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ORIGINAL RESEARCH

Extraction and characterization of cellulosic fiber from *Centaurea solstitialis* for composites

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Abstract With increasing ecological awareness, researchers around the world seek to make composites reinforced with more eco-friendly natural fibers. In this study, *Centaurea solstitialis* (yellow star thistle) fibers were characterized as a potential natural fiber reinforcement for green composites. Generally, *Centaurea solstitialis* plants are undesirable because of their detrimental effect on other plants. In this respect, they are generally considered economically worthless in terms of the economical aspect. From this point of view, characterizing *Centaurea solstitialis* and using them as a natural fiber reinforcement material can be more appropriate in terms of waste management. To characterize the *Centaurea solstitialis* fibers, Scanning Electron Microscopy, Fourier Transform Infrared Spectroscopy, Thermogravimetric Analysis, X-ray Photoelectron Spectroscopy, X-Ray Diffraction Analysis, single fiber tensile test, determinations of density and chemical composition were performed. *Centaurea solstitialis* fibers have 11.2% hemicellulose and 57.20% cellulose content. The crystallinity index and density of fibers were determined as 71.43% and 1.37 g/cm^3, respectively. Also, fibers exhibited relatively high tensile strength with 111.85 ± 24.97 MPa and Young's modulus with 3.41 ± 0.62 GPa. The thermal resistance temperature of *Centaurea solstitialis* fibers was found as 273 °C. It is suggested that *Centaurea solstitialis* fiber is a suitable reinforcement candidate for composites with low density, low cost, abundancy and relatively high tensile strength.

Keywords *Centaurea solstitialis* · Natural fiber · Green composites · Waste management

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Introduction

Today, the usage of composite materials becomes widespread in terms of their superior performance and economical aspect when comparing with traditional materials. Initially, composite materials were used in military and special aerospace structures because of their benefical mechanical properties to lightweight ratio and thermal stabilities at high temperatures. Over the years, composite materials have become prominent for civil applications. In many composite applications, synthetic fibers like aramids, and glass are generally used as reinforcement. Although composite materials have already proved their dominance comparing to conventional materials, nowadays researchers seek to make them more cost-effective and eco-friendly (Chandramohan and Marimuthu 2011). In this respect, with increasing energy consumption and environmental contamination, and also increasing environmental awareness across the world, interest toward green composites has been increased (Ganapathy et al. 2019).

Recently, since the natural fibers meet the main requirements of composites as a reinforcement such as accessibility, affordability and comparable properties regarding traditional composites they have been investigated in many studies as an alternative reinforcement instead of traditional reinforcements. Green composites have been used in many applications such as construction, aerospace, defense, and automobile industries owing to their desirable properties namely low cost, lightweight, eco-friendly, renewability, recyclability, and biodegradable properties (Chandramohan and Marimuthu 2011; Arthanarieswaran et al. 2015; Kilinc et al. 2016; Manimaran et al. 2016). Cellulose-based natural fibers provide relatively high mechanical strength, stiffness, and modulus values as compared to artificial fibers. Therefore, they are evaluated as suitable and more eco-friendly reinforcement material in green composite applications (Kalusuraman et al. 2019).

Centaurea solstitialis, also known as yellow starthistle comes from the Asteraceae family. Although *Centaurea solstitialis* is native to the Mediterranean region, it has spread to the six continents (Hierro et al. 2009; Dukes et al. 2011). On the other hand, *Centaurea solstitialis* is a highly invasive and ecologically impactful plant. They are generally infamous with their detrimental effect on other species and toxic properties (Lu-Irving et al. 2019). From this point of view, they are generally undesirable plants and not valuable in terms of economic aspects. In this respect, *Centaurea solstitialis* fiber can be a prominent alternative reinforcement for the production of the green composites in terms of availability and abundancy across the world and also in terms of cost-effectiveness.

In this study, the structural, thermal, morphological and mechanical properties of *Centaurea solstitialis* fibers have been investigated for the first time. From this point of view, X-Ray Diffraction Analysis (XRD), Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetric Analysis (TGA), X-ray Photoelectron Spectroscopy (XPS), density measurement, chemical analysis, and single fiber tensile tests were carried out for characterization of *Centaurea solstitialis* fibers.

Experimental

Fiber preparation

Centaurea solstitialis plants were obtained from the city of Çanakkale, which is located about the Aegen region of Turkey. In order to prepare *Centaurea solstitialis* plants for extraction, plants were cleaned with distilled water, and then plants were cut into pieces. To remove impurities from the fibers and to permit microbial degradation, the soaking process was implemented for four weeks. To obtain fibers, the metal comb was used for the separation of fiber from stems. To evaporate the moisture, fibers were oven- dried for 24 h at 60 °C. The images of the (a) *Centaurea solstitialis* plant and (b) extracted fibers are presented in Fig. 1.

Characterization

Fiber composition

Centaurea solstitialis fibers were dried to remove moisture at 105 °C in an oven and then fibers were kept in a desiccator before chemical processes in order to avoid humidity. The procedure of the processes is given in previous studies (Mylsamy and Rajendran 2010; Kilinc et al. 2016).

Density measurements

The density of fibers calculated according to Eq. 1 in ASTM D8171-18, Archimedes Method (Method B), and three samples with approximately 1 grams weight were used to get the average density of fibers.

$$d = \frac{W_d}{W_d - W_s} \quad (1)$$

where d is the density, Wd and Ws are the dry mass of the fibers after 24 h of drying at 60 °C, and the submerged weight of the samples after 24 h of boiling in deionized water, respectively.

Fourier transform infrared (FTIR) analysis

Functional groups of *Centaurea solstitialis* fibers were determined with the Perkin Elmer Spectrum BX

(a) (b)

Fig. 1 Images of the *Centaurea solstitialis*, **a** plants and **b** fibers

Fourier Transform Infrared spectrophotometer. Measurements were obtained in between 650 and 4000 cm^{-1} wavenumber with a scan rate of 40 acquisitions and at a resolution of 2 cm^{-1}.

TGA analysis

The thermal behavior of *Centaurea solstitialis* fibers was characterized by using a Shimadzu DTG-60H instrument. Measurement was conducted in the range of 25–800 °C with a heating rate of 10 °C/min under a Nitrogen atmosphere.

XPS analysis

To determine surface chemistry and characteristic of fibers, X-ray Photoelectron Spectroscopy (XPS) was utilized. Measurement was conducted with Thermo Scientific instrument using Al-Kα X-ray source (1486.7 eV) between 1350 and 10 eV with a resolution of 1 eV. Before the analysis, the surface of samples was cleaned with Ar gas, and analysis was recorded with 20 scans.

XRD analysis

Crystallinity index (CI) calculation was performed by using the XRD pattern obtained from Rigaku Ultima 3 device. Copper X-ray tube was used as the radiation source (λ-Cu-Kα_1 = 1.54 Å) and power was kept at 40 kV − 30 mA. Scanning was done between 5° and 80° 2θ range with the scan rate of 2°/min. CI calculation was completed by using the empirical formula (see Eq. 2) suggested by Segal et al. (1959).

$$CI = \frac{(I_{200-}I_{am})}{I_{200}} \times 100 \quad (2)$$

where I_{200} represents the peak with the highest intensity that relates to the lattice plane (200) between 22° and 23°, and I_{am} represents the minimum intensity near 18.5° (Seki et al. 2018).

Tensile test

Centaurea solstitialis fiber samples were subjected to tensile tests using INSTRON 4411 universal testing machine with 1 mm/min cross-head speed and 50 mm gauge length. Before the tensile tests, all samples were conditioned in accordance with EN ISO 139:2005, and tests were carried out at standard atmospheric conditions. The average fiber diameter was measured using an optical microscope from the longitudinal view of fiber in order to determine cross-sectional area and accordingly the mechanical properties of *Centaurea solstitialis* fibers. 11 different measurements were conducted from each fiber and the average diameter was calculated.

Morphological characterization

Before morphological characterizaiton, in order to prevent charging effect *Centaurea solstitialis* fibers were coated. Secondary electron images were taken by JEOL-JJM 6060 model scanning electron microscope (SEM), to examine the surface and cross-sectional surface morphologies of *Centaurea solstitialis* fibers. The accelerating voltage value was kept as 5 kV.

Result and discussion

Chemical composition of *Centaurea solstitialis* fibers

Generally, lignin, hemicellulose, and cellulose are considered as the major components of lignocellulosic fibers. *Centaurea solstitialis* fiber consists of experimentally 57.2% cellulose. Table 1 lists the cellulose contents of *Centaurea solstitialis* and some lignocellulosic fibers to make a possible comparison. Chemical analysis of *Centaurea solstitialis* showed comparable cellulose content with common cellulosic fibers such as flax, kenaf, and linden fibers which can ensure its reinforcement effect for polymeric composites. The cellulose content of *Centaurea solstitialis* is higher than recently characterized *Tridax procumbens, Conium maculatum* and *Curcuma longa L.* fibers (Vijay et al. 2019; Kılınç et al. 2018; Ilangovan et al. 2018; Shyam Kumar et al. 2019). The estimated hemicellulose content of *Centaurea solstitialis* is 11.2% that is lower than banana (38.5%), bagasse (56.7%), ramie (5–16.7%) and kudzu fiber (11.6%) (Jústiz-Smith et al. 2008; Moshi et al. 2020). The higher amount of hemicellulose may have an adverse effect on fiber strength (Moshi et al. 2020).

Density measurements

The density of *Centaurea solstitialis* bast fibers was calculated as 1.37 g/cm^3. As seen from Table 2, the density of *Centaurea solstitialis* fibers is similar to many common bast fibers used in the composite field, such as ramie, flax, and kenaf.

FT-IR analysis

Fourier Transform Infrared (FTIR) spectroscopy was used for clarifying the functional groups (ester, ketone, and alcohol) and the main component of fibers (lignin, cellulose, and hemicellulose) (De Rosa et al. 2010; Fan et al. 2012; Sanjay et al. 2018). Figure 2 shows the FTIR spectrum of *Centaurea solstitialis* fibers. The band at 3340 cm^{-1} is associated with the stretching vibrations of O–H bondings in cellulose (Porras et al. 2015). The little peak located at 2907 cm^{-1} is regarding stretching vibrations of C–H bondings of hemicellulose in fibers (Oh et al. 2005). The band ranging from 1700 to 1600 cm^{-1} may be attributed to water content in fibers (Manimaran et al. 2018). The absorption peak at 1510 cm^{-1} can indicate lignin content in the fiber (Kılınç et al. 2018). The

Table 1 Cellulose fractions of *Centaurea solstitialis* and some lignocellulosic fibers

Fiber	Cellulose content (%)	References
Centaurea solstitialis	57.20	In current study
Hierochloe Odarata	70.40	Dalmis et al. (2020b)
Cereus Hildmannianus	58.40	Subramanian et al. (2019)
Tridax procumbens	32.00	Vijay et al. (2019)
Curcuma longa L.	50.00	Ilangovan et al. (2018)
Epipremnum aurem	66.34	Maheshwaran et al. (2018)
Conium maculatum	49.50	Kılınç et al. (2018)
Jute	79.00	Bulut and Aksit (2013)
Kenaf	52.00	Abdul Khalil et al. (2010)
Flax	64.10	Nilsson and Gustafsson (2007)
Hemp	68.10	Nilsson and Gustafsson (2007)
Coir	36–43	Rout et al. (2001)

Table 2 Density values of *Centaurea solstitialis* fibers with some commonly used natural bast fibers

Fiber source	Density (g/cm^3)	References
Centaurea solstitialis	1.37	Present study
Ramie	1.38	Munawar et al. (2007)
Flax	1.30–1.44	Nabi Saheb and Jog (1999)
Kenaf	1.45	Holbery and Houston (2006)
Jute	1.40–1.5	Li et al. (2007)
Hemp	1.48	Wambua et al. (2003)

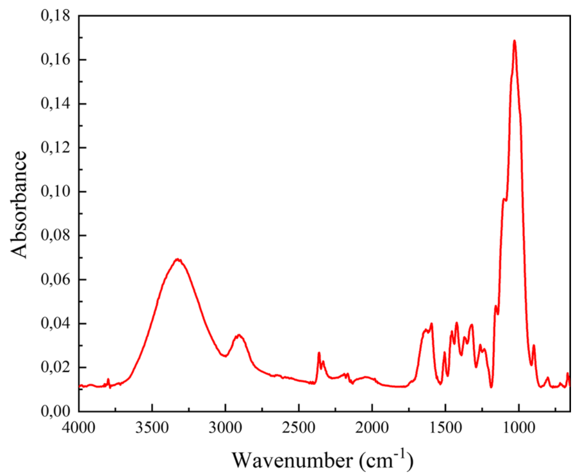

Fig. 2 FTIR spectrum of *Centaurea solstitialis* fibers

peaks at 1428 and 1377 cm^{-1} may confirm bending vibrations of C–H bondings of aromatic rings in hemicellulose (Sreenivasan et al. 2011). The two little peaks located at 1330 and 1250 cm^{-1} indicate stretching vibrations of the C–O bondings of the acetyl groups in hemicellulose (Tawakkal et al. 2016). The extensive band is located at 1028 cm^{-1} related to the stretching vibrations of C–O bondings in fibers (Dalmis et al. 2020a, b).

Thermogravimetric analysis

Figure 3 lists the thermogravimetric analysis of the fibers. The first decomposition occurred between 25 and 100 °C due to the dehydration of the fibers with an

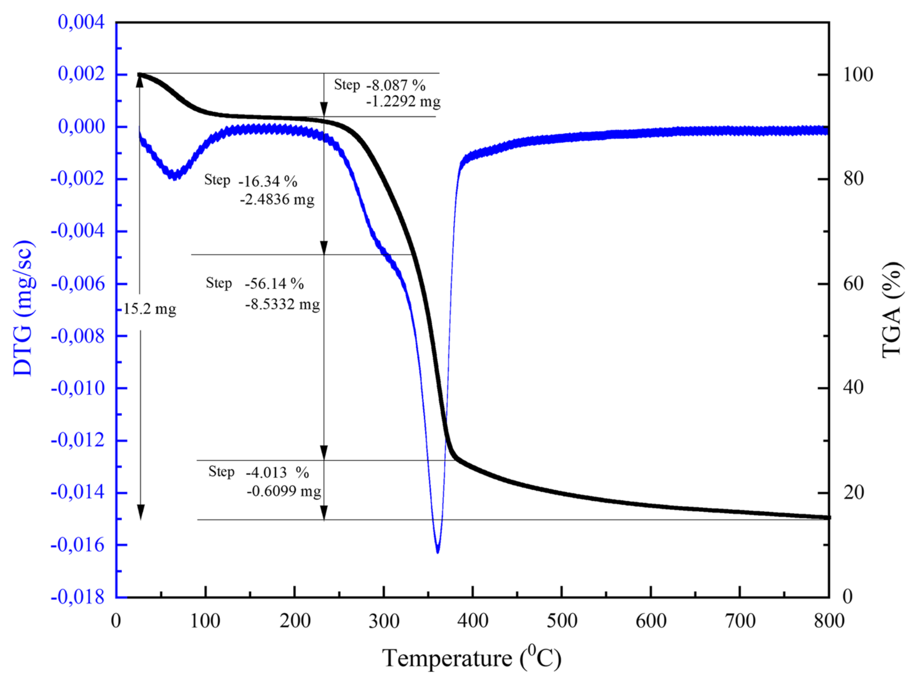

Fig. 3 TG/DTG analysis of the *Centaurea solstitialis* fibers

Table 3 Thermal degradation temperature comparison of CS fibers with other most used natural fibers

Fiber Source	DTG Peak (°C)	References
Centaurea Solstitialis	360	–
Kenaf	364	Poletto et al. (2015)
Jute	365	Alvarez et al. (2006)
Sisal	340	Manfredi et al. (2006)
Flax	345	Manfredi et al. (2006)

8.087% weight loss (Ridzuan et al. 2016). The next weight loss of 16.34% occurred at 273 °C which is regarding the degradation of hemicellulose in fibers (Arthanarieswaran et al. 2015). The major weight loss was recorded between 280 and 420 °C which shows a distinct peak at 360 °C with 56.14% owing to the decomposition of the cellulose in fiber (Baskaran et al. 2018; Amroune et al. 2019). The same results were recorded in the previous studies and listed in Table 3 for comparison with other natural fibers. Up to 800 °C, 4.013% weight loss was recorded associated with residual content of fiber (Balasundar et al. 2018). Consequently, 273 °C was determined as the thermal resistance temperature of *Centaurea solstitialis* fibers.

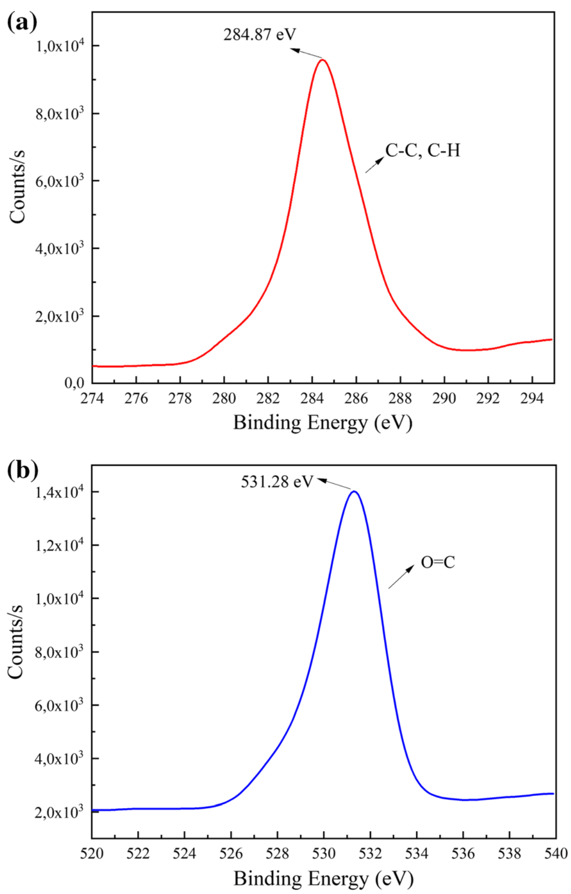

Fig. 4 The high-resolution spectra of **a** C1s and **b** O1s peaks.

XPS analysis

The XPS spectra of fibres related to the C1s and O1s peaks were given in Fig. 4. Also, the elemental composition of fibers was given in Table 4. According to results, carbon and oxygen amounts of fibers were found as 59.08 and 32.17%, respectively. To determine the surface characteristic of *Centaurea solstitialis*, the ratio of Oxygen /Carbon (O/C) and Carbon/ Oxygen (C/O) was calculated as 0.54 and 1.83, respectively. The O/C ratio of *Centaurea solstitialis* is higher than the most utilized fibers used in natural fiber-reinforced composite such as flax (0.156) (Csiszár et al. 2013), jute (0.46) (Bulut and Aksit 2013), kenaf (0.45) (Sgriccia et al. 2005) fibers. In this regard, with higher C/O ratio (1.83) of *Centaurea solstitialis* fibers show a hydrophobic surface characteristic that important parameter for cellulose-based reinforced composites (Šernek et al. 2004).

The high-resolution XPS spectra of C1s and O1s peaks were given in Fig. 4. The peaks located at 284.87 and 531.28 eV can be associated with C–C/C–H and O=C groups, respectively (Kılınç et al. 2018; Dalmis et al. 2020b). Deconvolution analysis was conducted to the O1s and C1s peaks to calculate the amounts of functional groups. The ratio of C–C/C–H calculated as 51.73% and C=O is founded as 33.32%. It is clear that the great proportion belongs to the C–C/C–H groups. The existence of C=O groups may confirm the existence of hemicellulose.

XRD analysis

The main peak seen in the XRD pattern (see Fig. 5) is related to the cellulose (200) lattice plane, which is at

22.18°. The first peak at 15.86° is is mainly attributed to cellulose-I, that (110) and $(1\bar{1}0)$ lattice planes overlap. The minimum intensity value between these peaks can be seen at 18.34°. Also, a weak peak at 34° can be assigned to the (004) plane (Oh et al. 2005).

The crystallinity index (CI) of *Centaurea solstitialis* fibers is 71.43%, which is higher than most the natural fibers which used as reinforcement in many composite applications without applying any chemical treatment, such as okra (63.5, (Seki et al. 2019)), jute (50, (Frederick and Norman 2004)), sisal (57, (Frederick and Norman 2004)), and hemp (64.87, (Sunny et al. 2020)). Higher CI is generally attributed to higher tensile properties, and higher thermal degradation temperature because of the cellulosic chains become more regularly aligned (Ehrenstein 2012).

Mechanical properties of *Centaurea solstitialis* fibers

The single fiber test was used to determine the mechanical properties of *Centaurea solstitialis* fibre. The mechanical data of *Centaurea solstitialis* in comparison with some common cellulosic fibers are tabulated in Table 5. The results showed that *Centaurea solstitialis* fiber has comparable mechanical properties with *Phoenix dactylifera L.*, *Pennisetum purpureum*, *Hierochloe Odarata*, *Juncus effecus L.*, napier grass and pineapple leaf (Amroune et al. 2015; Ridzuan et al. 2016; Maache et al. 2017; Dalmis et al. 2020b). The load-displacement curve of *Centaurea solstitialis* fiber shows that the fiber has a linear characteristic like other cellulosic fibers and exhibit a brittle behavior when fiber failure occurs at maximum load (Fig. 6).

Surface morphology of the fibers

The longitudinal and cross-sectional SEM images of the *Centaurea solstitialis* fibers were presented in Fig. 7 to characterize the morphology of the fibers. It can be interpreted from the longitudinal fiber section (Fig. 7a) that, *Centaurea solstitialis* fiber sample has a diameter of about 123 μm. Some particles which might be wax, lignin or impurity (Senthamaraikannan and Kathiresan 2018); and porosity holes can be observed on the surface of *Centaurea solstitialis* fiber. These irregularities endorse a big advantage with increased surface area, for composite systems by providing better adherence to the fiber with the matrix (Indran et al. 2014). Moreover, as can be seen from the

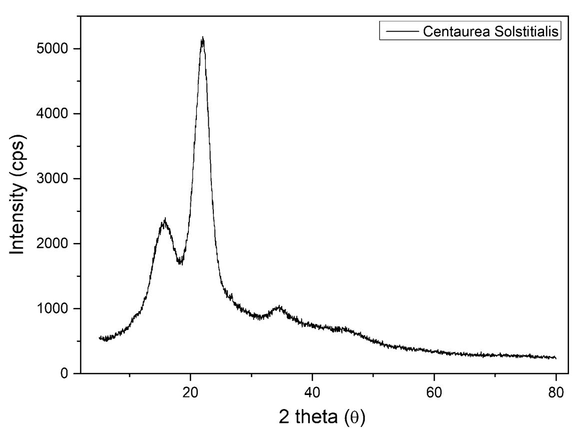

Fig. 5 X-ray diffraction pattern of *Centaurea solstitialis* fibers

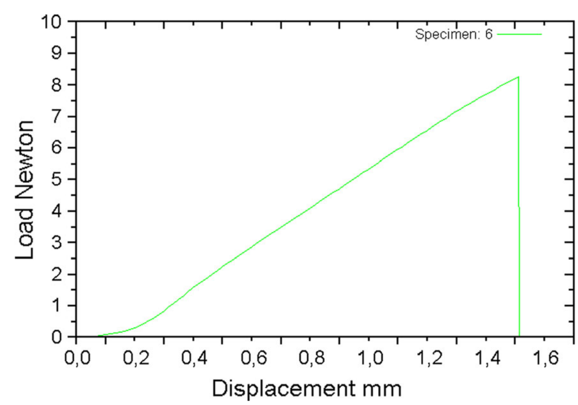

Fig. 6 Load-displacement graph of *Centaurea solstitialis* fiber

Table 4 Surface elemental composition of *Centaurea solstitialis* fibers

	C1s (%)	O1s (%)	Si2p (%)	O/C	C/O
Centaurea solstitialis	59.08	32.17	8.75	0.54	1.83

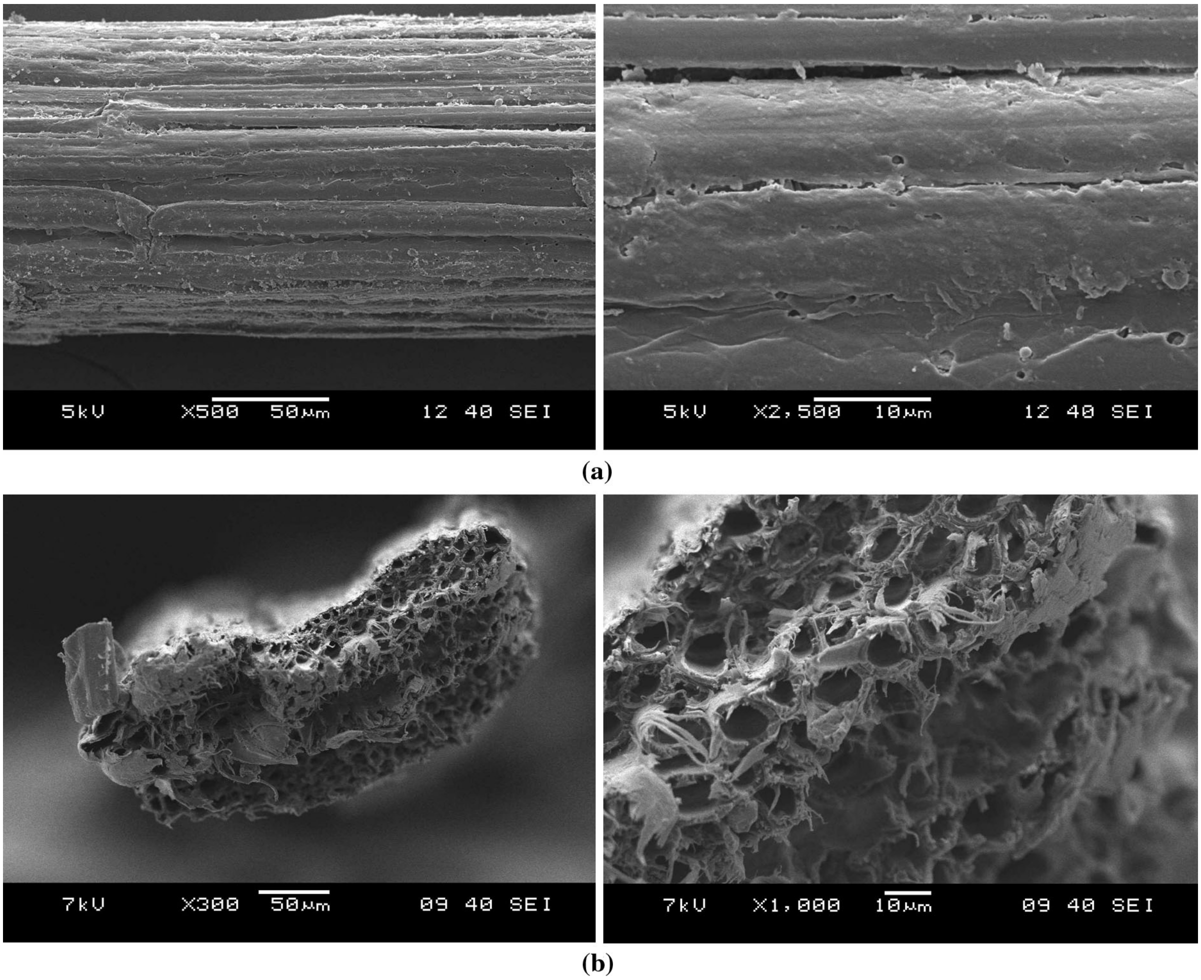

Fig. 7 **a** The longitudinal and **b** cross-sectional SEM images of the *Centaurea solstitialis* fibers

longitudinal fiber section in Fig. 7a, elementary fibers consist of the main *Centaurea solstitialis* fibers which are clearly noticeable and can contribute to increasing fiber surface roughness.The presence of these elementary fibers can be demonstrated by cross-sectional SEM images in Fig. 7b. As can be seen from cross-sectional SEM images, the fiber has an eliptical profile. Section geometry is an important parameter for composite applications (Bezazi et al. 2020). *Centaurea solstitialis* fiber consists of many several elementary fibers bounding together by pectin or other non-cellulosic compounds similar to the other plant fibers (Kılınç et al. 2018). Another important issue is about lumen, which is basically empty space of an elementary fiber. High lumen diameter causes good insulation and absorbance properties to the fiber (Dalmis et al. 2020a). As can be concluded from Fig. 7b, *Centaurea solstitialis* fiber has a diameter of 11 μm, while the cell wall thickness is about 2 μm and lumen diameter is about 7.5 μm.

Energy dispersive X-ray (EDX) spectrum results and elemental mapping analysis images of the *Centaurea solstitialis* fiber are presented in Fig. 8. As can be seen from Fig. 8d (a), C, O, and Ca elements present on the fiber surface at the weight ratios of 60.377%, 37.796%, and 1.827% respectively. The most important thing in these results is O/C ratio. O/C ratio of the cellulose, hemicellulose, and pectin known as 0.83 while lignin has a ratio of 0.35 according to the literature (Sgriccia et al. 2008). As mentioned in the

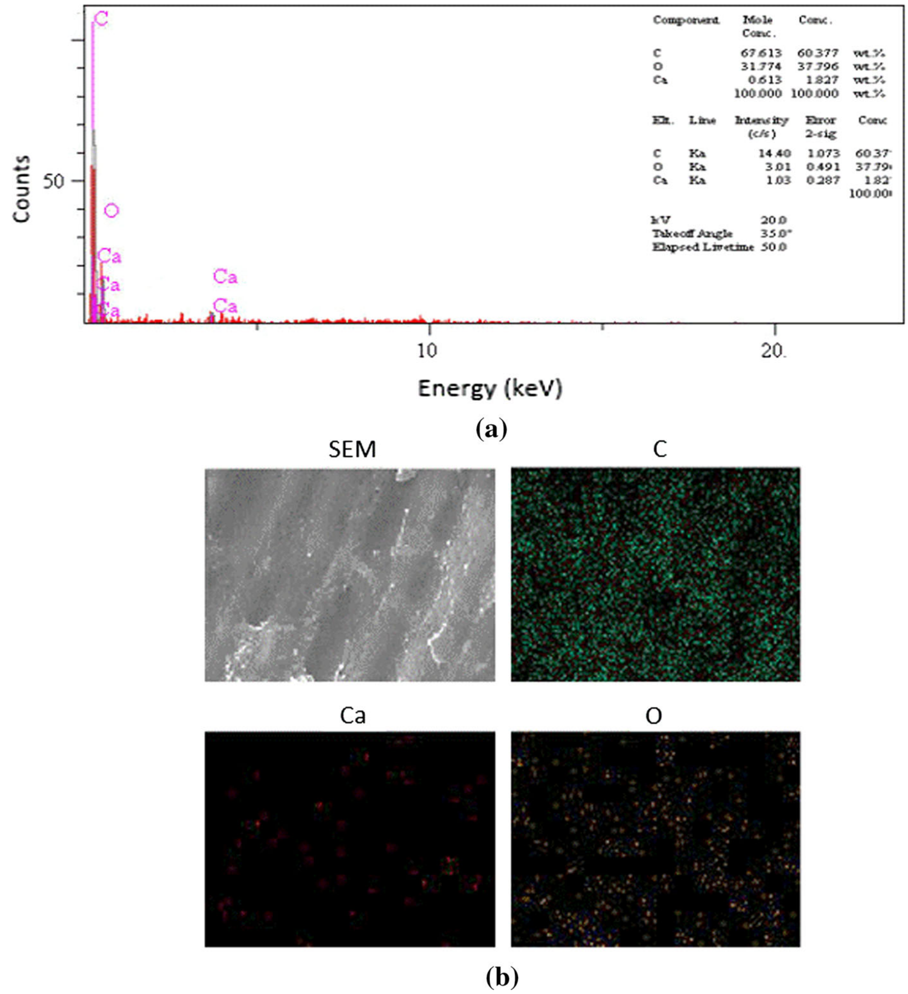

Fig. 8 **a** Energy dispersive X-ray spectrum and **b** elemental mapping analysis images of the *Centaurea solstitialis* fiber

XPS part, the fiber has 57.2% cellulose and 11.2% hemicelluloses, in total, 68.4% of the Centaurea solstitialis fiber consist of these components. In this respect, *Centaurea solstitialis* fiber has O/C ratio of 0.626. According to XPS analysis the O/C ratio of the fiber is close to the ratio of the cellulose and hemicellulose and this supports the EDX results. Also, elemental mapping analysis images of the *Centaurea solstitialis* fiber reveals the distribution of the related elements on the fiber surface (See Fig. 8b). Calcium atoms appear to be rarer while carbon and oxygen atoms are more dominant on the surface, which is coherent with the EDX results. Also, carbon and oxygen distribution seem quite homogeneous while calcium is clustered in some regions.

Conclusions

The main goal of this study is to extract fibers from *Centaurea solstitialis* plant and then characterize some properties essential for usability in polymeric composites. For this purpose,chemical composition, density, surface chemistry and morphology, chemical bonds, crystallinity, thermal decomposition and tensile properties of *Centaurea solstitialis* fibers were investigated. The cellulose content of fibers was determined as 57.20%, which is compatible with thermal analysis. According to thermal analysis, fibers are stable up to 273 °C. The XPS results show that *Centaurea solstitialis* have C–C/C–H and O=C groups and more hydrophobic surface characteristics

Table 5 Mechanical properties of *Centaurea solstitialis* fibre and other fibers

Fiber	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)	References
Centaurea solstitialis	111.85 ± 24.97	3.41 ± 0.62	3.32 ± 0.67	In current study
Piassava	134–143	1.07–4.59	7.8–21.9	Indran et al. (2014)
Phoenix dactylifera L	117 ± 35	4.3 ± 1.4	3.13 ± 0.7	Amroune et al. (2015)
Pennisetum purpureum	73 ± 6	5.68 ± 0.14	1.40 ± 0.23	Ridzuan et al. (2016)
Hierochloe Odarata	105.73 ± 35.42	2.56 ± 0.98	2.37 ± 0.95	Dalmis et al. (2020b)
Juncus effecus L.	113 ± 36	4.38 ± 1.37	2.75 ± 0.6	Maache et al. (2017)
Arundodonax	248	9.4	3.24	Fiore et al. (2014)
Ficus Racemosa	270	67.45	2.57	Manimaran et al. (2019)
Napier grass	88.4	13.1	0.99	Kommula et al. (2013)
Cyperus pangorei	196 ± 56	11.6 ± 2.6	1.69	Vijay and Singaravelu (2016)
Pineapple leaf	126.6	4.405	2.2	Arib et al. (2006)

according to the O/C ratio (0.54) in comparison with some plant fibers. Density, CI and tensile strength of fibers are determined as 1.37 g/cm^3, 71.43%, and 111.85 ± 24.97 MPa, respectively. According to SEM images, *Centaurea solstitialis* fiber contains many elementary fibers bonded together by pectin or other non-cellulosic compounds. Consequently, with relatively high tensile strength and low density, and also economical aspect in terms of waste management, *Centaurea solstitialis* fiber is a suitable candidate for green composites as a reinforcement.

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