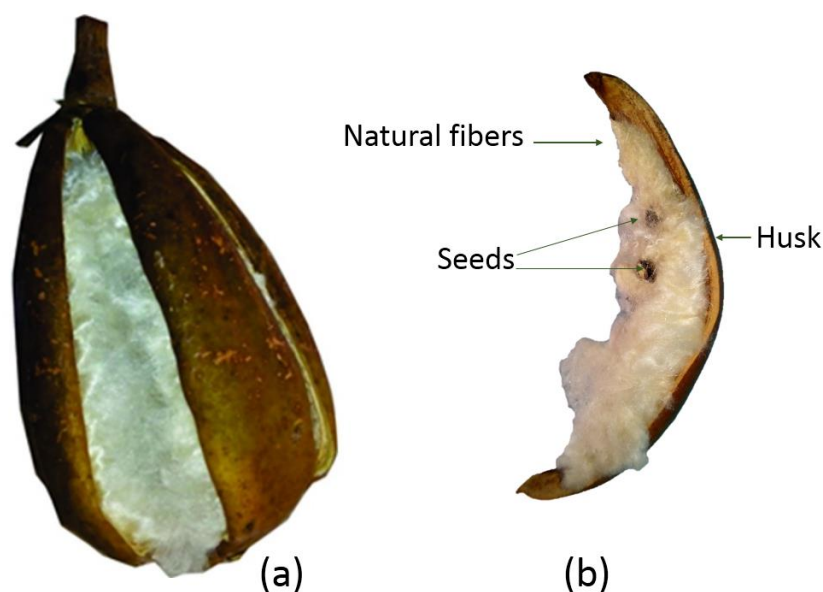


Fig. 1. *Ceiba aesculifolia* pod (a) and white natural fibers around the seeds (b)

The bunches of fibers used in the present study were collected after drying 10 pods under shade for a couple of months. Separation of seeds, husk, and fibers was performed. From these pods, 4.3, 8.3, and 87.4 wt% were determined for fibers, seeds, and husks, respectively. The results are in the range of those reported by Velázquez-Rosas *et al.*

(2017). The fibers were then collected and kept for further characterization. Some were directly characterized, labelled as natural fibers (NF), or were washed with deionized water prior to further characterization. The washed fibers were denoted as WF.

Characterization

The aim of washing the fibers with deionized water was to remove some extractives and alkali metals from this material (Benítez-Guerrero *et al.* 2014; Rocha *et al.* 2017; He *et al.* 2018). By treating the fibers under dissimilar procedures, differences in fiber features were expected. A scanning electron microscope (SEM) (JSM-7600F, JEOL Ltd., Tokyo, Japan) provided with an energy dispersive X-ray spectrometer (EDS) was used to find fiber morphology and composition. To determine the mean diameter and wall thickness, 20 fibers were observed under the microscope. The FTIR spectra of the previously ground fibers were acquired directly with no KBr. The equipment used for this purpose was a Perkin Elmer Spectrum 400 (Waltham, MA, USA) provided with an attenuated total reflectance (ATR) accessory. Data were acquired in 64 scans with a resolution of 4 cm^{-1} in the range of 650 to 4000 cm^{-1} with a germanium crystal. The thermal stability of the fibers was evaluated in a thermal gravimetric analyzer (TGA)-differential scanning calorimeter (DSC) device (STA 449 F3 Jupiter; Netzsch, Selb, Germany). In a typical experiment, 6 mg of natural fibers was collocated into an aluminum crucible and placed into the TGA chamber to be treated under nitrogen flow from ambient temperature to $600\text{ }^{\circ}\text{C}$ at a heating rate of $5\text{ }^{\circ}\text{C}/\text{min}$.

RESULTS AND DISCUSSIONS

Natural and washed Fibers

The following figure shows photographs of *Ceiba aesculifolia*.



Fig. 2. Photographs of *Ceiba aesculifolia*: tree (a), flower (b), and pods (c)

Figure 3 presents images of the NF and WF obtained by SEM. No apparent differences were observed. These figures show that the fibers present in the pods of this tree are indeed comprised of tubes of centimeters in length. A mean diameter of *ca.* 27 μm and a wall thickness of *ca.* 0.62 μm were calculated after analyzing various SEM images.

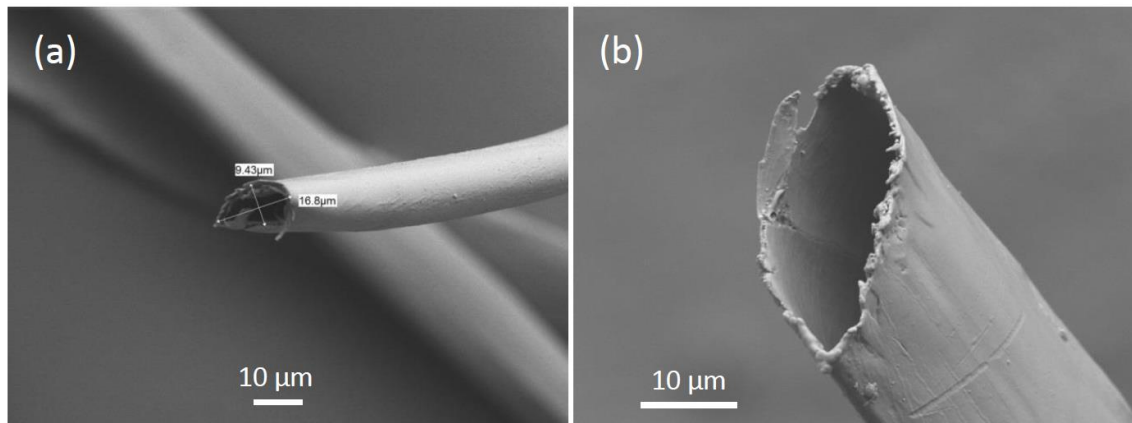


Fig. 3. SEM images of the natural tubes found in pods of the CA tree at two magnifications: 1000X (a) and 2000X (b)

FTIR

It is expected that the development of the fibers of the CA pods follows various stages, as those for cotton fibers. For the latter fibers, the overlapping stages are differentiation, initiation, polar elongation, secondary cell wall development, and maturation. The FTIR spectra for cotton wool at different periods were measured and showed that vibration positions shift slightly during biogenesis (Nelson and Mares 1965; Sathishkumar *et al.* 2013). Figure 4 shows the FTIR spectra of the NF and WF.

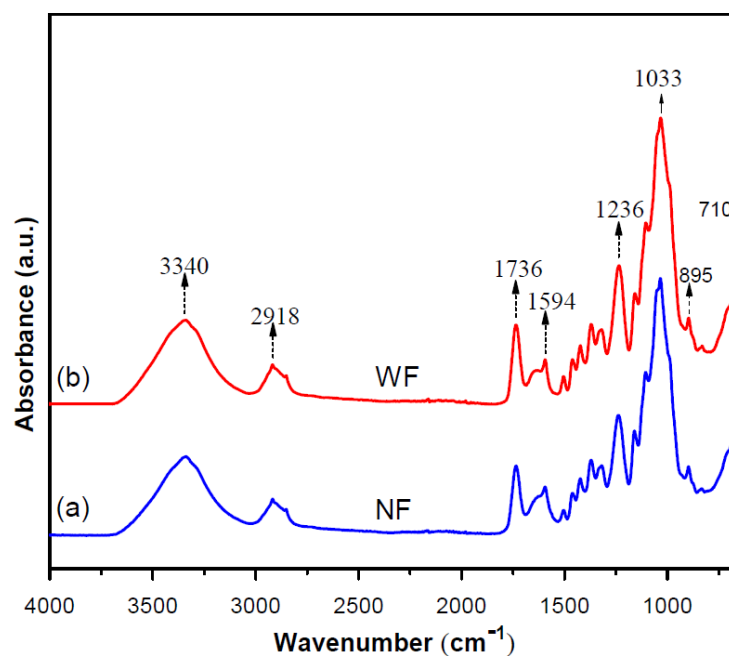


Fig. 4. FTIR spectra of the natural (a) and washed fibers (b)

The FTIR spectra show that fibers of the CA pods are typically composed of cellulosic material, such as those reported for cotton by Abidi *et al.* (2014) and for *Lygeum spartum* L. fibers reported by Belouadah *et al.* (2015). Both spectra in Fig. 4 look similar, suggesting that washing was unnecessary. However, other physical properties which were not measured, such as tensile strength and bending, might be changed by this treatment. The broad band with a maximum at 3340 cm^{-1} was attributed to -OH stretching vibrations resulting from hydrogen bonding in cellulose, whereas those at approximately 2918 and 2850 cm^{-1} that are overlapped, were assigned to -CH₂ asymmetrical and symmetrical stretching, respectively (Nelson *et al.* 1965) and could result from the presence of wax substances on the surface of the primary cell wall (Abidi *et al.* 2008). The peaks at approximately 1736 , 1640 , and 1594 cm^{-1} are attributed to C=O stretching of lignin and hemicellulose (Sathishkumar *et al.* 2013), while that at approximately 1594 cm^{-1} is assigned to the C=C groups of lignin (De Rosa *et al.* 2011). The signal at 1236 cm^{-1} was attributed to -COO groups of cellulose (Reddy *et al.* 2014). The peak at 1033 cm^{-1} is attributed to the stretching mode of C-O of cellulose (Ilharco *et al.* 1997); the signal located at 1157 cm^{-1} is assigned to the stretching vibration of bridge C-O-C of cellulose and hemicellulose (De Rosa *et al.* 2011), whereas that at 1104 cm^{-1} is due to anti-symmetric in-plane ring stretching band (Ilharco *et al.* 1997). The vibration at 1236 cm^{-1} is attributed to C=O stretching or NH₂ deformation (Nelson *et al.* 1965). The peak at approximately 895 cm^{-1} is assigned to the β -linkage (Alonso-Simón *et al.* 2011). The vibration located at 710 cm^{-1} is attributed to CH₂ rocking vibration in cellulose I β (Akerholm *et al.* 2004), which is also characteristic of the cellulose in native cotton (Abidi *et al.* 2014).

Thermogravimetric Analysis

The thermal stability of the fibers obtained from the pods of the CA tree was evaluated using TGA-DTG equipment. The profile for WF was similar to that for NF and it is not shown. Figure 5 displays the thermogravimetric profile of NF.

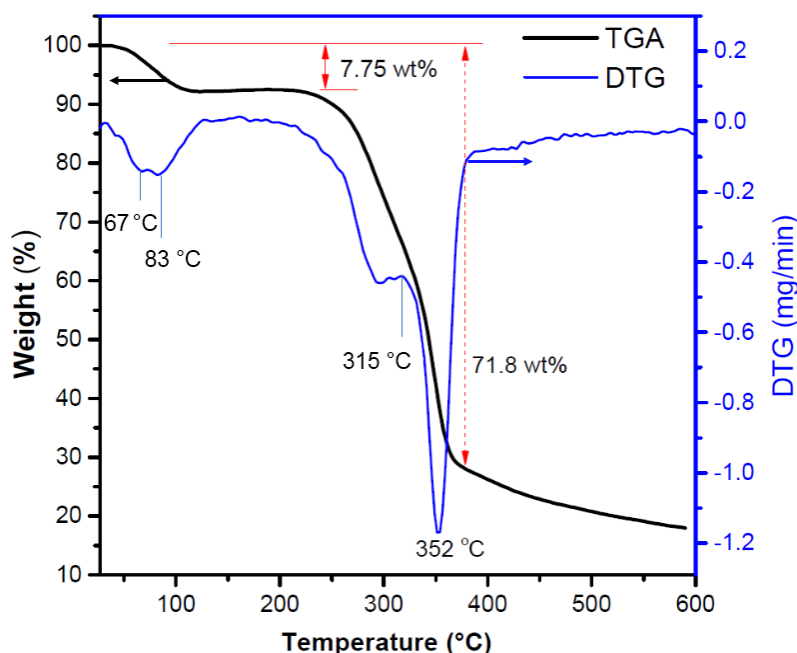


Fig. 5. TGA-DTG profiles of the NF from the pods of the CA tree

Three main zones of weight reduction were observed in this figure. The first was related to the release of physisorbed water and occurred at temperature below 125 °C with a total weight decrease of 7.75 wt%. The second weight decrease can be divided into two steps; one took place from approximately 210 to 315 °C and is assigned to the elimination of hemicellulose, and the glycosidic bonds of cellulose (Indran *et al.* 2014). The other occurred from 315 to 377 °C and is ascribed to cellulose I and α -cellulose (Fiore *et al.* 2014). The latter step exhibited the highest reduction rate of the whole thermal treatment. The total reduction in the second zone was 63.1 wt%. The third zone of weight reduction was observed above 377 °C and is attributed to the decomposition of lignin. The total reduction in the entire thermal treatment was 82.25 wt%. The results are in agreement with previous research studies (Broido 1969; Seki *et al.* 2013; Belouadah *et al.* 2015; Mohamad *et al.* 2017). Seki *et al.* (2013) reported for the thermal decomposition of *Ferula communis* fibers that cellulose decomposes in the temperature range of 240 to 350 °C, hemicellulose from 200 to 260 °C, and lignin from 280 to 500 °C. In the research work published by Belouadah *et al.* (2015) and Mohamad *et al.* (2017), the authors reported a similar thermogravimetric profile for the cellulosic fiber from *Lygeum spartum* which is in agreement to ours in the present study.

With the aim to obtain the apparent activation energy, the method reported by Broido (1969) and Saravanakumar *et al.* (2013) was followed. To this purpose, the TGA-DSC results were used and plotted using the following equation,

$$\ln \left[\ln \left(\frac{1}{y} \right) \right] = -\frac{E}{R} \left[\left(\frac{1}{T} + K \right) \right] \quad (1)$$

where R is the ideal gas constant, K is a constant, E is the apparent activation energy (KJ/mol), T is the absolute temperature (Kelvin), and y is the normalized weight, defined as w_0/w , being w_0 the initial sample weight (g) and w the weight (g) at any time. When plotting the experimental data using the above equation, a straight line with correlation coefficient of 0.9998 was obtained. From the graph, the apparent activation energy of 96.8 kJ/mol was calculated. It is worth commenting that activation energies for other natural materials are in the range 60 to 170 kJ/mol (Yao *et al.* 2008; Saravanakumar *et al.* 2013; Indran *et al.* 2014; Belouadah *et al.* 2015). Indran *et al.* (2014) reported an apparent activation energy of 74.2 kJ/mol for *C. quadrangularis* natural fibers; Saravanakumar *et al.* (2013a) of 76.7 kJ/mol for *P. juliflora* natural fibres; Belouadah *et al.* (2015) of 68.8 kJ/mol for *Lygeum spartum* L. natural fibers, and Yao *et al.* (2008) reported values of 160-170 kJ/mol for wood, bamboo, agricultural residue, and bast fibers. Table 1 exhibits additional values reported for other fibers.

X-Ray Diffraction

Native cellulose is believed to possess only a unique structure, called cellulose I. However, it was proposed that it consists of two crystalline allomorphs, I_α and I_β (Atalla and VanderHart 1984). According to literature, cellulose I_α presents a triclinic one-chain unit cell ($a = 6.717 \text{ \AA}$, $b = 5.962 \text{ \AA}$, $c = 10.400 \text{ \AA}$, $\alpha = 118.08^\circ$, $\beta = 114.80^\circ$, and $\gamma = 80.37^\circ$) (Sugiyama *et al.* 1991) with parallel cellulose chains that are piled up by Van der Waals interactions. In addition, the structure shows a progressive shear along the chain axis. Cellulose I_β presents a monoclinic two-chain unit cell ($a = 7.784 \text{ \AA}$, $b = 8.201 \text{ \AA}$, $c = 10.38 \text{ \AA}$, $\alpha = \beta = 90^\circ$, and $\gamma = 96.5^\circ$) (Nishiyama *et al.* 2002) with stacking of parallel chains and alternating shear (Sugiyama *et al.* 1991). Furthermore, cellulose I_α can be transformed into cellulose I_β by hydrothermal treatment or with solvents following various treatments

(Debzi *et al.* 1991). The X-ray diffraction pattern of the fibers around the seeds of the CA tree is presented in Fig. 6, which is characteristic of cellulosic materials. Since the XRD pattern for WF was similar to that for NF, it is not shown. The main reflections occurred at approximately 14.0 , 22.0 , and 34.5° (2θ). The assigning of reflections to crystal planes of a natural material was not straightforward due to the presence of cellulose, hemicellulose, and lignin. The broad reflection at approximately $14^\circ 2\theta$ was composed of overlapped signals, for instance, the peaks at approximately 10.5 , 14.7 , and $16.9^\circ 2\theta$ were assigned to the Miller indexes of (001), (100), and (010) of cellulose I_α , and those at approximately 14.9 , $16.8^\circ 2\theta$ for (1-10) and (110) planes of cellulose I_β , could well be fitted within this region. Similarly, for reflections at 22 and $34.2^\circ 2\theta$. Both phases may therefore be present in the material; however, more characterization would be needed to elucidate this. It has been reported that cellulose I_β is dominant in higher plants, whereas cellulose I_α phase was only found in algal and bacterial cellulose (Igarashi *et al.* 2007; Habibi *et al.* 2010). However, Atalla and VanderHart (1984) found several anomalies in the nuclear magnetic resonance (NMR) spectra of celluloses from vascular plants, algae, bacteria, and tunicates, which seem to indicate that vascular plants may contain only cellulose I_β . Figure 6 shows the XRD pattern of the NF from the pods of CA tree and the reflections of the cellulose I_β phase as a reference (French 2014).

Following the method reported by Segal *et al.* (1959), the index of crystallinity can be calculated by taking the heights of the intensity of the reflection at approximately $22^\circ 2\theta$ and that of the amorphous at approximately $18^\circ 2\theta$. The calculated index of crystallinity was 0.63. The higher this value the greater the fiber resistance to bacteria, thermal, and chemical treatments (Ma *et al.* 2012; Lu *et al.* 2016; Mohamad *et al.* 2017).

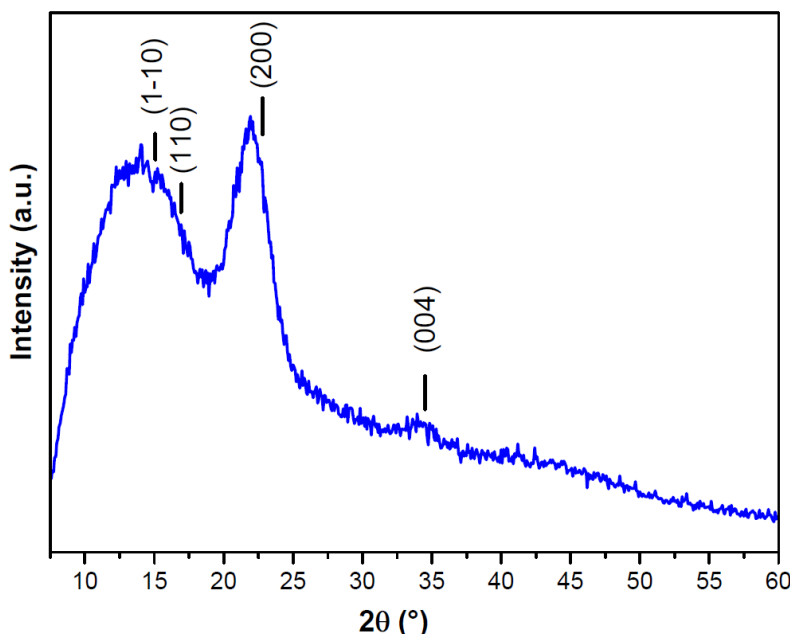


Fig. 6. The XRD pattern of the natural fibers surrounding the seeds of the CA tree and planes of reference for cellulose I_β

Table 1 summarizes some properties of the fibers of the CA tree determined in the present study and compares the values to those of other fibers reported in the literature.

Table 1. Characterization of the Fibers Covering the Seeds of the CA Tree Compared to Other Cellulose Fibers

Fibers	Mean fiber diameter (μm)	Mean wall thickness (μm)	Apparent activation energy (kJ/mol)	Crystallinity index (%)
Cotton (neat)	16.8 ^a	3.9	178 ^b	0.64 ^c
CA-present study	27	0.62	96.85	63
Eucalyptus wood	13.28	3.0	184.9 to 229.7 ⁱ	68 ^e
Flax	19 ^e	10 ^e	76.64 ^h	50 to 90 ^e
Pine (softwood)	20 to 40 ^d	8 to 11 ^e	161.5 ^f	52 to 62 ^e
Hemp	25 ^e	10 ^e	180.9 ^f	50 to 90 ^e
Jute	20 ^e	5.2 ^d	183.1 ^f	71 ^g
Bamboo	14 ^e	7 ^e	164.1 ^f	45.33 ^g

^a(Graham and Haigler 2021); ^b(Cabralés and Abidi 2010); ^c(Wang *et al.* 2014); ^d(Monti and Alexopoulou 2013); ^e(Fan and Fu 2017); ^f(Yao *et al.* 2008); ^g(Moshi *et al.* 2020); ^h(Vincent *et al.* 2015); ⁱ(Poletto *et al.* 2012); ^j(Wentze *et al.* 2019).

CONCLUSIONS

1. The characterization of the fibers covering the seeds found in the seed pods of the *Ceiba aesculifolia* (CA) tree is herein reported. The fibers are comprised of microtubes of millimeters in length, with a mean external diameter of about 27 μm and a wall thickness of 0.62 μm .
2. The CA tubes showed a characteristic Fourier transform infrared (FTIR) spectrum and the X-ray diffraction (XRD) pattern is similar to those reported for other cellulosic materials. The determined crystallinity index was 0.63.
3. The thermogravimetric analysis – differential scanning calorimetry (TGA-DSC) results indicated that the tubes are resistant below 220 °C, and above this temperature, the structure eventually collapsed and decomposed. Various applications of this natural source are envisaged, for instance, as an acoustic or electrical insulation, and as reinforcement of other polymeric materials. Furthermore, the tubes could be used as templates to synthesize other interesting inorganic structures, such as silica and titania, among others.

ACKNOWLEDGMENTS

The authors acknowledge the Scientific Research Council of the Universidad Michoacana de San Nicolas de Hidalgo (CIC-UMSNH) for their financial support.

REFERENCES CITED

- Abidi, N., Cabralés, L., and Haigler, C. H. (2014). “Changes in the cell wall and cellulose content of developing cotton fibers investigated by FTIR spectroscopy,” *Carbohydr. Polym.* 100, 9-16. DOI: 10.1016/j.carbpol.2013.01.074
- Abidi, N., Hequet, E., Cabralés, L., Gannaway, J., Wilkins, T., and Wells, L. W. (2008). “Evaluating cell wall structure and composition of developing cotton fibers using

- Fourier transform infrared spectroscopy and thermogravimetric analysis,” *J. Appl. Polym. Sci.* 107(1), 476-486. DOI: 10.1002/app.27100
- Åkerholm, M., Hinterstoisser, B., and Salmén, L. (2004). “Characterization of the crystalline structure of cellulose using static and dynamic FT-IR spectroscopy,” *Carbohydr. Res.* 339(3), 569-578. DOI: 10.1016/j.carres.2003.11.012
- Alonso-Simón, A., García-Angulo, P., Mérida, H., Encina, A., Álvarez, J. M., and Acebes, J. L. (2011). “The use of FTIR spectroscopy to monitor modifications in plant cell wall architecture caused by cellulose biosynthesis inhibitors,” *Plant. Signal. Behav.* 6(8), 1104-1110. DOI: 10.4161/psb.6.8.15793
- Atalla, R. H., and VanderHart, D. L. (1984). “Native cellulose: A composite of two distinct crystalline forms,” *Science* 223(4633), 283-285. DOI: 10.1126/science.223.4633.283
- Avendaño, A., Casas, A., Dávila, P., and Lira, R. (2006). “Use forms, management and commercialization of “pochote” *Ceiba aesculifolia* (H.B. & K.) Britten & Baker f. subsp. *parvifolia* (Rose) P.E. Gibbs & Semir (Bombacaceae) in the Tehuacán Valley, Central Mexico,” *J. Arid. Environ.* 67(1), 15-35. DOI: 10.1016/j.jaridenv.2006.02.004
- Belouadah, Z., Atib, A., and Rokbi, M. (2015). “Characterization of new natural cellulosic fiber from *Lygeum spartum* L.,” *Carbohydr. Polym.* 134, 429-437. DOI: 10.1016/j.carbpol.2015.08.024
- Benítez-Guerrero, M., López-Beceiro, J., Sánchez-Jiménez, P. E., and Pascual-Cospa, J. (2014). “Comparison of thermal behavior of natural and hot-washed sisal fibers based on their main components: Cellulose, xylan and lignin. TG-FTIR analysis of volatile products,” *Thermochim. Acta.* 581, 70-86. DOI: 10.1016/j.tca.2014.02.013
- Broido, A. (1969). “A simple, sensitive graphical method of treating thermogravimetric analysis data,” *J. Polym. Sci. A-2: Polym. Phys.* 7(10), 1761-1773. DOI: 10.1002/pol.1969.160071012
- Cabral, L., and Abidi, N. (2010). “On the thermal degradation of cellulose in cotton fibers,” *J. Therm. Anal. Calorim.* 10(2), 485-491. DOI: 10.1007/s10973-010-0911-9
- Carranza-González, E., and Blanco-García, A. (2000). “Bombacaceae [Bombacaceae],” in: *Flora del Bajío y de Regiones Adyacentes Fascículo 90*, Instituto de Ecología, A.C. Centro Regional del Bajío (Ed), Pátzcuato, Michoacan, México, pp. 1-13.
- Cascante-Marín, A. (1997). “La familia Bombacaceae (Malvales) en Costa Rica [Bombacaceae (Malvales) family in Costa Rica],” *Brenesia* 47-48, 17-36.
- Cheung, H. Y., Ho, M. P., Lau, K. T., Cardona, F., and Hui, D. (2009). “Natural fibre-reinforced composites for bioengineering and environmental engineering applications,” *Compos. Part B- Eng.* 40(7), 655-663. DOI: 10.1016/j.compositesb.2009.04.014
- De Rosa, I. M., Kenny, J. M., Maniruzzaman, M., Moniruzzaman, M., Monti, M., Puglia, D., Santulli, C., and Sarasini, F. (2011). “Effect of chemical treatments on the mechanical and thermal behaviour of okra (*Abelmoschus esculentus*) fibres,” *Compos. Sci. Technol.* 71(2), 246-254. DOI: 10.1016/j.compscitech.2010.11.023
- Debzi, E. M., Chanzy, H., Sugiyama, J., Tekely, P., and Excoffier, G. (1991). “The I α \rightarrow I β transformation of highly crystalline cellulose by annealing in various mediums,” *Macromolecules* 24(26), 6816-6822. DOI: 10.1021/ma00026a002
- Dittenber, D. B., and GangaRao, H. V. S. (2012). “Critical review of recent publications on use of natural composites in infrastructure,” *Compos. Part A- Appl. S.* 43(8) 1419-1429. DOI: 10.1016/j.compositesa.2011.11.019

- Djafari Petroudy, S. R. (2017). "Physical and mechanical properties of natural fibers," in: *Advanced High Strength Natural Fibre Composites in Construction*. Woodhead Publishing, 59-83. DOI: /10.1016/B978-0-08-100411-1.00003-0
- Fiore, V., Scalici, T., and Valenza, A. (2014). "Characterization of a new natural fiber from *Arundo donax* L. as potential reinforcement of polymer composites," *Carbohydr. Polym.* 106, 77-83. DOI: 10.1016/j.carbpol.2014.02.016
- French, A. D. (2014). "Idealized powder diffraction patterns for cellulose polymorphs," *Cellulose* 21, 885-896. DOI: 10.1007/s10570-013-0030-4
- Graham, B. P., and Haigler, C. H. (2021). "Microtubules exert early, partial, and variable control of cotton fiber diameter," *Planta* 253(47), 1-17. DOI: 10.1007/s00425-020-03557-1
- Habibi, Y., Lucia, L. A., and Rojas, O. J. (2010). "Cellulose nanocrystals: Chemistry, self-assembly, and applications," *Chem. Rev.* 110(6), 3479-3500. DOI: 10.1021/cr900339w
- He, M., Yang, G., Chen J., Ji, X., and Wang, Q. (2018). "Production and characterization of cellulose nanofibrils from different chemical and mechanical pulps," *J. Wood Chem. Technol.* 38(2), 149-158. DOI: 10.1080/02773813.2017.1411368
- Herrerías-Diego, Y., Quesada, M., Stoner, K. E., Lobo, J. A., Hernández-Flores, Y., and Sanchez Montoya, G. S. (2008). "Effect of forest fragmentation on fruit and seed predation of the tropical dry forest tree *Ceiba aesculifolia*," *Biol. Conserv.* 141(1), 241-248. DOI: 10.1016/j.biocon.2007.09.017
- Igarashi, K., Wada, M., and Samejima, M. (2007). "Activation of crystalline cellulose to cellulose III results in efficient hydrolysis by cellobiohydrolase," *FEBS J.* 274(7), 1785-1792. DOI: 10.1111/j.1742-4658.2007.05727.x
- Ilharcó, L. M., Garcia, A. R., Lopes da Silva, J., and Vieira Ferreira, L. F. (1997). "Infrared approach to the study of adsorption on cellulose: Influence of cellulose crystallinity on the adsorption of benzophenone," *Langmuir* 13(15), 4126-4132. DOI: 10.1021/la962138u
- Indran, S., Edwin Raj, R., and Sreenivasan, V. S. (2014). "Characterization of new natural cellulosic fiber from *Cissus quadrangularis* root," *Carbohydr. Polym.* 110, 423-429. DOI: 10.1016/j.carbpol.2014.04.051
- Lu, Q., Cai, Z., Lin, F., Tang, L., Wang, S., and Huang, B. (2016). "Extraction of cellulose nanocrystals with a high yield of 88% by simultaneous mechanochemical activation and phosphotungstic acid hydrolysis," *ACS Sustain. Chem. Eng.* 4(4), 2165-2172. DOI: 10.1021/acssuschemeng.5b01620
- Ma, H., Burger, C., Hsiao, B. S., and Chu, B. (2012). "Highly permeable polymer membranes containing directed channels for water purification," *ACS Macro Lett.* 1(6), 723-726. DOI: 10.1021/mz300163h
- Mohamed, M. A., Salleh, W. N. W., Jaafar, J., Ismail, A. F., Mutalib, M. A., Mohamad, A. B., Zain, M. F. M., Awang, N. A., and Hir, Z. A. M. (2017). "Physicochemical characterization of cellulose nanocrystal and nanoporous self-assembled CNC membrane derived from *Ceiba pentandra*," *Carbohydr. Polym.* 157, 1892-1902. DOI: 10.1016/j.carbpol.2016.11.078
- Monti, A., and Alexopoulou, E. (2013). *Kenaf: A Multi-Purpose Crop for Several Industrial Applications. New Insights from the Biokenaf Project*, Springer, Verlag London, UK.
- Moshi, A. A. M., Ravindran, D., Bharathi, S. R. S., Padma, S. R., Indran, S., and Divya D. (2020). "Characterization of natural cellulosic fiber extracted from *Grewia damine*

- flowering plant's stem," *Int. J. Biol. Macromol.* 164, 1246-1255. DOI: 10.1016/j.ijbiomac.2020.07.225
- Nelson, M. L., and Mares, T. (1965). "Accessibility and lateral order distribution of the cellulose in the developing cotton fiber," *Text. Res. J.* 35(7), 592-603. DOI: 10.1177/004051756503500703
- Nishiyama, Y., Langan, P., and Chanzy, H. (2002). "Crystal structure and hydrogen-bonding system in cellulose I β from synchrotron X-ray and neutron fiber diffraction," *J. Am. Chem. Soc.* 124(31), 9074-9082. DOI: 10.1021/ja0257319
- Poletto, M., Zattera, A. J., and Santana, R. M. C. (2012). "Thermal decomposition of wood: Kinetics and degradation mechanisms," *Bioresour. Technol.* 126, 7-12. DOI: 10.1016/j.biortech.2012.08.133
- Porras, A., Maranon, A., and Ashcroft, I. A. (2015). "Characterization of a novel natural cellulose fabric from *Manicaria saccifera* palm as possible reinforcement of composite materials," *Compos. Part B- Eng.* 74, 66-73. DOI: 10.1016/j.compositesb.2014.12.033
- Reddy, K. O., Ashok, B., Reddy, K. R. N., Feng, Y. E., Zhang, J., and Rajulu, A. V. (2014). "Extraction and characterization of novel lignocellulosic fibers from *Thespesia lampas* plant," *Int. J. Polym. Anal. Ch.* 19(1), 48-61. DOI: 10.1080/1023666X.2014.854520
- Rocha, E. P. A., Sermyagina, E., Vakkilainen, E., Colodette, J. L., de Oliveira, I. M., and Cardoso, M. (2017). "Kinetics of pyrolysis of some biomasses widely available in Brazil," *J. Therm. Anal. Calorim.* 130(3), 1445-1454. DOI: 10.1007/s10973-017-6138-2
- Saravanakumar, S. S., Kumaravel, A., Nagarajan, T., Sudhakar, P., and Baskaran, R. (2013). "Characterization of a novel natural cellulosic fiber from *Prosopis juliflora* bark," *Carbohydr. Polym.* 92(2), 1928-1933. DOI: 10.1016/j.carbpol.2012.11.064
- Sathishkumar, T. P., Navaneethakrishnan, P., Shankar, S., and Rajasekar, R. (2013). "Characterization of new cellulose *Sansevieria ehrenbergii* fibers for polymer composites," *Compos. Interface.* 20(8), 575-593. DOI: 10.1080/15685543.2013.816652
- Segal, L., Creely, J. J., Martin, A. E., and Conrad, C. M. (1959). "An empirical method for estimating the degree of crystallinity of native cellulose using the X-ray diffractometer," *Text. Res. J.* 29(10), 786-794. DOI: 10.1177/004051755902901003
- Seki, Y., Sarikanat, M., Sever, K., and Durmuşkahya, C. (2013). "Extraction and properties of *Ferula communis* (chakshir) fibers as novel reinforcement for composites materials," *Compos. Part B- Eng.* 44(1), 517-523. DOI: 10.1016/j.compositesb.2012.03.013
- Sugiyama, J., Vuong, R., and Chanzy, H. (1991). "Electron diffraction study on the two crystalline phases occurring in native cellulose from an algal cell wall," *Macromolecules* 24(14), 4168-4175. DOI: 10.1021/ma00014a033
- Townsend, T., and Sette, J. (2016). "Chapter: Natural fibres and the world economy," in: *Advances in Science and Technology towards Industrial Applications*, R. Fangueiro, and S. Rana (eds.), Springer Nature, Basel, Switzerland, pp. 381-390.
- Velázquez-Rosas, N., Ruiz-Guerra, B., Sánchez-Coronado, M. E., Gamboa-de Buen, A., and Orozco-Segovia, A. (2017). "Morphological variation in fruits and seed of *Ceiba aesculifolia* and its relationship with germination and seedling biomass," *Bot. Sci.* 95(1), 1-11. DOI: 10.17129/botsci.636

- Vincent, S. S., Mahinpey, N., and Mani T. (2015). “Torrefaction of flax straw biomass and its kinetic studies,” *Energ. Source. Part A.* 37(21), 2338-2345. DOI: 10.1080/15567036.2012.714053
- Wambua, P., Ivens, J., and Verpoest, I. (2003). “Natural fibres: Can they replace glass in fibre reinforced plastics?,” *Compos. Sci. Technol.* 63(9), 1259-1264. DOI: 10.1016/S0266-3538(03)00096-4
- Wang, M., She, Y., Xiao, Z., Hu, J., Zhou, R., and Zhang, J. (2014). “The green adsorption of chitosan tripolyphosphate nanoparticles on cotton fiber surfaces,” *Carbohydr. Polym.* 101, 812-818. DOI: 10.1016/j.carbpol.2013.10.023
- Wentze, M., Brischke, C., and Militz H. (2019). “Dynamic and static mechanical properties of *Eucalyptus nitens* thermally modified in an open and closed reactor system,” *Maderas, Cienc. Tecnol.* 21(2), 141-152. DOI: 10.4067/S0718-221X2019005000201
- Yao, F., Wu, Q., Lei, Y., Guo, W., and Xu, Y. (2008). “Thermal decomposition kinetics of natural fibers: Activation energy with dynamic thermogravimetric analysis,” *Polym. Degrad. Stabil.* 93(1), 90-98. DOI: 10.1016/j.polymdegradstab.2007.10.012
- Zakikhani, P., Zahari, R., Sultan, M. T. H., and Majid, D. L. (2014). “Extraction and preparation of bamboo fibre-reinforced composites,” *Mater. Design* 63, 820-828. DOI: 10.1016/j.matdes.2014.06.058
- Zeriahene, N., Prat, R., Goldberg, R., Catesson, A. M., and Harche-Kaid, M. (1998). “Cell walls of seed hairs from *Lygeum spartum*: Ultrastructure, composition and mechanical properties,” *Ann. Bot.* 81(1), 61-66. DOI: 10.1006/anbo.1997.0530

Article submitted: February 21, 2020; Peer review completed: June 13, 2020; Revised version received and accepted: April 2, 2021; Published: April 30, 2021.
DOI: 10.15376/biores.16.2.4200-4211