Characterizing *Teline monspessulana* as a Green Sustainable Source of Biofibers

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Invasive *Teline monspessulana* can be an important source of biomass to supply fibers for the rising demand of cellulose bioproducts, especially for the development of advanced materials. Its fibers can be extracted *via* a thermo-alkaline process at 170 °C with 40 g/L of sodium hydroxide (NaOH) and characterized by crystallographic, thermo-analytical, and mechanical techniques. The cellulose proportion in the wood of this species is approximately 47.6 wt.% ± 1.05 wt.%. However, its fibers are relatively small, and they have a wide range of aspect ratios from 25 to 287, with an average diameter of 9.3 µm ± 2.5 µm. These characteristics and mechanical properties make the fibers unattractive for the textile and paper industries. Meanwhile, crystalline cellulose was prevalent in the monoclinic phase, with a crystalline index and crystalline portion of 78 and 41%, respectively, observing crystal domains of *c.a.* 3.2 nm. Nanoindentation tests revealed favorable values of elastic modulus and hardness of *c.a.* 16 GPa and 0.28 GPa, respectively. Thus, this bioresource is expected to see promising applications in materials engineering, such as reinforcement in material composites, in drug delivery carrier, and electronic devices, among other biomultifunctional components.

Keywords: Teline monspessulana; Non-traditional biofiber; Mechanical properties; Crystallinity

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INTRODUCTION

Biofibers can be obtained from non-traditional lignocellulosic resources that have evolved rapid and intelligent functional systems to adapt to a wide range of climatic and edaphic conditions (Tashiro and Kobayashi 1991; Carroll and Somerville 2009; Celis *et al.* 2014). These resources are an attractive challenge for the development of sustainable, future technologies. Currently, these species are generating substantial attention due to their bioorganic components for phytotechnology applications.

Teline monspessulana (Cape broom) a Papilionaceae (Leguminosae shrub) is biogeographically native to the Mediterranean regions of Europe and western North Africa. The species is highly invasive in temperate macro-zones (Pauchard *et al.* 2008, Zuloaga *et al.* 2008), in areas frequently affected by fires, and in regions with a temperate continental climate (Canada, New Zealand, and USA) (Alexander 1997; García *et al.* 2010; Geerts *et al.* 2013; CABI 2016). This Fabaceae is an interesting bioresource, along

with other similar plant species of the genera *Chusquea*, *Ulex*, *Cytisus*, *Calycotome*, and *Spartium*, among others. They all have a very efficient functional system that develops rapidly, and is characterized by a high speed of maturation and aggressiveness to establish themselves within the environment. This generates a highly optimized macro-, meso-, micro-, and ultrastructure system that is advantageous under highly unfavorable edapho-climatic conditions (CABI 2016). However, apart from the interesting properties reported from a non-traditional perspective, each of the species possesses biofibers of unique characteristics and properties due to their genesis, complex structure, chemical composition, dimensions, defects, maturity, and effects of extractive processes (Bledzki and Gassan 1999; Dufresne 2008; Eichhorn *et al.* 2010). These characteristic features determine their potential technological applications. Currently, research is being conducted on *Teline monspessulana* due to the presence of alkaloid contained in its leaves called quinolizidine. This alkaloid has neuroprotective effects related to the modulation of acetyl and/or butyrylcholinesterase (AChE/BuChE), namely the $\alpha 7$ subtype. This subtype is particularly interesting due to its association with the activation of anti-apoptosis PKT (Protein kinase B or Akt) in the control of diseases such as Alzheimer's (Araya *et al.* 2014; Fuentealba and Saez-Orellana 2014). The nutritional properties of this species as cattle feed have also been studied (Berhe and Tothill 1997). Further attempts have been made on *Teline* fibers in polymer matrices of low-density polyethylene (LDPE) and high impact polystyrene (HIPS), using different concentrations of the crosslinking agent dicumyl peroxide. The composite presses showed interesting improvements in their mechanical properties, as expected in short fibers, but questions arose regarding the tension of the distribution of the fibers within the polymer matrix (Tabón *et al.* 2014). In view of the fact that *Teline* is a large available biomass mainly useful for the development of cellulose-based materials, it could be a great source of advanced materials for industrial applications in nanotechnology (Varshney and Naithani 2011), agriculture, nanosheet, drug delivery, *etc.* In this preliminary study, we investigate the structural, microstructural, physic-mechanical, thermal, and nanomechanical characteristics of the highly resilient and invasive *Teline* species fibers for potential sustainable green applications.

EXPERIMENTAL

Materials

Green source characterization

    Teline monspessulana shrubs of approximately 3 to 4 meters high were collected in autumn 2016 from pastures and underbrush in the area of Morrompulli, approximately 24 km south of Valdivia, Chile (39°58'47"S 73°08'25"W). The collected biomass was reduced to approximately 20 mm to 30 mm using a model 150XP HD Bandit chipper (Bandit Industries, Inc., Remus, MI, USA) without discriminating the size and anatomy of the bioresources.

    Quantitative determination of holocellulose, cellulose, and lignin was performed on the *T. monspessulana* stems according to Poljak's method (Poljak 1948), Kurschner-Hoffer cellulose method (Kurschner and Hoffer 1931), and the standard method of the Technical Association of the Pulp and Paper Industry (TAPPI) T222 om-88 (1988), respectively. Extraction in cold and boiling water with 1 wt.% sodium hydroxide (NaOH), ethanol, and toluene was also conducted according to the method established in

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the TAPPI T204 om-88 (1988) standard. Furthermore, quantification of the bioorganic components in fibers of *Teline* was performed according to the following the American Society for Testing and Materials (ASTM) standards: cellulose (ASTM D1103-60 (1977)), lignin (ASTM D1106-96 (2013)), ash (ASTM D1102-84 (2013)), extractives (ASTM D1105-96 (2013)), and moisture (ASTM D4442-07 (2007)), respectively.

Extraction of fibers

        T. monspessulana fibers were obtained from the reduced biomass following a laboratory based alkaline pulping process protocol. Thermochemical treatment consisted of mixing the fibrous material in an M/K 610 Mini-Mill digester (MK System Inc., Peabody, MA, USA) with an aqueous solution (60 g/L) of NaOH, taking into account a fiber release rate of at least 10%, *i.e.* a Kappa number lower than 67. Temperature was applied (Table 1) to remove the lignin bound to the fibers so that the *Teline* chips could be easily defibered (Casey 1990). The process was conducted under constant conditions of NaOH concentration, pressure, temperature, and time according to the reports of Torres and Rodriguez (1991) on *Eucalyptus globulus* (Table 1). The purpose of this process was to form a minimum of fiber bundles (bonded fiber packages). Finally, the process attained a yield of approximately 75% NaOH base, with an obtained Kappa number of 22.

Table 1. Parameters for Obtaining Fibers by Chemical Pulping Process

Properties	Values
Dry wood (kg)	1
Maximum temperature (°C)	170
Time until maximum temperature (min)	65
Time at maximum temperature (min)	120
Concentration of NaOH (g/L)	60
Hydromodule (w/v)	4/1
Pressure (mPa)	0.827

Methods

X-ray diffraction method

        X-ray diffraction (XRD) patterns were collected on a Bruker D2 Phaser Advance Bragg-Brentano diffractometer operating a copper tube (Cu K_α λ = 1.5418 Å) at 30 kV and 10 mA and equipped with a nickel filter and LYNXEYE™ detector (Bruker, Karlsruhe, Germany). The goniometer system comprises a high-resolution setup (0.3° divergence slit, 2.5° incident and diffracted beam Soller slits, 6 mm receiving slit) and a curved-crystal graphite monochromator, providing a narrow and symmetrical instrumental profile over the investigated angular range. The instrumental resolution function was characterized with the National Institute of Standard's Standard Reference Material (NIST SRM 1976b) (2015) (corundum) standard: all peak profiles were simultaneously fitted with symmetrical pseudo-Voigt functions whose width and shape were constrained according to the Caglioti *et al.* (1958) formulae. Prior to the measurements, 1 g of the *T. monspessulana* fibers was dried at 60 °C for 24 h in a Binder ED53 (BINDER GmbH, Tuttlingen, Germany) chamber oven. Subsequently, the sample was pressed in a 25-mm diameter die at 4 kPa for 5 min. The disks obtained were mounted on a rotary sample holder, and rotated at a speed of 15 rpm in the diffractometer

during data acquisition. The XRD patterns of the specimen was recorded in the 5° to 45° 2θ range with a step size of 0.01° and a counting time of 5 s per step.

Due to the prevalence of monoclinic or triclinic phases in the cellulose of biofibers, the Z-discriminant function developed by Wada and Okano (2001) was applied. The crystallinity index (CI) was calculated according to the method of Segal *et al.* (1959) using the statistical software OriginLab Pro® V 8.0 (OriginLab Corporation, Northampton, MA, USA), while crystalline portion (Cx) was calculated from the integrated intensity of the amorphous and crystalline phases contribution to the total diffraction pattern, suggesting that the effect of the absorption is not influenced because the different phases of the samples are same chemically (Klug and Alexander 1974; Zevin and Kimmel 1995).

In this study, the Scherrer formula (1918) was considered valid only for comparison purposes, but not for a true quantitative assessment of the domain size of cellulose.

Differential calorimetry analysis

Differential scanning calorimetry (DSC) was performed on a TA Instruments DSC-Q20 thermal analyser (TA Instruments, New Castle, DE, USA) under a controlled, mixed atmosphere of 50 mL N_2/min and 50 mL air/min. Approximately 7 mg of *Teline* fibers were placed in an Al_2O_3 crucible, subjected to a linear heating ramp of 25 ℃ to 450 ℃ at a rate of 10 ℃/min. The characteristic calorimetric parameters were processed using Platinum™ Software (TA instruments, New Castle, DE, USA).

Fiber morphology

By JEOL JSM-6610LV scanning electron microscope (SEM) (JEOL, Akishima, Tokyo, Japan) in low-vacuum condition (0.9 torr) with a voltage of 15 keV, images of *T. monspessulana* fibers were obtained. The images were developed with JEOL MP-45030TDI's three-dimensional image software (Akishima, Tokyo, JAPAN) of 30 *Teline* fibers. Furthermore, microfibrillar angles were obtained by analyzing images at 1100x.

Nanoindentation

T. monspessulana fibers were randomly collected, and three cubes of *c.a.* 5 mm^3 of stem were dried for 3 h at 70 °C and then were mounted in epoxy resin. The sides of *Teline* samples placed in resin, were smoothed with a razor blade, and finely polished by a Leica RM2265 rotary microtome (Leica, Wetzlar, Germany) with a roughing of *c.a.* 250 nm using a glass and diamond knife to secure a good surface of indentation according to the technique employed in previous literature (Jakes *et al.* 2007).

A Hysitron TriboIndenter TI-900 (Hysitron Inc., Minneapolis, MN, USA) nanoindenter, equipped with *in situ* scanning probe microscopy (SPM) imaging technology and a diamond cube-corner tip of pyramidal shape with a curvature radius of approximately 100 nm was used for the indentation tests, with a load displacement resolution of 50 nN and 0.1 nm.

The nanomechanical responses were obtained by charge-discharge cycles of the tip on the middle lamella (S2 layer). The tests were performed with a progressive increment of displacement up to a maximum load of 100 μN, stopping the indenter tip for 5 s, as was done by Bhustan and Li (2003) and Valenzuela *et al*. (2012).

According to the Oliver and Pharr (2004) procedure, the *P-h* data curve provides maximum load value (P_{max}) and maximum penetration depth (h_{max}). These parameters

make it possible to calculate the hardness (H) of fibers, which was obtained from the ratio of the maximum load (P_{max}) of the indenter with regards to the contact area (A_c) of the tip. The elastic modulus was obtained from the reduced or effective (E_r) modulus estimated by the Sneddon ratio (Sneddon 1965), which was calculated according to the relationship of the Berkovich indenter (Fischer-Cripps 2006), considering an angle of $\theta = 35.26°$ for the cube-corner tip (Fischer-Cripps 2006). In fact, the effective elastic modulus takes account of the mechanical behavior of all the materials involved in the assembly of the samples.

Mechanical behavior of fiber sheets

Fibers of T. *monspessulana* prepared in form of fiber paper sheets, according to the standard procedures stated in TAPPI T205 om-88 (1988).

Paper strips of 15-mm-width and 160-mm-length were procured to evaluate the rupture length using a Thwing-Albert 37-4 electro hydraulic mechanical testing machine (Thwing-Albert Instrument Company, West Berlin, NJ, USA), according to the TAPPI T404 cm-92 (1992) standard technique. Furthermore, using an Elmendorf Tearing Tester from Thwing-Albert Instruments (Thwing-Albert Instrument Company, West Berlin, NJ, USA), were obtained the resistance to tearing, as has been suggested in the TAPPI T414 om-88 (1988) standard. As a final point, the burst index was measured with a Mullen Testers AH (Chicopee, USA) testing machine, following the TAPPI T403 om-91 (1991) standard.

RESULTS AND DISCUSSION

Bioorganic Components of Green Source

The chemical components of *T. monspessulana* are reported in Table 2, where the fiber humidity is not included. Cellulose, lignin, hemicellulose, extractives, and ash contents were within the ranges of typical reference values of Fabaceae shrubs (Dalimova and Abduazimov 1994; González-Andrés and Ortiz 1996; González-Andrés and Ceresuela 1998; Foxcroft *et al.* 2017). However, these contents are subject to the sensitivity of the different species to the prevailing edaphoclimatic conditions of their native origins (González-Andrés and Ortiz 1996; González-Andrés and Ceresuela 1998; Bar-On *et al.* 2014). Nevertheless, the contents of the main bioorganic components in *Teline* were comparable to the values reported by other industrial sources of Chilean biomass, such as *Eucalyptus* and Radiata pine (Mansilla *et al.* 1991). Still, the extractives and ash content were higher than those sources. When comparing these contents with other typical non-wood species (Khalil *et al.* 2012), there was no correlation characteristic of bioorganic composition. In fact, *Teline* has the characteristics of a hardwood plant, as already indicated for other shrub species with similar functional characteristics, such as *Ulex europaeus* (Pesenti *et al.* 2017).

Table 2. Comparative Data of Bioorganic Composition of Green Sources

	T. monspessulana		*Pinus radiata* (Mansilla *et al.* 1991)
Main Compounds	Stem (wt.%)	Fiber (wt.%)	Wood (wt%, <2% error)
Extractives	7.5 ± 0.05	3.7 ± 0.09	1.80
Ash	0.95 ± 0.02	1.5 ± 0.05	0.46

Lignin	19.6 ± 0.60	11.3 ± 0.40	27.5
Hemicellulose	25.2 ± 1.46	29.8 ± 0.64	28.2
Cellulose	47.6 ± 1.05	54.8 ± 0.88	45.6

Despite the aggressiveness in the thermos-alkaline pulping process, the *Teline* fibers still retain an important amount of residual lignin. This phenomenon may be due to the particular characteristics of lignin of this species (Dalimova and Abduazimov 1994). Therefore, it is possible that *Teline* fibers have a high amount of insoluble lignin that reacts nucleophilically. When this lignin is dissolved it contains a large number of free phenolic groups as compared to the reduced amount of aryl ether β-O-4 bonds and an increased amount of C-C bonds, which prevent further fragmentation reactions; such a situation can cause the residual lignin not to be reactive to the chemicals of the process (Gierer 1986; Tsutsumi *et al.* 1995; Brännvall 2017).

Morphology of the Fiber

Figure 1 shows libriform fibers with length and width averages of approximately 693 μm and 9.4 μm, respectively. The maximum aspect ratio was 185:1, and the minimum was 25:1 (Table 3). The fiber length value is very similar to *Eucalyptus* as reported by Chinga-Carrasco *et al.* (2011) who observed a length of about 640 μm. However, the width is two times bigger than that of the *Teline* fibers. The Radiata pine fibers have a length of about 2,070 μm and a width of *c.a.* 34.2μm: dimensionally, these values are higher than the *Teline* fibers parameters (Chinga-Carrasco *et al.* 2011). Furthermore, the fiber had a compressed circular-shaped section of cells according to the packaging design of the microfibrils (Fig. 1).
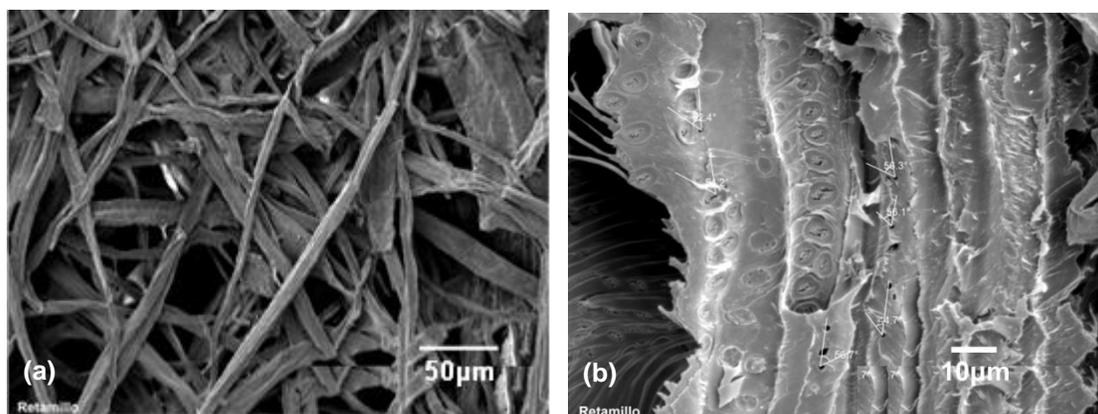

Fig. 1. SEM Images of *Teline* fibers (a: isolated fibers by thermo-alkaline treatment, and b: fiber in wood)

The average thickness of the S2 layer ranged from 3 μm to 4 μm with an approximate microfibril angle of 54.9°. From the point of view of mechanical optimization, these characteristics show a juvenile wood of great extensibility and high elongation of the fibers (Lichtenegger *et al.* 1999; Burgert and Fratzl 2009; Bar-On *et al.* 2014). Nevertheless, fibers growth and development depends on the ability of cells to enlarge during their differentiation. Normally, young microfibril have an angle of about 37° to 55°; however these parameters depend of species, genotype, wood age, hydrostatic pressure, external loads, soil, *etc.* (Barnett and Bonham 2004)

**Table 3.** Morphological Characteristic of Fibers

Measurements	Number of Measurements	Min.	Max	Mean	Median	Standard Deviation
Length (μm)	56	375	1128	693	680	167
Diameter (μm)	56	6.1	15.1	9.4	9.3	2.1
Microfibril angle (Sexag.)	40	43.537	58.500	54.875	55.967	3.325

Structure/Microstructure and Thermal Analysis of Cellulose

The XRD patterns of *Teline* fibers (Fig. 2) showed a combination of triclinic I_α (space group P1) and monoclinic I_β cellulose (P21) phases (Gardner and Blackwell 1975; Sugiyama *et al.* 1991; Nishiyama *et al.* 2002), corresponding ICDD PDF-2 cards for the two cellulose polymorphs, 56-1719 and 56-1718, respectively.

The Z-discriminant allows for the determination of the principal cellulose polymorph by analyzing the peak position of the (101) / (10-1) doublet (Wada and Okono 2001). Hence, the cellulose in *T. monspessulana* was described as a cotton-ramie type, considering a prevalence of the I_β polymorph. This feature was typically detected in hardwood species.

The CI and Cx of the fibers were 78 and 41%, respectively, while crystalline domain size of the cellulose in the fibers was *c.a.* 3.2 nm. Therefore, the fibers are disposed of small I_β/I_α domains embedded in an amorphous matrix (Atalla and Vanderhart 1984). Something similar occurs with other interesting leguminous plants such as *Ulex europeaus* (Celis *et al.* 2014; Peseti *et al.* 2017). The morphological data of the fibers seemed to identify the main characteristics of a shrub plant of the family Fabaceae, a species of fast vegetative growth, with quick adaptability and high aggressiveness with their surroundings.
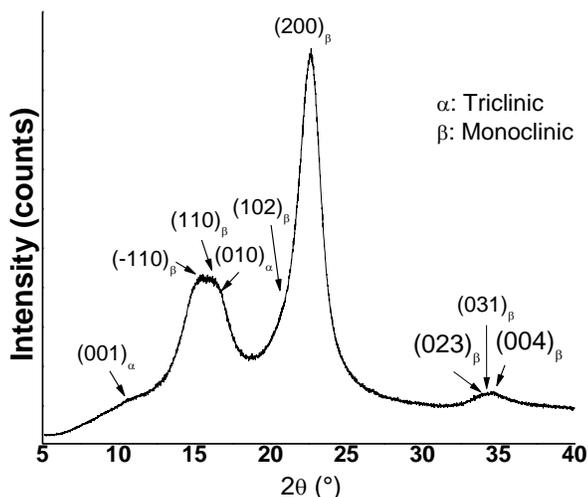
Fig. 2. X-ray diffraction pattern of *T. monspessulana*

The DSC plot in Fig. 3 shows the decomposition of the molecular structure of the bioorganic polymers found within the *Teline* fibers. In the first stage, *c.a.* 147 °C to 300 °C, a weak and broad endothermic peak was observed at 229 °C, possibly due to the decomposition of hemicellulose (Tsujiyama and Miyamori 2000; Yildiz and Gümüşkaya 2007). A large endothermic signal with two split peaks occurring at *c.a.* 355.3 °C and *c.a*

361.0 °C, could be attributed to the process of phase transition and fusion of the cellulose, respectively (Ramiah 1970; Shen *et al.* 2010). A further exothermic peak, observed at 422.9 °C, could be associated with the molecular decomposition of the cellulose and lignin (Raemy and Schweizer 1983). Above 430 °C there was a gradual increase of the thermal signal, possibly due to the continuous molecular degradation of lignin (Shen *et al.* 2010).
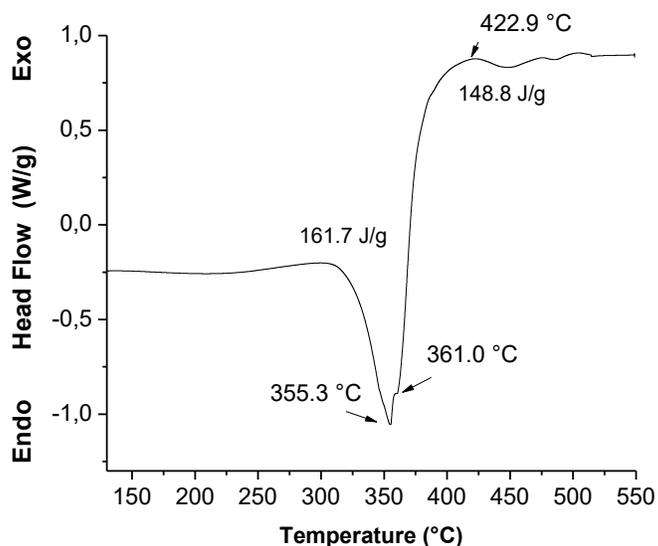

Fig. 3. DSC thermogram of the *Teline* fibers

Nanomechanical Properties

Within the context of mechanical properties (Table 4), the elasticity modulus for *Teline* fibers showed an average of 10 GPa in the isolated fibers. However, fibers measured directly on the wood had a much higher average value, *c.a.* 16 GPa. Meanwhile, the data of wood fibers showed a greater dispersion, compared to those prepared in resin.

Table 4. Nanomechanical Data of *T. monspessulana* Fibers

	Mean	Median	Standard Deviation
Elasticity of the fiber in resin (GPa)	10	9.3	3.4
Elasticity of the fiber in wood (GPa)	16	13.1	7.5
Hardness Fibers in resin (MPa)	290	300	0.048
Hardness Fibers in Wood (MPa)	280	270	0.055

According to the differences in the elasticity values between the different forms of preparation of the samples, the resin fibers showed the lowest values. This could be due to the mode of release of the fibers, because the process of delignification is carried out through a thermo-alkaline process that also acts strongly on the cell walls of the fibers and promotes the detriment of mechanical properties (Moon *et al* 2009).
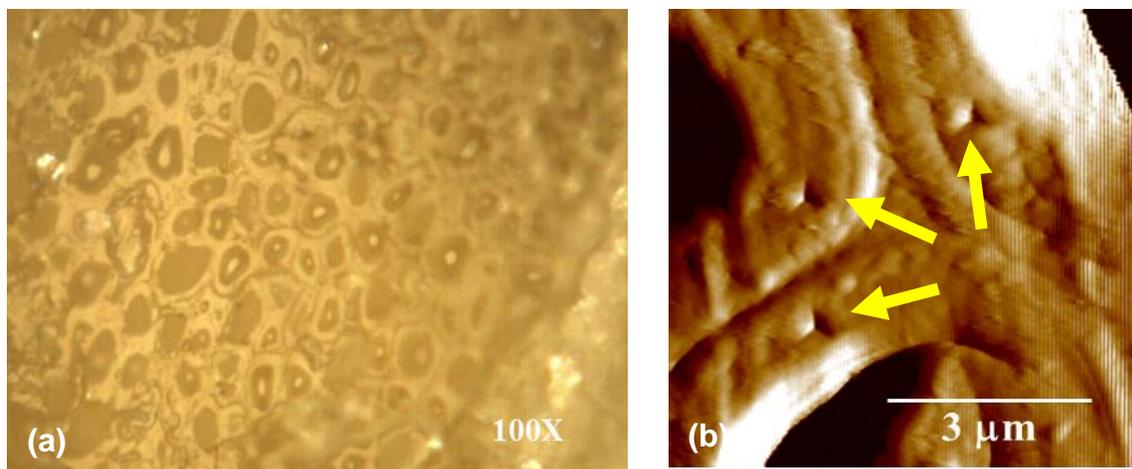
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Fig. 4. (a) Image of traversal section of fiber in wood, and (b) indentation SCM Images: diamond cube-corner is shown the tip impression in S2 lamellar wall

In contrast, regarding the hardness of the fiber in the resin encapsulated and wood modes, it is possible to observe values *c.a.* 290 MPa and 280 MPa, respectively. These values, together with their standard deviations, do not present a great dispersion; they are within both the expected values as well as the hardness values obtained by the fibers of *Ulex europeaus* (Celis *et al.* 2014). This notwithstanding, industrial fibers such as *Pinus radiata* showed a nanoindentation hardness of *c.a.* 358 MPa in latewood according to data reported by Monn *et al.* (2009). *Eucalyptus nitens* fibers also exhibited hardness and elastic modulus of about 310MPa and 12.52GPa respectively (Muñoz *et al.* 2012). According to the tendency of nanomechanical properties of fibers; the reported values are in the range of about 13.5 GPa (earlywood) and 21.0 GPa (latewood) for modulus of elasticity and 250 MPa (earlywood) and 330 MPa (latewood) for hardness (Wimmer *et al.* 1997).

Physico-mechanical Properties of Fiber Sheets

From the calculated physical properties, *Teline* fiber paper had an average density of 0.48 g/cm^3. A very similar value was reported for *Ulex europeaus* fiber papers (approximately 0.46 g/cm^3). In contrast, the mechanical properties of pressed fiber paper sheets of *Teline* were slightly higher than those of *Ulex europeaus* fiber paper, reaching a tensile strength of 2 kg (Table 5).

The reduction of mechanical properties of the *T. monspessulana* sheets could be attributed to having a fiber length that was about 3 to 4 times shorter than that of commercial papers. This discussion was also considered for *Ulex europeaus* (Celis *et al.* 2014; Pesenti *et al.* 2017). Additionally, there are a considerable number of tracheids and vessel elements in the fiber paper that influence its physical and mechanical properties, including a lesser morphogenic maturity of *Teline* fibers. These effects cause a decrease in the resistance to breakage and deteriorates the quality of the fiber paper sheets.

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Table 5. Physico-mechanical Properties of Fiber Sheets

Test Parameter	Teline monspessulana	Ulex europaeus*	Pinus radiata*
Density (g/cm^3)	0.48	0.46	0.52
Tensile strength (kg)	2.0	1.8	3.9
Moisture content wet basis (%)	5.82	5.49	6.07
Rupture length (km)	1.79	2.13	4.43
Tensile index (kNm/kg)	16	20.9	43.5
Tear index (Nm^2/kg)	8.9	8.6	21.2
Burst index (mN/kg)	1.3	1.2	4.1
Maximum explosion strength in 2 sheets (kPa)	156.4	137.8	489

* Celis *et al.* 2014

CONCLUSIONS

1. Information about *Teline monspessulana*, as a renewable biomass source having a large availability, was investigated. Bioorganic components analysis revealed that, despite the aggressiveness in the thermos-alkaline pulping process, there remain a high content of residual lignin. However, the cellulose in teline stem is present about to 4% more than in the Pinus radiata wood referred in this publication.

2. The natural controlled crystalline ultra-structure of cellulose in the fibers was of about 3.2 nm, despite the significant proportion of amorphous cellulose of *c.a.* 41%. This finding means that this green source can be a potential candidate to develop controlled sizes of nanocrystalline cellulose for advanced nanoreinforced materials.

3. The nanoindentation hardness and elastic modulus values for the fibers in the stem were *c.a.* 280 MPa and 16 GPa, respectively, while the values for the isolated fibers were 290 MPa and 10 GPa, respectively. The mechanical properties of the *Teline* fibers compared well with other industrial source of fibers such as the *Eucalyptus, pine* and others biofiber sources. However, dimensional aspect in *Teline* fibers with length *c.a.* 693 μm, is not attractive for conventional applications because this is related with the mechanical properties in textile applications and papers sheets which uses low tensile strength, 1.79 kg, and low rupture length, 1.79 km.

4. This green source could be useful in high-end technological applications such as reinforcements in foams, nanopapers, cements, hierarchical materials and modern electronic materials (breadboards/computer mother board substrates). Due to its insulating characteristics, *Teline* biofibre-based composites may find areas of applications in automotive door/ceiling panels and panels separating the engine and passenger compartments. Moreover, it is a good opportunity to deepen research into this species, since it is a potential sustainable source of cellulose, which has a high growth rate and can adapt to a wide range of climatic and edaphic conditions.

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