



Maache, M., Bezazi, A., Amroune, S., Scarpa, F., & Dufresne, A. (2017). Characterization of a novel natural cellulosic fiber from *Juncus effusus* L. *Carbohydrate Polymers*, 171, 163-172.
<https://doi.org/10.1016/j.carbpol.2017.04.096>

Peer reviewed version

License (if available):
CC BY-NC-ND

Link to published version (if available):
[10.1016/j.carbpol.2017.04.096](https://doi.org/10.1016/j.carbpol.2017.04.096)

[Link to publication record on the Bristol Research Portal](#)
PDF-document

This is the author accepted manuscript (AAM). The final published version (version of record) is available online via Elsevier at <http://www.sciencedirect.com/science/article/pii/S0144861717304952>. Please refer to any applicable terms of use of the publisher.

University of Bristol – Bristol Research Portal

General rights

This document is made available in accordance with publisher policies. Please cite only the published version using the reference above. Full terms of use are available:
<http://www.bristol.ac.uk/red/research-policy/pure/user-guides/brp-terms/>

Characterization of a novel natural cellulosic fiber from *Juncus effusus L.*

Mabrouk Maache,¹ Abderrezak Bezazi,¹ Salah Amroune,^{1,2} Fabrizio Scarpa,³ Alain Dufresne^{4*}

¹ Laboratoire de Mécanique Appliquée des Nouveaux Matériaux (LMANM), Université 08 Mai 1945, B.P. 401 Guelma 24000, Algeria

² Département de Génie Mécanique, Université de M'sila BP 166, Algeria

³ Advanced Composites Centre for Innovation and Science (ACCIS) University of Bristol, BS8 1TR Bristol, UK.

⁴ Univ. Grenoble Alpes, CNRS, Grenoble INP, LGP2, F-38000, France

* Corresponding author: alain.dufresne@pagora.grenoble-inp.fr

Keywords: Lignocellulosic fibers, *Juncus effusus L.*, tensile behavior, FTIR, X-ray diffraction.

Abstract

This study aims to assess the morphology and properties of fibers extracted from a wild natural plant largely available in Algeria known as *Juncus effusus L.* (JE). The morphology and diameter of the fiber bundles extracted from the stem of the JE plant were characterized by optical and scanning electron microscopy. The functional groups of the extracted lignocellulosic JE fibers were studied by FTIR, their thermal degradation behavior was investigated by TGA and their crystallinity was determined using X-ray diffraction technique. In addition, mechanical characterization was carried out using tensile tests on the lignocellulosic fiber in order to evaluate their strength, strain at break and Young's modulus. In view of the dispersion in the obtained experimental results, the latter were analyzed using the Weibull statistical laws with two and three parameters.

Introduction

Environmental concerns and governmental legislations tend to redirect the interest of the scientific community towards the use of materials of natural origin, thus focusing the research topics on recyclable, renewable, and sustainable materials with reduced impact on nature. The development of the next generation of materials and processes is therefore strongly influenced by the principles of sustainability, eco-efficiency, and green chemistry (De Rosa, Kenny, Puglia, Santulli & Sarasini, 2010). Lignocellulosic fibers like flax (Baley & Bourmaud, 2014), agave Americana (Bezazi, Belaadi, Bouchak, Scarpa & Boba, 2014), hemp (Beckermann & Pickering, 2009), sisal (Belaadi, Bezazi, Bouchak, Scarpa & Zhu, 2014), and jute (Dobah, Bouchak, Bezazi, Belaadi, Scarpa, 2016; Virk, Hall & Summerscales, 2009) have been proposed as lignocellulosic fibers capable of substituting synthetic fibers, specially glass fibers, in many applications because of their biodegradability, lightweight and good physical and mechanical properties. Indeed, despite some advantages of synthetic fibers for many applications, they have serious disadvantages such as (i) non-renewability, (ii) non-

recyclability, (iii) high energy consumption in the manufacturing process, (iv) health hazard at inhalation and (v) non-biodegradability (Cheung, Ho, Lau, Cardona & Hui, 2009).

Lignocellulosic fibers are used in several sectors such as automotive and packaging (Dittenber & Ganga Rao, 2012; Majeed, Jawaid, Hassan, Bakar, Khalil et al., 2013), but also building (Di Bella, Fiore, Galtieri, Borsellino & Valenza, 2014). Recently, the use of biocomposite materials using lignocellulosic fibers with bio or non-biopolymer has increased due to the advantages of these materials, namely good strength, lightweight, corrosion resistance and low maintenance costs (Azwa, Yousif, Manalo & Karunasena, 2013). These materials are also renewable and have relatively high strength and stiffness, do not cause skin irritation (Hornsby, Hinrichsen & Tarverdi, 1997; Bledzki, Reihmane & Gassan, 1996), and are easy to handle. Despite the advantages offered by these lignocellulosic fibers in composite materials, their use is still limited for non-structural applications under low and medium-scale load conditions because of their low thermal stability, moisture absorption, and low fire resistance. In order to obtain good mechanical characteristics of the fiber, it is recommended to use a suitable extraction method which leads to undamaged fibers, in addition to a chemical or physical treatment aiming at improving their mechanical properties. The literature showed that the extraction technique has a significant effect on the chemical composition of the fibers, their length and their mechanical properties (Sreekala, Kumaran, Joseph, Jacob & Thomas, 2000; Malainine et al., 2003).

A lot of research has been done recently on the subject and new lignocellulosic fibers have been investigated. Morphological characterization, physicochemical, mechanical and thermal properties have been investigated further on new lignocellulosic sources such as *Arundo donax L.* leaf fibers (Scalici, Fiore & Valenza, 2016), *Lygeum spartum L.* (Belouadah, Ati & Rokbi, 2015), *Cissus quadrangularis* stem (Indran & Raj, 2015), *Cissus quadrangularis* root

(Indran, Raj & Sreenivasan, 2014), and *Arundo donax L.* (Fiore, Scalici & Valenza, 2014), among others.

The aim of the present investigation is to introduce a new fiber extracted from the stem of the wild plant *Juncus effusus L.* (JE), for which very few physico-chemical and mechanical characterization works have been carried out, to lay the foundations for future research related to the development of this fiber as potential reinforcement in biocomposite materials and to compare its behavior and characteristics with other lignocellulosic fibers existing in the literature. This lignocellulosic fiber is proposed for the first time to be used as potential reinforcement in biocomposite materials. For this purpose, morphological and physico-chemical characteristics of JE fibers were examined by scanning electron microscopy (SEM), Fourier transform infrared (FTIR) and X-ray diffraction (XRD). In addition, a mechanical characterization of the JE fibers was carried out using fiber bundle tensile tests. Finally, the results were treated by Weibull statistical analysis with two and three parameters.

Materials and experimental procedures

Fiber Extraction

JE plants were collected in Guelma city in the north eastern region of Algeria, during the period of October. The climate in Guelma is subtropical humid, with an average temperature of 6.4 °C in winter and 35.6 °C in summer. During summer the temperature can reach 45 °C and even more (Annual Climatological Report, Guelma, 2014). The JE plants are in the form of a clump of grass (Fig. 1a), consisting of hollow rods of cylindrical shape 4 to 8 mm in diameter (Fig. 1c) filled with a white spongy material. The maximum height is about 1 m.

Juncus effusus L. (JE) is a plant species that is part of the *Juncaceae* family that grows in wet places like river, mountain, etc. The roots and marrow parts of JE have been used as medicinal plants in oriental medicine (Park, Won, Hwang & Han, 2014). The JE rod was formerly used for making mats, ottomans and baskets. The fiber bundles were extracted from

their stem (Fig. 1b) by a manual process after 3 hours of boiling in water, drying at ambient temperature of 24 °C in a dry natural state for one week before being analyzed. The extracted fibers have an average diameter of 280 μm and a length of up to 200 mm. They are shown in Figure 1d.

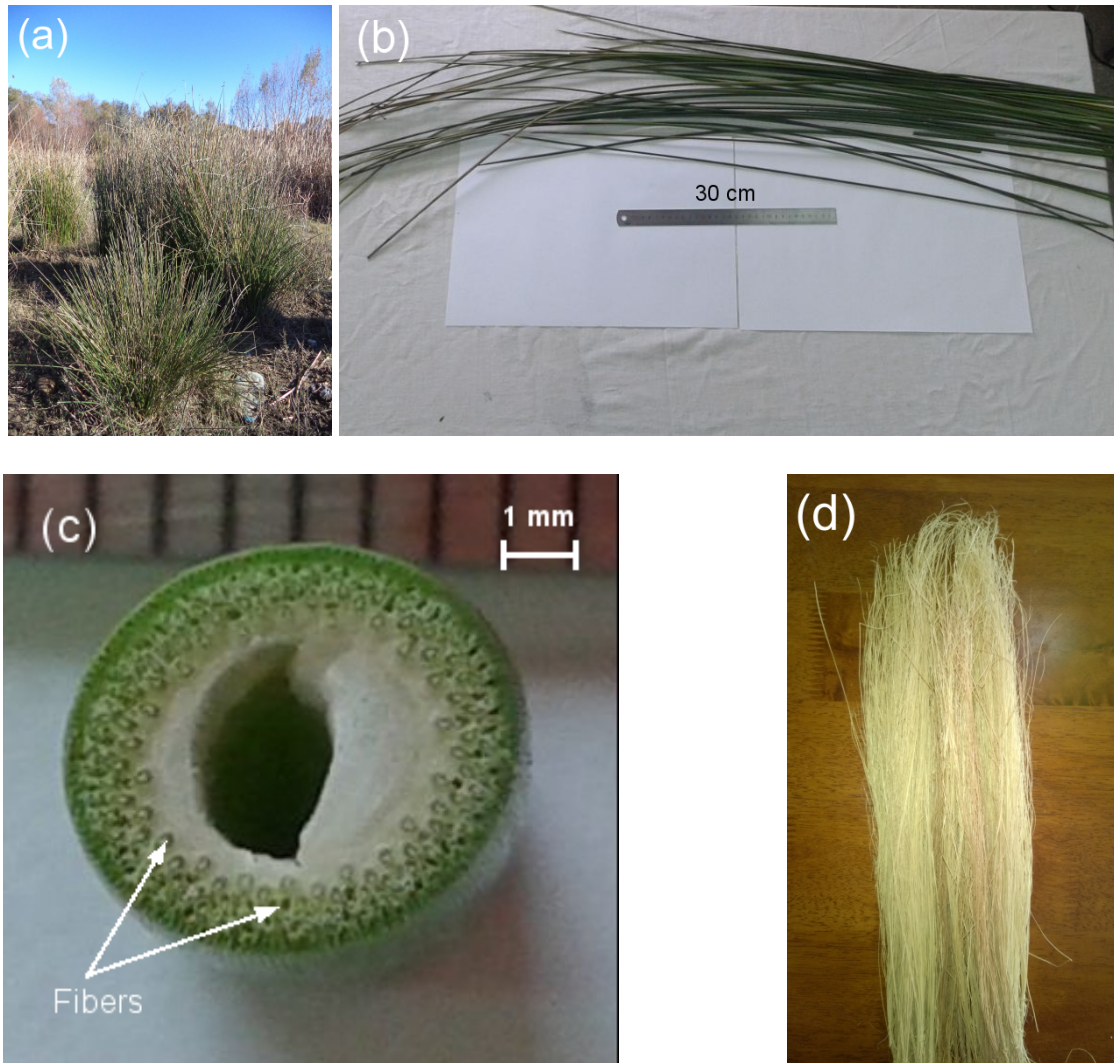


Figure 1. Photographs of (a) JE plant, (b) stem of JE plant, (c) transverse section of the stem (d) fibers extracted from the stem.

Characterization of JE fiber bundles

Morphological characteristics of JE fiber bundles. The microstructure and the morphology of the lignocellulosic fibers were investigated by scanning electron microscopy (SEM) type ESEM Quanta 200 (EIF) equipped with a motorized stage Peltier (-5 °C + 55 °C), an infra-red camera, a micro-injector and a micromanipulator. The accelerating voltage ranged from 0.2 to

30 kV. The image resolution was up to 3584 x 3094 pixels (16 bits). The fiber bundles were coated with a thin layer of gold. The JE fiber diameter was measured using an optical microscope (ZEISS) equipped with a Moticam 2500 digital control camera and driven by Motic Images Plus V2.0 image processing software. The diameter of the fibers may vary along their length, and their effective cross-section was determined using the average diameter value while assuming a cylindrical shape (ignoring the presence of lumen) in order to simplify the analysis. It is a method commonly used in many articles dealing with the investigation of lignocellulosic fibers and their composites (Virk et al., 2009; Li, Pickering & Farrell, 2009; Amroune, Bezazi, Belaadi, Zhu, Scarpa et al., 2015; Bezazi et al., 2014). The average fiber bundle diameter was determined from nine measurements performed at three different locations along the length (mid-section and both ends), i.e. three measurements were performed at each location.

Fourier transform infrared (FTIR) analysis. In order to identify the functional groups of the JE fiber, FTIR measurements were carried out using a Thermo Scientific Nicolet iS10 type device with its own quantitative analysis software. The spectrum was obtained with a scanning speed of 32 acquisitions between 500 and 4000 cm^{-1} with a resolution of 2 cm^{-1} .

Thermal Analysis. Thermogravimetric analysis (TGA) was performed using a Mettler TG 50 module. The weight loss and DTG curves were obtained for a sample at a heating rate of 10 $^{\circ}\text{C}.\text{min}^{-1}$, from room temperature to 950 $^{\circ}\text{C}$ under nitrogen atmosphere (flow rate 100 $\text{mL}.\text{min}^{-1}$).

X-ray diffraction (XRD) analysis. XRD analysis was carried out on a X'Pert Pro SW diffractometer system with $\text{CuK}\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$). The diffracted intensity of the radiation was recorded for 2θ values ranging from 10° to 70° with a pitch of 0.016° , under a voltage of 40 kV and an intensity of 40 mA. The crystallinity index (CrI) of the JE fiber was

calculated according to the empirical method developed by (Segal, Creely, Martin & Conrad, 1959).

$$CrI\% = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (1)$$

where I_{002} is the maximum intensity of the (002) lattice diffraction peak around $2\theta = 22^\circ$ for cellulose I (native cellulose) and I_{am} is the intensity scattered by the amorphous domains of the sample corresponding to the lowest intensity around $2\theta = 18^\circ$.

The crystallite size (Cr_{size}) was calculated by using Scherrer's equation (Saravanakumar et al., 2013):

$$Cr_{size} = \frac{K \lambda}{\beta \cos\theta} \quad (2)$$

Where K is the Scherrer's constant (0.9), λ is wavelength of the radiation, β is the peak's full-width at half-maximum (FWHM) and θ is the diffraction angle.

Fibre density. This measurement was carried out by using a pycnometer for solids with distilled water as a liquid of immersion according to ASTM D 2320 – 98 (2003). The density of JE fiber was calculated using the relation:

$$\rho_{JE} = \frac{(W_3 - W_1)}{[(W_2 - W_1) - (W_4 - W_3)]} \rho_w \quad (3)$$

Where ρ_w is the density of distilled water (0.9970 g.cm^{-3} at 25°C), W_1 is the mass of the empty pycnometer, W_2 is the mass of the pycnometer filled with distilled water at 25°C , W_3 is the mass of the pycnometer filled with chopped fibers and W_4 is the mass of the pycnometer filled with chopped fibers and distilled water at 25°C .

Tensile tests. The tensile tests were carried out on the fiber bundle using universal Zwick/Roell Z005 machine with a 5 kN load cell according to ASTM D3822-07. The ends of the fibers were attached directly to the jaw. The manually operated clamps used during the tests were self-aligning (concentric) using mechanical springs. Static tensile tests were carried

out with a constant speed of $1 \text{ mm}\cdot\text{min}^{-1}$. Due to the variability of the JE fibers, 30 samples subjected to quasi-static tests were loaded up to the break. All tests were performed at an ambient temperature of $23 \text{ }^\circ\text{C}$ and a relative humidity of 49%.

Statistical analysis. Experimental results from tests performed on lignocellulosic fibers are difficult to analyze due to the dispersion of the results which are an inherent characteristic of this type of fibers. This variability can be explained by the distribution of defects in the fiber or on the surface of the fiber (de Andrade Silva, Chawla & de Toledo Filho, 2008) and it is therefore necessary to use statistical approaches in order to evaluate its average mechanical properties. The statistical analysis of the experimental data obtained from the uniaxial tensile tests performed for JE fibers presented in this work was treated using the Minitab 16 software with the two- and three-parameter Weibull model which was used by several authors for different lignocellulosic fibers (Amroune et al., 2015; Belaadi et al., 2014; Fiore et al., 2014) in order to estimate their mechanical properties.

The cumulative distribution function for the three-parameter Weibull distribution is defined by:

$$F(x) = 1 - \text{Exp} \left[- \left(\frac{x-s_0}{s} \right)^m \right] \quad (4)$$

where x , s , s_0 , and m are all positive real, with s_0 being the threshold that represents an average value of the parameter x , $s > 0$ corresponds to the scale parameter (characteristic value), and m is the shape parameter or Weibull modulus. Stress, Young's modulus and elongation at break were determined at a 95% confidence level to obtain significant results. The two-parameter Weibull model is obtained by assuming that the threshold is equal to zero ($s_0 = 0$).

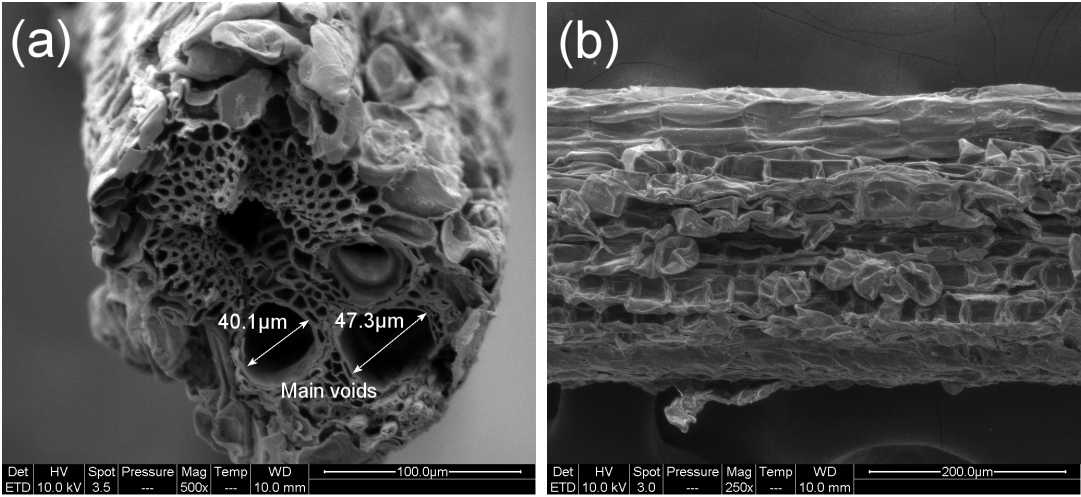
$$F(x) = 1 - \text{Exp} \left[- \left(\frac{x}{s} \right)^m \right] \quad (5)$$

Results and discussion

Figure 2a displays the SEM image of the cross-section of a JE fiber bundle showing its cell structure which is similar to other lignocellulosic fibers such as fiber bundles from date palm

fruit branches (Amroune et al., 2015). It is also found that the section of the fiber consists of porous cells and main voids (or main lumens) which are used for transporting the cellular sap, namely aqueous fluids which circulate through the plant and carry the food and other substances to various tissues (Diaz, de Andrade Silva & Moraes d'Almeida, 2016). The fibrous cells (Fig. 2c) are connected to each other via the lignin-rich medial lamella (Diaz et al., 2016; Beakou, Ntenga, Ateba & Ayina, 2008).

Figure 2b shows the morphology of the overall microstructure of the JE fiber bundle which has rough cavities on its outer surface, with small voids similar to those observed by (Indran et al., 2014). The presence of these cavities can improve the mechanical anchoring (i.e. improve the quality of the fiber/matrix interface) when using the fiber in biocomposite materials. Figure 2d shows the presence of impurities (mainly lignin, but also wax) which is typical for untreated fibers that conditions the mechanical characteristics and the interfacial bond for biocomposites (Fiore et al., 2014).



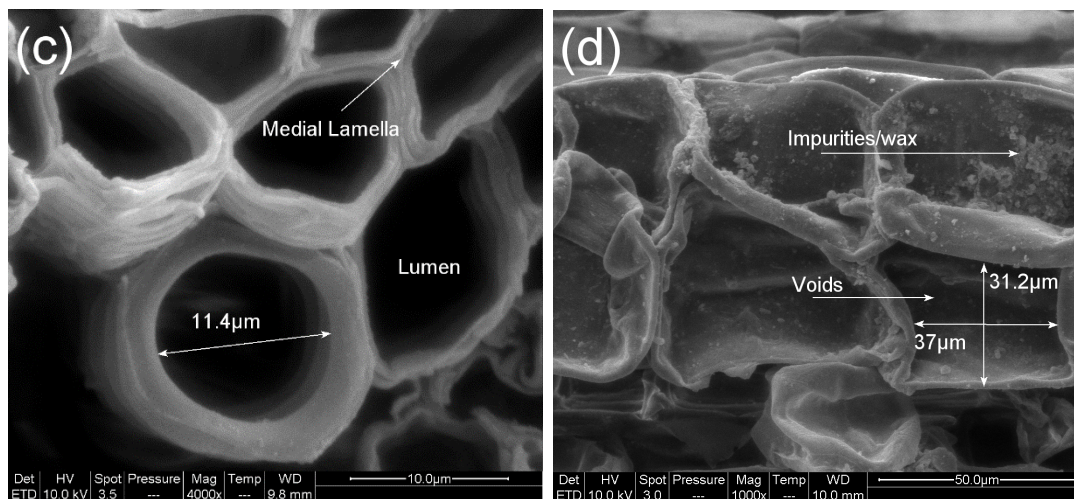


Figure 2. SEM micrographs of (a) the cross-section and (b) longitudinal view of JE fiber. (c) and (d) correspond to a zoom of the cross section and longitudinal view, respectively, of JE fiber.

Figure 3a corresponds to the FTIR spectrum for the JE fibers showing the main IR bands corresponding to the vibrations of the different groups. The bands obtained from this FTIR spectrum are compared with those reported in the literature (Table 1). The peak at 665 cm^{-1} is assigned to out of plane vibrations involving ring structures (Jaouadi, M'sahli & Sakli, 2009), the peak at 894 cm^{-1} can be attributed to the presence of β -glycosidic linkages between the monosaccharides (Reddy, Ashok, Reddy, Feng, Zhang et al., 2014; De Rosa et al., 2010). The peak centered at 1035 cm^{-1} can be associated to the (C-O) stretching modes of hydroxyl and other groups in cellulose (Paiva, Ammar, Campos, Cheikh & Cunha, 2007; Fiore et al., 2014). The absorbance peak centered at 1248 cm^{-1} is due to the (C-O) stretching vibration of the acetyl group in lignin (Rao & Rao, 2007; Liu, Mohanty, Drzal, Askel & Misra, 2004). The absorbance at 1315 cm^{-1} is associated to the C-O groups of the aromatic ring in polysaccharides (Liu, Han, Huang & Zhang, 2009). The peak observed at 1380 cm^{-1} is attributed to the bending vibration of (C-H) (Le Troedec, Sedan, Peyratout, Bonnet, Smith et al., 2008; Bezazi et al., 2014). The peak centered at 1425 cm^{-1} is associated to the CH_2 symmetric bending present in cellulose (Belouadah et al., 2015; Sgriccia, Hawley & Misra,

2008). A small band at 1519 cm^{-1} indicates the presence of (C=C) groups from lignin (Belouadah et al., 2015). The peak at 1621 cm^{-1} may be due the presence of water in the fiber (De Rosa et al., 2010). The double peak observed at 2854 cm^{-1} and 2921 cm^{-1} is attributed to CH stretching vibration from CH and CH_2 in cellulose and hemicellulose (Alvarez & Vásquez, 2006; Paiva et al., 2007). The peak at 3310 cm^{-1} belongs to the hydroxyl group (OH) (Bezazi et al., 2014).

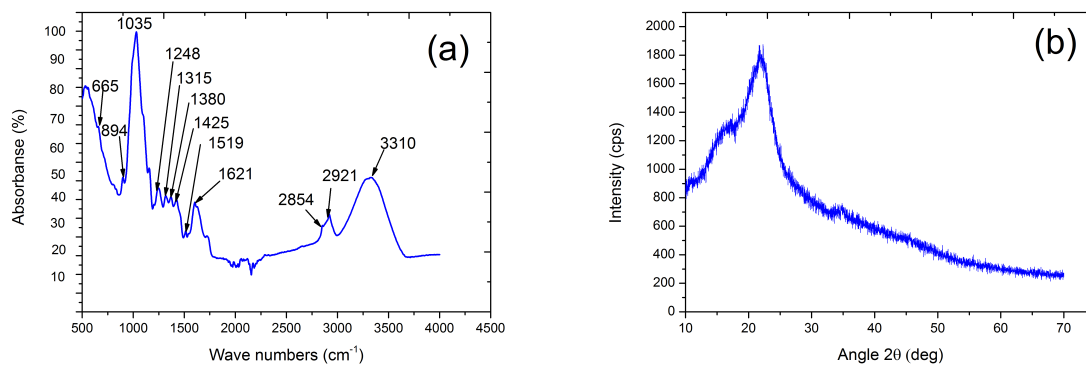


Figure 3. (a) FTIR spectrum and (b) XRD pattern for JE fiber.

Figure 3b shows the X-ray diffraction pattern obtained for JE fiber which is characterized by a strong peak around 22° and two less defined peaks around 15° and 35° in accordance with (Arthanarieswaran, Kumaravel & Saravankumar, 2015; Saravanakumar, Kumaravel, Nagarajan, Sudhakar & Baskaran, 2013), confirming the semi-crystalline nature of the JE fiber bundle. These peaks correspond to native cellulose (cellulose I) and their poor definition results from the contribution of amorphous cellulose and other amorphous components (mainly lignin and hemicellulose) to the diffracted intensity.

The crystallinity index of the JE fiber bundle was calculated using Eq. (1) and a CrI value of 33.4% was obtained. It is higher than the value (19.9%) found for untreated date palm fiber (Abdal-hay, Suardana, Choi & Lim, 2012), or untreated coconut fiber (29.93%) (Carvalho, Mulinari, Voorwald & Cioffi, 2010), but it is lower than that reported for fibers extracted from *Acacia leucophloea* bark (51%) (Arthanarieswaran et al., 2015), *Lyceum spartum L.*

(46.19%) (Belouadah et al., 2015), *Cissus quadrangularis* stem (47.15%) (Indran & Raj, 2015), Napier grass (62.43%) (Kommula, Reddy, Shukla, Marwala, Reddy et al., 2016), *Cissus quadrangularis* root (56.6%) (Indran et al., 2014) and *Prosopis juliflora* bark (46%) (Saravanakumar et al., 2013).

The crystalline size was calculated using the Scherrer's equation (Eq. (2)) and a $C_{r_{size}}$ value of 3.6 nm was obtained for the first crystallographic plane (002). This value is within the range of crystallite size reported for other natural fibers such as 16 nm for ramie fibers, 5.5 nm for cotton fibers, 3.8 nm for cornstalk fibers and 2.8 nm for flax fibers (Indran et al., 2014).

The density of JE fibers (determined by pycnometer method) calculated using Eq. (3) is equal to 1.139 g.cm^{-3} . This value represents one of the lowest values for lignocellulosic fiber (see Table 2). This low density can lead to the reduction of mass for certain specific applications.

Figure 4 displays the TGA curve and its DTG derivative obtained for the JE fiber bundle which allows us to investigate its thermal stability which is an important factor limiting the use of lignocellulosic fibers as a reinforcement in some biocomposites (Fiore et al., 2014).

The first mass loss (5.65%) was noted between 30 and 110 °C, which is attributed by several authors (Belouadah et al., 2015; De Rosa et al., 2010; Fiore et al., 2014) to the vaporization of the water absorbed into the fiber, which confirms its hydrophilic nature. Then, a thermal stability is noted up to about 200 °C where no significant peak is observed in the DTG curve in good agreement with the work of (De Rosa et al., 2010). The second mass loss process which represents the degradation initiation of the JE fiber bundle was observed between 220°C and 360 °C. This can be attributed to the thermal decomposition of hemicelluloses and glycosidic linkages of cellulose (Indran et al., 2014). The peak observed around 300 °C indicates the possible decomposition of cellulose I and α -cellulose (Saravanakumar et al., 2013). Similar peaks have been reported in the literature for bamboo, hemp, jute and kenaf fibers at 321, 308.2, 298.2 and 307.2°C, respectively (Indran et al., 2014). The peak observed

at 455 °C is attributed to the degradation of lignin which can occur when it is heated between 280 and 500 °C (Seki, Sarikanat, Sever & Durmuşkahya, 2013). In addition, a residual mass or char residue of 3.64% was observed. The end products of cellulose degradation contain carbon residues and non-degraded fillers.

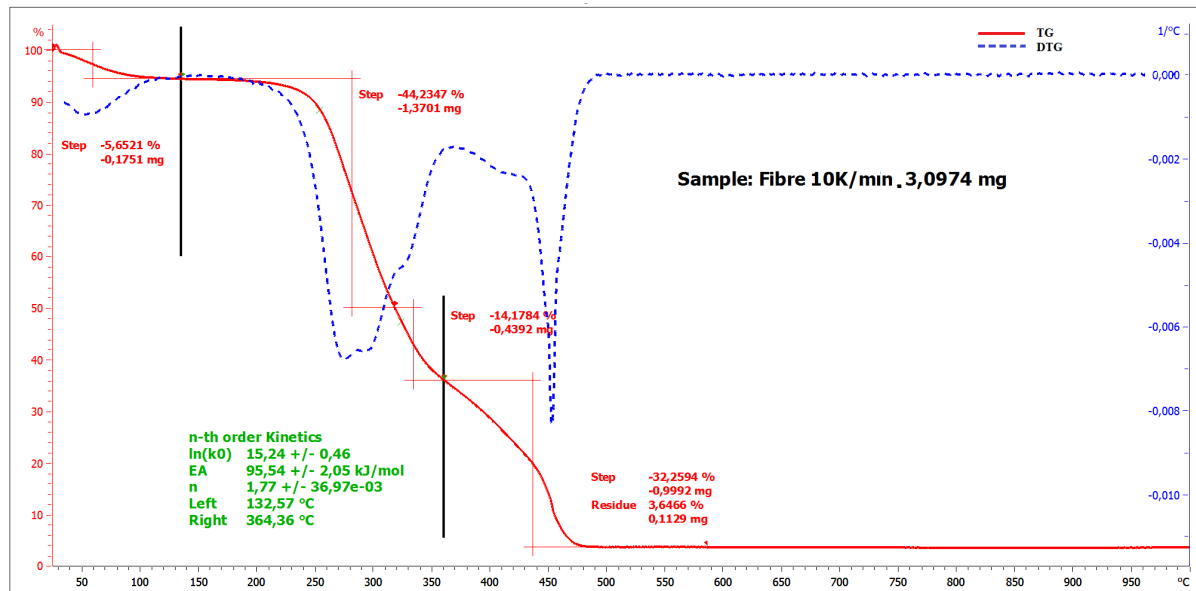


Figure 4. TGA and DTG curves for JE fiber.

A total of thirty fibers was chosen at random in a given batch and mechanically tested under static tensile test at a gage length (GL) equal to 40 mm in order to have a good estimation of the mechanical properties. The Young's modulus was calculated from the elastic part of the stress/strain curves for strain values ranging between 0.35 and 1% (Fig. 5a). The mechanical properties obtained, namely Young's modulus, strength and strain at break were calculated individually for all thirty specimens and average values are summarized in Table 2, and compared with values obtained for other lignocellulosic fibers. Figure 5a illustrates a typical curve of the stress-strain behavior for the JE fiber bundle under monotonic tensile loading. This behavior is quasi-plateau until a strain of 0.2% and then quasi-linear until reaching the ultimate stress (strength) where a brutal failure of the JE fiber occurs which is characterized by a sudden drop in the stress value. This behavior is similar to that found by (Bezazi et al., 2014) and (Belouadah et al., 2015), in the case of sisal and *Lygeum spartum L.* fibers,

respectively. The JE fiber bundles are fragile and small (average diameter 280 μm). Nevertheless, their tensile behavior is difficult to analyze in view of the large dispersions obtained. According to (de Andrade Silva et al., 2008), these dispersions can be mainly related to three factors: parameters and test conditions, plant characteristics and section measurement. Figures 5b and 5c show that the mechanical properties of JE fibers are influenced by their diameter. For the smaller diameters (200 to 250 μm) a larger dispersion in the stress value (Fig. 5b) with a higher average value are observed compared to the larger ones (350 to 400 μm). On the other hand, almost no influence of the fiber bundle diameter is recorded for the deformation (Figure 5C). This behavior is similar to other lignocellulosic fibers such as technical fibers from date palm fruit branches (Amroune et al., 2015), and sisal (Belaadi et al., 2014). The average mechanical properties found for JE fibers are: strength of 113 ± 36 MPa, strain at break of $2.75 \pm 0.68\%$, and Young's modulus of 4.38 ± 1.37 GPa.

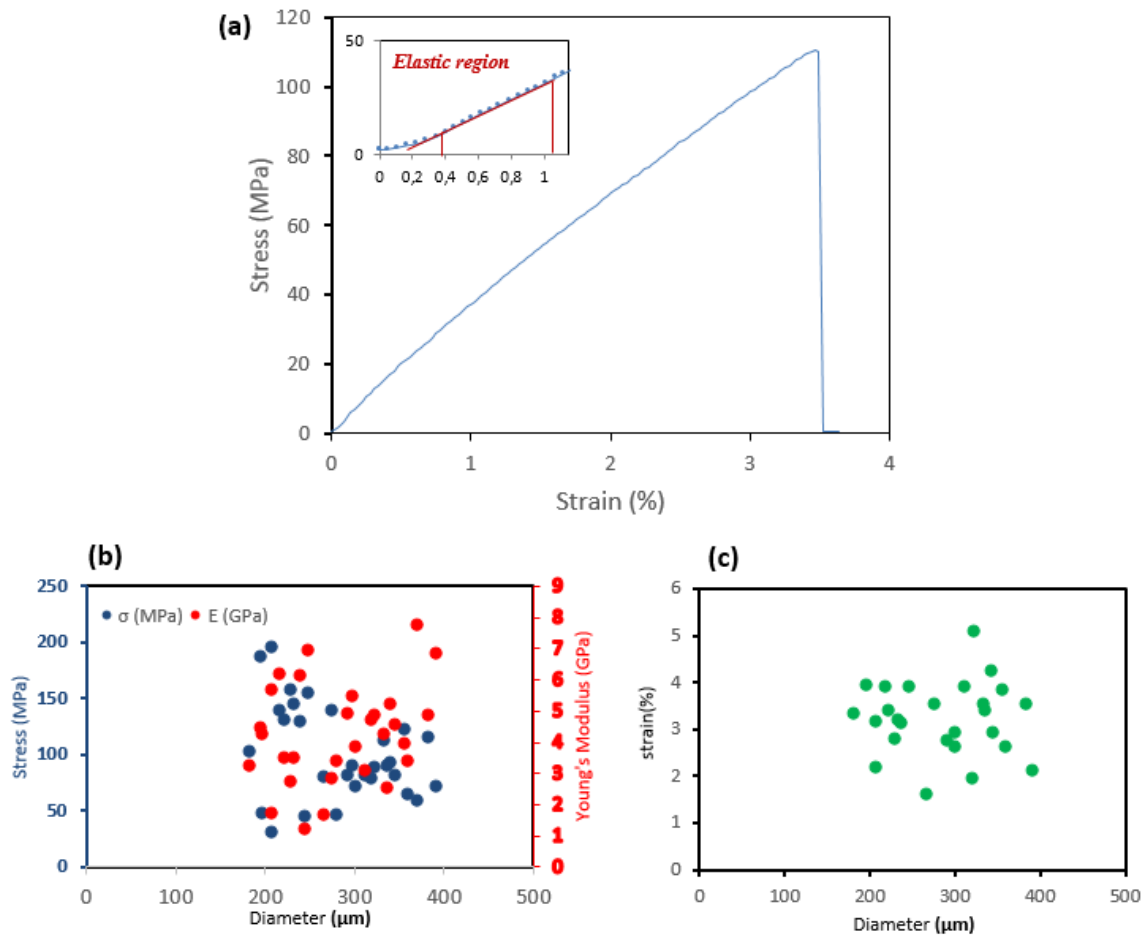
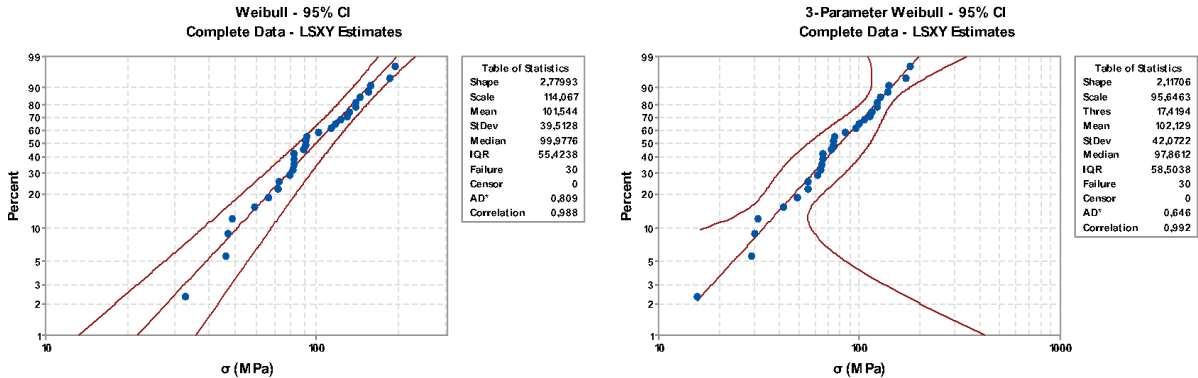


Figure 5. a) Typical stress–strain curve for *Juncus effusus L.* fiber with magnification of the elastic region, b) and c) diameter dependence of the tensile properties of the JE fiber at GL (40mm).

The Young's modulus value found in the present work is substantially equivalent to 4.33 ± 1.4 GPa reported by (Amroune et al., 2015) for technical fibers from date palm fruit branches but remains higher than for other lignocellulosic fibers as reported by (Bezazi et al., 2014) 1.83 ± 0.94 GPa for agave Americana fiber, (Sathishkumar, Navaneethakrishnan, Shankar & Rajasekar, 2013) 3.14 GPa for Spatha fiber, and (Izani, Paridah, Anwar, Nor & H'ng, 2013) 2,763 GPa for oil palm empty fruit bunches fiber. On the other hand, the value of the Young's modulus reported in this work for the JE fiber bundle is considerably lower than that reported by (Belouadah et al., 2015) 13.2 GPa for *Lygeum spartum* fiber and (Fiore et al., 2014) 9.4

GPa for Arundo donax fiber. The strength and strain at break obtained for JE are 113 ± 36 MPa and $2.75 \pm 0.68\%$, respectively, which are very close to that found by (Amroune et al., 2015) for *Phonix dactylifera L.* fibers (117 ± 35 MPa and $3.13 \pm 0.70\%$), and are within the range of values reported by (Belouadah et al., 2014), (Al-Sulaiman, 2002), (Arib, Sapuan, Ahmad, Paridah & Zaman, 2006), and (Bezazi et al., 2014) for *Lygeum spartum L.*, for palm leaves, pineapple leaf and agave Americana fibers, respectively (see Table 2).

The Weibull LS (least squares) statistical distribution curves with two and three parameters of the mechanical properties resulting from the experimental results, namely ultimate stress and strain and the Young's modulus for the JE fibers are presented in Figure 6. It can be noticed that the experimental data are not far from the line and fit correctly the Weibull distribution showing a good agreement with a correlation factor around $R^2 = 0.98$. The modulus m and the characteristic stress σ_0 have a confidence level of 95% for 2 parameters of 2.78 and 114.06 MPa, respectively, and for three parameters the Weibull modulus is 2.11 and $\sigma_0 = 95.64$ MPa. However, the values found for the Young's modulus (E_0) and the strain (ϵ_0) for the two parameters Weibull distribution are equal to 4.80 GPa and 3.43%, respectively, and those for the three parameters Weibull distribution are 5.24 GPa and 2.66%, respectively. It can be clearly seen that the Weibull distribution with two parameters allowed to give values of the mechanical properties close to the average values obtained experimentally. The values of these various parameters are presented in Table 2.



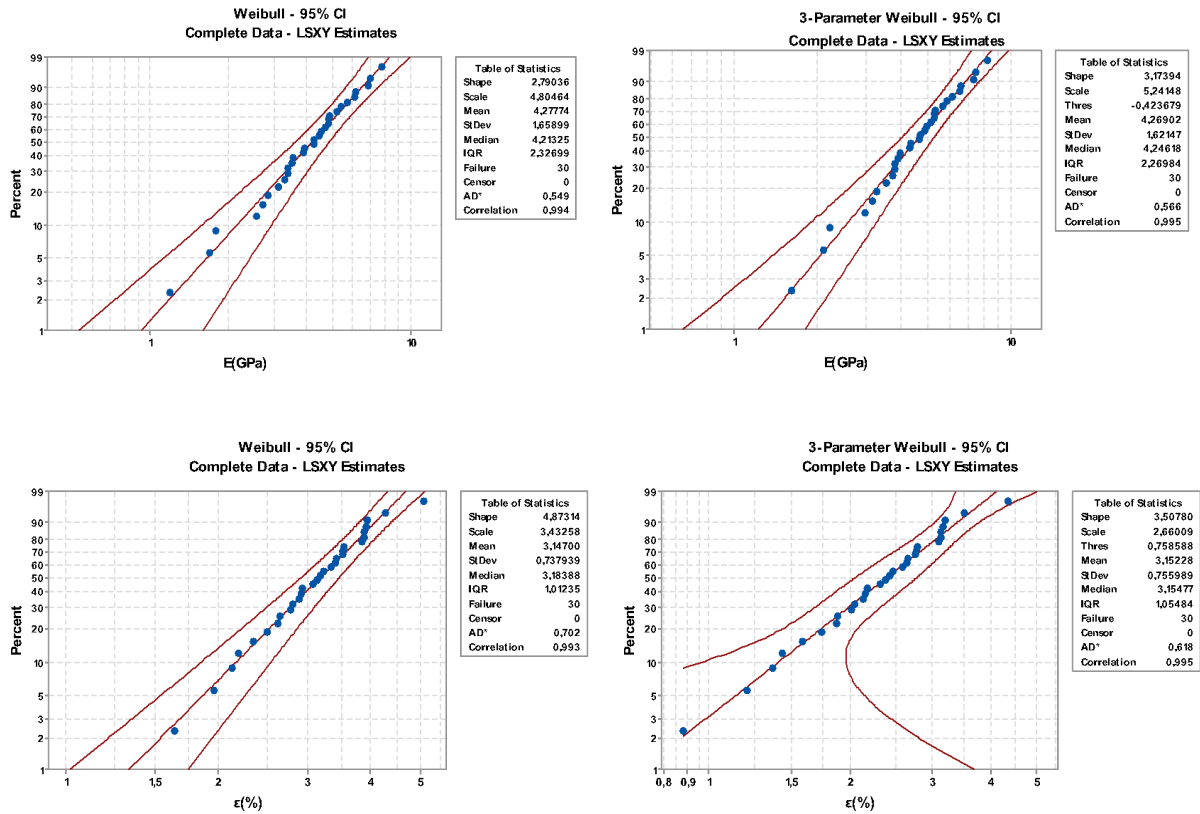


Figure 6. Two and three Weibull distribution for the tensile strength, strain at break, and Young's modulus.

Conclusions

In this article, the properties of a new lignocellulosic fiber extracted from *Juncus effusus L.* (JE) were investigated in order to evaluate the possibility of using it as reinforcement in biocomposite materials. The following conclusions were drawn from the results of the physicochemical, mechanical and thermal characterization of this new natural fiber.

- The study of the surface morphology by SEM revealed that the cross-section of the JE fiber bundle has a cellular shape similar to other fibers, as reported in the literature for sisal or branch fruit of date palm. On the other hand, the longitudinal section of the JE fiber was characterized by the presence of rough surfaces (presence of small voids) which should greatly improve the mechanical anchoring in the manufacture of biocomposites.

- The bands from the FTIR spectrum obtained for JE fiber were analyzed and compared with those reported in the literature for other lignocellulosic fibers.
- The XRD analysis showed the semicrystalline nature of JE fiber. The JE fiber bundle had a crystallinity index of 33.4%.
- Thermogravimetric analysis of JE fiber evidenced a thermal stability up to 220 °C which confirms the possibility of its use as reinforcement for polymer (bio or not bio) matrix composite materials.
- The mechanical properties resulting from the monotonic tensile tests performed on the JE fiber bundle, i.e. strength of 113 ± 36 MPa, strain at break of $2.75 \pm 0.68\%$, and Young's modulus of 4.38 ± 1.37 GPa, showed that these values are globally similar to other plant fibers, making it an alternative for conventional synthetic fibers such as glass fibers used in composite structures.
- The analysis of the tensile tests results showed that the mechanical characteristics estimated by LS-Weibull with 2 parameters are very close to those obtained experimentally compared to LS-Weibull with 3 parameters.

Acknowledgements

The authors would like to thank Ms. Bertine Khelifi from Pagora LGP2 (Grenoble, France) for her help on acquiring the SEM images. The authors are also gratefully to Mr. Haouanoh Djedjiga (Materials and Environment Research Unit of the University of Boumerdes Algeria) for his help in acquiring the XRD data. LGP2 is part of the LabEx Tec 21 (Investissements d'Avenir - grant agreement no. ANR-11-LABX-0030) and of the PolyNat Carnot Institut (Investissements d'Avenir - grant agreement no. ANR-11- CARN-030-01).

References

Abdal-hay, A., Suardana, N.P.G., Choi, K.S., Lim, J.K. (2012). Effect of diameters and alkali treatment on the tensile properties of date palm fiber reinforced epoxy composites. *International Journal of Precision Engineering and Manufacturing*, 13, 1199-1206.

Al-Sulaiman, F.A. (2002). Mechanical properties of date palm fiber reinforced composites. *Applied Composite Materials*, 9, 369-377.

Alvarez, V.A., Vázquez, A. (2006). Influence of fiber chemical modification procedure on the mechanical properties and water absorption of MaterBi-Y/sisal fiber composites. *Composites Part A: Applied Science and Manufacturing*, 37, 1672-1680.

Amroune, S., Bezazi, A., Belaadi, A., Zhu, C., Scarpa, F., Rahatekar, S., Imad, A. (2015). Tensile mechanical properties and surface chemical sensitivity of technical fibres from date palm fruit branches (*Phoenix dactylifera L.*). *Composites Part A: Applied Science and Manufacturing*, 71, 95-106.

Annual Climatological Report, Guelma (2014). <http://www.infoclimat.fr/climatologie/annee/2014/guelma/valeurs/60403.html>.

Arib, R.M.N., Sapuan, S.M., Ahmad, M.M.H.M., Paridah, M.T., Zaman, H.K. (2006). Mechanical properties of pineapple leaf fibre reinforced polypropylene composites. *Materials & Design*, 27, 391-396.

Arthanarieswaran, V.P., Kumaravel, A., Saravanakumar, S.S. (2015). Characterization of new natural cellulosic fiber from *Acacia leucophloea* bark. *International Journal of Polymer Analysis and Characterization*, 20, 367-376.

Azwa, Z.N., Yousif, B.F., Manalo, A.C., Karunasena, W. (2013). A review on the degradability of polymeric composites based on natural fibres. *Materials & Design*, 47, 424-442.

Baley, C., Bourmaud, A. (2014). Average tensile properties of French elementary flax fibers. *Materials Letters*, 122, 159-161.

Beakou, A., Ntenga, R., Lepetit, J., Ateba, J.A., Ayina, L. O. (2008). Physico-chemical and microstructural characterization of “*Rhectophyllum camerunense*” plant fiber. *Composites Part A: Applied Science and Manufacturing*, 39, 67-74.

Beckermann, G.W., Pickering, K.L. (2009). Engineering and evaluation of hemp fibre reinforced polypropylene composites: Micro-mechanics and strength prediction modelling. *Composites Part A: Applied Science and Manufacturing*, 40, 210-217.

Belaadi, A., Bezazi, A., Bouchak, M., Scarpa, F., Zhu, C. (2014). Thermochemical and statistical mechanical properties of natural sisal fibres. *Composites Part B: Engineering*, 67, 481-489.

Belouadah, Z., Ati, A., Rokbi, M. (2015). Characterization of new natural cellulosic fiber from *Lygeum spartum L.* *Carbohydrate polymers*, 134, 429-437.

Bezazi, A., Belaadi, A., Bouchak, M., Scarpa, F., Boba, K. (2014). Novel extraction techniques, chemical and mechanical characterisation of Agave Americana L. Natural fibres. *Composites Part B: Engineering*, 66, 194-203.

Bledzki, A.K., Reihmane, S., Gassan, J. (1996). Properties and modification methods for vegetable fibers for natural fiber composites. *Journal of Applied Polymer Science*, 59, 1329-1336.

Bledzki, A.K., Franciszczak, P., Osman, Z., Elbadawi, M. (2015). Polypropylene biocomposites reinforced with softwood, abaca, jute, and kenaf fibers. *Industrial Crops and Products*, 70, 91-99.

Carvalho, K.C.C., Mulinari, D.R., Voorwald, H.J.C., Cioffi, M.O.H. (2010). Chemical modification effect on the mechanical properties of hips/coconut fiber composites. *BioResources*, 5, 1143-1155.

Cheung, H.Y., Ho, M.P., Lau, K.T., Cardona, F., Hui, D. (2009). Natural fibre-reinforced composites for Bioengineering and environmental engineering applications. *Composites Part B: Engineering*, 40, 655-663.

De Andrade Silva, F., Chawla, N., de Toledo Filho, R.D. (2008). Tensile behavior of high performance natural (sisal) fibers. *Composites Science and Technology*, 68, 3438-3443.

De Rosa, I.M., Kenny, J.M., Puglia, D., Santulli, C., Sarasini, F. (2010). Morphological, thermal and mechanical characterization of okra (*Abelmoschus esculentus*) fibres as potential reinforcement in polymer composites. *Composites Science and Technology*, 70, 116-122.

Di Bella, G., Fiore, V., Galtieri, G., Borsellino, C., Valenza, A. (2014). Effects of natural fibres reinforcement in lime plasters (kenaf and sisal vs. Polypropylene). *Construction and Building Materials*, 58, 159-165.

Diaz, J.P.V., De Andrade Silva, F., Moraes d'Almeida, J.R. (2016). Effect of peach palm fiber microstructure on its tensile behavior. *BioResources*, 11, 10140-10157.

Dittenber, D.B., GangaRao, H.V.S. (2012). Critical review of recent publications on use of natural composites in infrastructure. *Composites Part A: Applied Science and Manufacturing*, 43, 1419-1429.

Dobah, Y., Bouchak, M., Bezazi, A., Belaadi, A., Scarpa, F. (2016). Multi-axial mechanical characterization of jute fiber/polyester composite materials. *Composites Part B: Engineering*, 90, 450-456.

Fiore, V., Valenza, A., Di Bella, G. (2011). Artichoke (*Cynara cardunculus* L.) fibres as potential reinforcement of composite structures. *Composites Science and Technology*, 71, 1138-1144.

Fiore, V., Scalici, T., Valenza, A. (2014). Characterization of a new natural fiber from *Arundo donax* L. as potential reinforcement of polymer composites. *Carbohydrate polymers*, 106, 77-83.

Ganan, P., Mondragon, I. (2004). Figue fiber-reinforced polyester composites: effects of fiber surface treatments on mechanical behavior. *Journal of materials science*, 39, 3121-3128.

Hornsby, P.R., Hinrichsen, E., Tarverdi, K. (1997). Preparation and properties of polypropylene composites reinforced with wheat and flax straw fibres: Part II Analysis of composite microstructure and mechanical properties. *Journal of Materials Science*, 32, 1009-1015.

Indran, S., Raj, R.E., Sreenivasan, V.S. (2014). Characterization of new natural cellulosic fiber from *Cissus quadrangularis* root. *Carbohydrate polymers*, 110, 423-429.

Indran, S., Raj, R.E. (2015). Characterization of new natural cellulosic fiber from *Cissus quadrangularis* stem. *Carbohydrate polymers*, 117, 392-399.

Izani, M.N., Paridah, M.T., Anwar, U.M.K., Nor, M.M., H'ng, P.S. (2013). Effects of fiber treatment on morphology, tensile and thermogravimetric analysis of oil palm empty fruit bunches fibers. *Composites Part B: Engineering*, 45, 1251-1257.

Jaouadi, M., M'sahli, S., Sakli, F. (2009). Optimization and characterization of pulp extracted from the Agave americana L. fibers. *Textile Research Journal*, 79, 110-120.

Kommula, V.P., Reddy, K.O., Shukla, M., Marwala, T., Reddy, E.S., Rajulu, A.V. (2016). Extraction, modification, and characterization of natural ligno-cellulosic fiber strands from napier grass. *International Journal of Polymer Analysis and Characterization*, 21, 18-28.

Le Troedec, M., Sedan, D., Peyratout, C., Bonnet, J.P., Smith, A., Guinebretiere, R., Gloaguen, V., Krausz, P. (2008). Influence of various chemical treatments on the composition and structure of hemp fibres. *Composites Part A: Applied Science and Manufacturing*, 39, 514-522.

Li, Y., Pickering, K.L., Farrell, R.L. (2009). Determination of interfacial shear strength of white rot fungi treated hemp fibre reinforced polypropylene. *Composites Science and Technology*, 69, 1165-1171.

Liu, D., Han, G., Huang, J., Zhang, Y. (2009). Composition and structure study of natural Nelumbo nucifera fiber. *Carbohydrate polymers*, 75, 39-43.

Liu, W., Mohanty, A.K., Drzal, L.T., Askel, P., Misra, M. (2004). Effects of alkali treatment on the structure, morphology and thermal properties of native grass fibers as reinforcements for polymer matrix composites. *Journal of Materials Science*, 39, 1051-1054.

Majeed, K., Jawaid, M., Hassan, A., Bakar, A.A., Khalil, H.A., Salema, A.A., & Inuwa, I. (2013). Potential materials for food packaging from nanoclay/natural fibres filled hybrid composites. *Materials & Design*, 46, 391-410.

Malainine, M.E., Dufresne, A., Dupeyre, D., Mahrouz, M., Vuong, R., Vignon, M.R. (2003). Structure and morphology of cladodes and spines of *Opuntia ficus-indica*. Cellulose extraction and characterisation. *Carbohydrate Polymers*, 51, 77-83.

Mohanty, A.K., Wibowo, A., Misra, M., Drzal, L.T. (2004). Effect of process engineering on the performance of natural fiber reinforced cellulose acetate biocomposites. *Composites Part A: Applied Science and Manufacturing*, 35, 363-370.

Paiva, M.C., Ammar, I., Campos, A. R., Cheikh, R.B., Cunha, A.M. (2007). Alfa fibres: Mechanical, morphological and interfacial characterization. *Composites Science and Technology*, 67, 1132-1138.

Park, S.N., Won, D.H., Hwang, J.P., Han, S.B. (2014). Cellular protective effects of dehydroeffusol isolated from *Juncus effusus* L. and the mechanisms underlying these effects. *Journal of Industrial and Engineering Chemistry*, 20, 3046-3052.

Placet, V. (2009). Characterization of the thermo-mechanical behaviour of Hemp fibres intended for the manufacturing of high performance composites. *Composites Part A: Applied Science and Manufacturing*, 40, 1111-1118.

Rao, K.M.M., Rao, K.M. (2007). Extraction and tensile properties of natural fibers: Vakka, date and bamboo. *Composite structures*, 77, 288-295.

Reddy, K.O., Ashok, B., Reddy, K.R.N., Feng, Y.E., Zhang, J., Rajulu, A.V. (2014). Extraction and characterization of novel lignocellulosic fibers from *Thespesia lampas* plant. *International Journal of Polymer Analysis and Characterization*, 19, 48-61.

Saravanakumar, S.S., Kumaravel, A., Nagarajan, T., Sudhakar, P., Baskaran, R. (2013). Characterization of a novel natural cellulosic fiber from *Prosopis juliflora* bark. *Carbohydrate Polymers*, 92, 1928-1933.

Sathishkumar, T.P., Navaneethakrishnan, P., Shankar, S., Rajasekar, R. (2013). Characterization of new cellulose *Sansevieria ehrenbergii* fibers for polymer composites. *Composite Interfaces*, 20, 575-593.

Scalici, T., Fiore, V., Valenza, A. (2016) Effect of plasma treatment on the properties of *Arundo donax L.* leaf fibres and its bio-based epoxy composites: a preliminary study. *Composites Part B: Engineering*, 94, 167-175.

Segal, L., Creely, J.J., Martin, A.E., Conrad, C.M. (1959). An empirical method for estimating the degree of crystallinity of native cellulose using the X-ray diffractometer. *Textile Research Journal*, 29, 786-794.

Seki, Y., Sarikanat, M., Sever, K., Durmuşkahya, C. (2013). Extraction and properties of *Ferula communis* (chakshir) fibers as novel reinforcement for composites materials. *Composites Part B: Engineering*, 44, 517-523.

Sgriccia, N., Hawley, M.C., Misra, M. (2008). Characterization of natural fiber surfaces and natural fiber composites. *Composites Part A: Applied Science and Manufacturing*, 39, 1632-1637.

Sreekala, M.S., Kumaran, M.G., Joseph, S., Jacob, M., Thomas, S. (2000). Oil palm fibre reinforced phenol formaldehyde composites: influence of fibre surface modifications on the mechanical performance. *Applied Composite Materials*, 7, 295-329.

Virk, A.S., Hall, W., Summerscales, J. (2009). Multiple Data Set (MDS) weak-link scaling analysis of jute fibres. *Composites Part A: Applied Science and Manufacturing*, 40, 1764-1771.

Table 1: FTIR bands observed for JE fiber.

Band position in this work (cm⁻¹)	Wave number range (cm⁻¹)	Origin	Reference
3310	3600–3100	Hydrogen bonded O-H stretching	(Amroune et al., 2015; Bezazi et al., 2014)
2921-2854	2950 and 2854	C-H stretching vibration from CH and CH ₂ in cellulose and hemicellulose	(Alvarez et al., 2006; Paiva et al., 2007)
1621	1740–1600	Presence of water in the fiber	(De Rosa et al., 2010)
1519		(C=C) groups of lignin	(Belouadah et al., 2015)
1425	1430	CH ₂ symmetric bending from cellulose	(Belouadah et al., 2015; Sgriccia, et al., 2008)
1380	1377	Bending vibration of (C-H)	(Le Troedec et al., 2008; Bezazi et al., 2014)
1315	1320	C-O groups of the aromatic ring in polysaccharides	(Liu et al., 2009)
1248	1243	C-O stretching vibration of the acetyl group in lignin	(Rao & Rao, 2007; Liu et al., 2004)
1035	1055	C-O stretching modes of hydroxyl and ether groups in cellulose	(Amroune et al., 2015; Paiva et al., 2007; Fiore et al., 2011)
894	894	β-glycosidic linkages between the monosaccharides	(Reddy et al., 2014; De Rosa et al., 2010)
665	670–620	Out of plane vibrations involving ring structure	(Jaouadi et al., 2009)

Table 2: Mechanical properties and statistical Weibull parameters for JE fiber and other natural fibers: gauge length (GL), diameter (D), density, strength (σ), Young's modulus (E), and strain at break (ϵ), characteristic strength (σ_0), characteristic strain (ϵ_0), characteristic Young's modulus (E_0), and Weibull modulus (m_σ , m_ϵ , m_E).

Fiber	Experimental results						Weibull 2P Stress		Weibull 3P Stress			Weibull 2P Young Modulus		Weibull 3P Young Modulus			Ref.
	GL (mm)	D (μm)	Density (g.cm^{-3})	σ (MPa)	E (GPa)	ϵ (%)	m_σ	σ_0	m	σ_0	σ_u	m_E	E_0	m_E	E_0	E_u	
flax	-	16.8 \pm 2.7	1.38	945 \pm 200	52.5 \pm 8.6	2.07 \pm 0.45	-	-	-	-	-	-	-	-	-	-	(Baley et al 201
hemp		42	1.07	285	14.4	2.2	-	-	-	-	-	-	-	-	-	-	(Placet 2009)
jute	-	-	1.3–1.45	340–470	1.3–42.2	1.15–1.5	-	-	-	-	-	-	-	-	-	-	(Bledzki et al 20
kenaf	-	-	1.19–1.2	470–785	25.1	1.75–1.9	-	-	-	-	-	-	-	-	-	-	(Bledzki et al 20
<i>CQ stem</i>	10-50	770-870	1.22	2300-5479	56-234	3.75-11.14	-	-	-	-	-	-	-	-	-	-	(Indran et al 201
<i>CQ root</i>	10-50	610-725	1.51	1857-5330	68-203	3.57-8.37	-	-	-	-	-	-	-	-	-	-	(indran et al 201
<i>Lygeum spartum L.</i>	40	180-433	1.4997	64.63-280	4.47-13.27	1.49-3.74	-	-	-	-	-	-	-	-	-	-	(Belouadah et al., 2
<i>Arundo donax</i>	30	-	1.168	248	9.4	3.24	3.12	248.44	-	-	-	4.29	9.38	-	-	-	(Fiore et al., 201
Fique		50-200	0.870	200	8-12	4-6	-	-	-	-	-	-	-	-	-	-	(Ganan & Mondra 2004)
Artichoke	10	300	1.579	182	10.7	2.8	3.71	201	-	-	-	4.47	11.62				(Fiore et al, 201
Pineapple leaf			1.07	126.6	4.405	2.2	-	-	-	-	-	-	-	-	-	-	(Arib et al., 200
<i>Phoenix dactylifera L.</i>	50	577 \pm 83	-	117 \pm 35	4.3 \pm 1.4	3.13 \pm 0.7	4.45	128.23	2.42	80.73	45.68	3.73	4.78	1.76	2.85	1.82	(Amroune et al., 2
Agave	40	239 \pm 68	-	132 \pm 66	1.83 \pm 0.94	33.29 \pm 13.5 5	1.93	128.37	-	-	-	2.02	2.47	-	-	-	(Bezazi et al. 20
Palm leaf			-	97-196	2.5-4.7	2-4.5			-	-	-			-	-	-	(Al-Sulaiman, 20
Oil palm empty fruit bunch	25	330-340	-	49	2.76	7	-	-	-	-	-	-	-	-	-	-	(Izani et al., 201
<i>Juncus effusus L.</i>	40	280 \pm 56	1.139	113 \pm 36	4.38 \pm 1.37	2.75 \pm 0.6	2.78	114.06	2.11	95.64	17.41	2.79	4.80	3.17	5.24	0.42	Present work

