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ORIGINAL ARTICLE

Natural cellulosic fiber from *Carex panicea* stem for polymer composites: extraction and characterization

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Abstract

Nowadays, commercial natural fibers cannot meet the increasing industrial demand. In order to meet this demand, recommending a new natural fiber for the composites industry is very important. In this paper, *Carex panicea* fibers were characterized for the first time and introduced as a potential natural fiber. Physical, chemical, thermal, mechanical, and morphological properties of the *Carex panicea* fibers were characterized using scanning electron microscopy, Fourier transform infrared spectroscopy, thermogravimetric analysis, X-ray photoelectron spectroscopy, and X-ray diffraction analysis. *Carex panicea* fibers consist of 65.70% cellulose and 27.8% hemicellulose content. The density and crystallinity index of the fiber were found as 1.247 g/cm^3 and 56.42%, respectively. Tensile strength and Young's modulus of fibers were determined as 143 ± 41 MPa and 5.5 ± 1.86 GPa, respectively. *Carex panicea* fibers are thermally stable up to 219.4 °C. *Carex panicea* fibers are potential bio-degradable reinforcement material for light-weight polymeric composites with relatively enhanced mechanical properties and decomposition temperature.

Keywords Cellulose · *Carex panicea* · Natural fiber · Composites · Characterization

1 Introduction

Recently, with the increasing environmental awareness and economical concerns, the utilization of renewable and eco-friendly resources became important [1–4]. Today, petroleum-based fibers are used as reinforcement for the manufacturing of polymer matrix composites, and these composites are utilized in many applications such as aerospace, automobile, and military [5–9]. While petroleum-based fibers possess high mechanical and physical properties, they have also negative impacts in terms of environmental and economic aspects [10–13]. Nowadays, the advancement of sustainable green technology has increased in the composite industry. However, ensuring high demand for natural fiber-reinforced composites by using commercial plant fibers is difficult. Therefore, the industry has sought a new plant fiber with desired thermal, physical, and mechanical properties [1, 14, 15]. To overcome the drawbacks of petroleum-based fibers and to meet the industrial reinforcement demand for composites, scientists try to replace man-made fibers with eco-friendlier natural fibers [16]. The utilization of natural fibers may help to protect the environment by reducing waste disposal, usage of hazardous material for the production of petroleum-based fibers, and increasing the usage of renewable sources [17].

Cellulose-based natural fibers can be extracted from different plants such as jute [18], kenaf [19], and hemp [20]. These fibers are considered to be a potential alternative to traditional petroleum-based and other synthetic fibers due to their attractive properties such as low density, biodegradability, renewability, cost-effectiveness, and abundancy around the world [3, 17, 21, 22]. Also, natural fibers can provide comparable mechanical properties to artificial fibers such as relatively high mechanical strength, stiffness, and modulus [10, 11, 23]. Researchers currently have focused on the identification and characterization of new natural fibers due to the increasing demand in the composite industry. Within this framework, new natural fibers such as *Leucas aspera* [24], *Conium maculatum* [25], *Eleusine indica* [2], *Hierochloe odarata* (Dalmis et al. 2020), *Ellettaria cardamomum*

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[26], *Purple bauhinia* [27], *Trachelospermum jasminoides* [28], *Cordyline australis* [29], *Lavender stem* [30], *Atriplex halimus* [31], *Coccinia grandis L.* [32], *Lygeum spartum L.* [33], *Cissus vitiginea* [34], *Grewia damine* [35], *Grewia flavescens* [35], *Chrysanthemum morifolium* [36], *Vachellia farnesiana* [37], and *Althea officinalis L* [38]. fibers were identified and characterized. In order to give desired properties to fibers, many modification and treatment studies have been carried out such as *Inula viscosa* [39], *Symphirema involucratum* [40], and *Agave Americana* [41].

Recently, researchers and experts have focused on the characterization of new cellulosic fibers because they represent alternatives to traditional reinforcements [42–51]. *Carex panicea*, also known as grass like plant, comes from *Cyperaceae* family. While *Carex panicea* is native to Europe, they have spread around Asia and America [52, 53]. The availability and abundancy of *Carex panicea* plants around the world make them attractive candidate as reinforcement for green composites. According to literature, *Carex panicea* fibers have not identified yet and within this framework, *Carex panicea* fibers were extracted and characterized for the first time as an alternative reinforcement material for composites. Physical, chemical, thermal, morphological, and mechanical properties of the *Carex panicea* fibers were characterized by the help of Archimedes density method, determination of chemical composition, Fourier transform infrared spectroscopy (FTIR), X-ray diffraction analysis (XRD), X-ray photoelectron spectroscopy (XPS), thermogravimetric analysis (TGA), scanning electron microscope (SEM), and tensile tests.

2 Materials and methods

2.1 Fiber extraction

Carex panicea plants were harvested from Uzunalan, Çanakkale that is located in the west coast of Turkey. Images of *Carex panicea* plants and fiber extraction steps were given in the schematic diagram in Fig. 1. Conventional water fiber extraction technique is utilized to obtain fiber from the stem of *Carex panicea* plants. As a preparation for fiber retting, plants were cleaned and rinsed with distilled water for several times and cut into pieces to facilitate the extraction. For 6 weeks, the plants were placed in a plastic barrel filled with tap water and covered to enable microbial breakdown in order to facilitate fiber extraction from the plant. Fibers were then carefully removed from the stem, combed with a metal comb. Fiber separation process by metal comb was carried out under an aqueous medium. The obtained fibers about 200-µm diameter were washed until cleared completely and rinsed in distilled water. To remove excess moisture, the fibers were oven-dried for 24 h at temperature of 60 °C. Moisture content and moisture regain of the *Carex panicea* fiber were found to be 11.3% and 11.8%, respectively.

3 Characterization of fibers

3.1 Density measurement

The density of *Carex panicea* fibers was identified by using ASTM D8171-18 Method B (Eq. 1). The method is based

Fig. 1 The schematic diagram of the fiber extraction with the *Carex panicea* plant image

on Archimedes law, and approximately 1 g of fiber specimen, coiled like a ball, with three replications according to standard was used for measurements. Specimens were dried at 40 °C for 24 h in a low temperature oven and weighed by using a precision balance (Mettler Toledo ME 204 with density Kit). After dry weights were taken, specimens were immersed in a baker for 24 h, which is containing boiling water. Then submerged weights of fibers were measured.

$$d = \frac{W_d}{W_d - W_s} \tag{1}$$

where d is the density of fibers, W_d and W_s are dry weight, and weight of samples submerged in water, respectively.

3.2 Chemical analysis

The chemical composition of *Carex panicea* fibers was determined by the method explained in details in *related* previous studies [36, 54]. Prior to chemical analysis, *Carex panicea* fibers were oven-dried at temperature of 105 °C for 4 h and then kept in desiccator to avoid humidity.

3.3 Fourier transform infrared (FTIR) analysis

ATR-FTIR spectra were recorded of fibers recorded using the Perkin Elmer Spectrum BX instrument. Measurement was conducted in the range of 650–4000 cm^{-1} wave number with a resolution of 2 cm^{-1}.

3.4 Thermogravimetric analysis (TGA)

To identify the thermal behavior of *Carex panicea* fiber, thermogravimetric analysis was utilized. Shimadzu DTG-60H instrument was utilized and analysis was recorded from room temperature to 800 °C under nitrogen atmosphere at a heating rate of 10 °C/min. Approximately 15 mg sample was implemented for measurement.

3.5 X-ray photoelectron spectroscopy (XPS) analysis

X-ray photoelectron spectroscopy (XPS) was conducted to determine the surface chemistry of *Carex panicea* fibers. Measurement was performed with Thermo Scientific instrument using Al-Ka X-ray source (1486.7 eV) between 1350 and 10 eV with a resolution of 1 eV. The surface of samples was cleaned with Ar gas prior to the analysis that was recorded with 20 scans.

3.6 X-ray diffraction analysis

X-ray diffraction method was performed to identify the crystallite index (CI) and crystallite size of *Carex panicea* fibers. A 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 during scan. XRD pattern was obtained between 5 and 65° range with 2°/min scan rate.

Dried fibers were powdered in order to obtain sufficient intensity as a pattern, and CI was determined by using an empirical formula suggested by Segal et al. (1959) (Eq. 2). Three replications of XRD pattern were obtained and mean values of intensities were calculated to produce a more reliable pattern.

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

where I_{200} is the maximum intensity of cellulose crystal, which is (200) lattice plane, and I_{am} is the intensity of amorphous peak, which is located between 18 and 19°. The intensity of amorphous peak is determined by taking the minimum intensity between highest two peaks represented in the cellulose pattern [55].

3.7 Tensile test

A universal testing machine (Instron 4411) was used for the tensile test of *Carex panicea* fibers with 1 kgf load cell. The loading rate was 1 mm/min with a gauge length of 20 mm. Pneumatic grips which were used for clamping the fiber have of 0.5 MPa pressure. Tensile tests were conducted following ASTM D 3822 standard. Ten fiber specimens were tested to check repeatability.

3.8 Morphology analysis

Detailed morphological characterizations were carried out by scanning electron microscope (SEM) images taken from longitudinal and cross-sectional parts of the *Carex panicea* fiber. Observations were performed using a JEOL-JJM 6060 model SEM device. The surfaces of the *Carex panicea* fibers were coated by Au–Pd alloy using sputter coating before characterization in order to avoid the electron beam charging effect.

4 Results and discussion

4.1 Fiber density

The density of *Carex panicea* fibers was found as 1.247 g/cm^3. As a candidate reinforcement, the low density of fibers triggers an increase in specific strength and results in lighter component production. When compared, the density of *Carex panicea* fibers is a little lighter than many common, and industrial natural fibers, such as kenaf (1.4 g/cm^3), flax

(1.50 g/cm^3), hemp (1.48 g/cm^3), jute (1.46 g/cm^3), and sisal (1.45 g/cm^3) [56] that can be advantageous. Fiber density plays a vital role in the design of lightweight components [3].

4.2 Chemical composition

Determination of fiber composition is significant due to its possible effects on chemical structure, physical properties, and mechanical performance of fiber and further reinforced composites. Cellulose contents of *Carex panicea* and other cellulosic natural fibers are listed in Table 1. The cellulose may be the most important component of the fiber which determines tensile strength and stiffness based on its high crystallinity. *Carex panicea* presents comparable cellulose content with recently characterized lignocellulosic fibers. Hemicellulose fraction (27.8%) is higher than that of many cellulosic fibers such as *Acaicia niotica L.* (14.14%) [1], *Cajanus cajan* (10.43%) [57], *Acacia concinna* (12.78%) [11], and *purple bauhinia* (9.17%) [27]. Hemicellulose can influence thermal resistance and water absorption of cellulosic fibers. The thermal resistance of fibers can be also affected by the lignin component. The fraction of lignin and the rest of the other constituents of *Carex panicea* was determined to be 6.5% which act as an adhesive to keep elementary cellulose cells together. However, lignin may deteriorate the interphase adhesion between components of polymeric composites [58].

4.3 FTIR analysis

To identify the main components of *Carex panicea* fibers (cellulose, hemicellulose, and lignin) and functional groups in fiber, FTIR spectroscopy was conducted. FTIR spectrum of *Carex panicea* fiber was recorded in the range of 4000–650 cm^{-1} and given in Fig. 2. The band located at 895 cm^{-1} is associated with the O-C-O stretching vibrations of cellulose [64–66]. The broad band at 1030 cm^{-1} indicates C-O and C-H stretching vibrations of fiber. The peak at 1160 cm^{-1} is related to the C–O antisymmetric bridge stretching of cellulose [36, 67]. The band at 1244 cm^{-1} can be assigned to C-O vibrations of acetyl groups in hemicellulose [68, 69]. The peaks at 1316 and 1371 cm^{-1} correspond to C-O and O–H bending vibrations of cellulose [67, 70, 71]. The bands located at 1422 and 1451 cm^{-1} correspond to CH_2 bending vibrations and C-H deformation in groups of methyl, methoxyl, and methyl in lignin, respectively [54, 72, 73]. The band observed at 1506 cm^{-1} is associated with the C = C ring stretching of aromatic lignin in fiber [25, 74]. The absorption band at 1633 cm^{-1} corresponds to absorbed water in fiber [75, 76]. The band at 1730 cm^{-1} is related to the carbonyl groups of lignin [31, 77, 78]. The broad band at 3338 is related to the –OH, and 2920 cm^{-1} and 2853 cm^{-1} are related to the C-H stretching vibrations in cellulose and hemicellulose, respectively [79, 80]. FTIR results

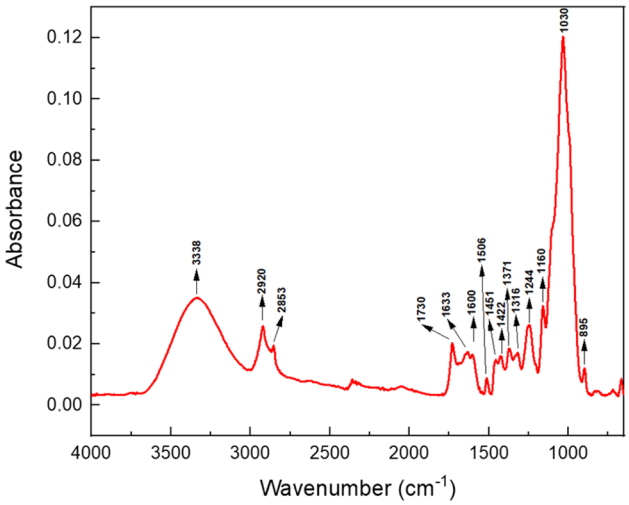

Fig. 2 FTIR spectra of *Carex panicea* fiber

Table 1 Cellulose content of *Carex panicea* and other some novel fibers

Fiber	Cellulose (wt%)	Hemicellulose (wt%)	Density (wt%)	References
Carex panicea	65.70	27.8	1.25	In current study
Grewia damine	57.78	14.96	1.38	[35]
Albizia amara	64.54	14.32	1.04	[59]
Cardiospermum halicababum	59.82	16.75	1.14	[10]
Heteropogon contortus	64.87	19.34	0.60	[60]
Conium maculatum	49.50	32.2	-	[25]
Aristida adscensionis	70.78	10.5	0.79	[61]
Cereus hildmannianus	58.40	17.14	1.364	[62]
Piliostigma racemosa	60.30	0.27	1.371	[63]
Cortaderia selloana grass	53.70	14.43	1.261	[2]

of *Carex panicea* agree with the most common natural fibers such as hemp [20], jute [81], and kenaf [19].

4.4 Thermogravimetric analysis

Thermogravimetry is a helpful characterization technique for understanding of the thermal behavior of cellulosic fibers. Because the production of polymer-based composites includes relatively high processing temperatures, the determination of the onset temperature of fibers is significant to benefit from fiber properties at a maximum rate [36, 82–85]. TG/DTG curves of *Carex panicea* fiber are shown in Fig. 3. As presented in Fig. 3, the first mass loss was observed between 25 and 100 °C with 5.36% due to the dehydration of water in fibers [86, 87]. The second mass loss occurred between 220 and 310 °C with 13.84% can be related to the degradation of hemicellulose in fibers [88]. The third and the major weight losses were recorded between 300 and 370 °C which indicates decomposition of cellulose in fiber with 36.53% [89, 90]. The temperatures of 219.4 °C and 351.59 °C were determined as onset decomposition temperature (T_{onset}) and maximum degradation temperature (T_{max}), respectively. Comparison of T_{onset} and T_{max} temperature of most utilized and some recent natural fibers were listed in Table 2. Compared to most utilized natural fibers such as kenaf (219 °C) and jute (205.1 °C), wood pine (234 °C), bagasse (222.3 °C), and cotton stalk (221.6 °C), *Carex panicea* is a proper reinforcement for polymeric composites due to high decomposition onset temperature [91]. The analysis was recorded up to 800 °C and after major decomposition, 28.905% mass loss was observed related to the residual content of fibers [92]. This weight loss may correspond to oxidative degradation of residues and lignin in the fiber [93].

Table 2 Comparison of thermal properties of *Carex panicea* fiber with other natural fibers

Plant name	T_{onset} (°C)	T_{max} (°C)	Reference
Carex panicea	219.4	351.59	Current study
Kenaf	219	364	[94]
Jute	205	365	[94]
Wood pine	234	328	[91]
Cotton stalk	221.6	345.2	[95]
Citrullus lanatus	-	325	[96]
Leucas aspera	-	325	[97]
Shwetark stem	225	350	[98]

4.5 XRD analysis

XRD pattern of *Carex panicea* fibers shows two main peaks (see Fig. 4.), first a broad peak at 15.96°, and a second peak at 22.21°. The first peak is formed by overlapping of two cellulose peaks that belongs to (110) and ($1\bar{1}0$) lattice planes, and the second peak is formed by cellulose (200) lattice (French 2014; Kılınç et al. 2018).

The crystallinity index (CI) and crystallite size (L) of *Carex panicea* fibers were found as 56.42% and 7.6 nm, respectively. CI of *Carex panicea* fibers generally lower than many plant fibers (Table 3). The low crystallinity of fibers can be also a sign of a low-strength material. The decrease in the strength of fiber can be explained with the absence of crystalline microfibrils cellulosic and chains which cannot align regularly to form an ordered structure [99]. On the other hand, the crystallite size of *Carex panicea* fibers shows similarities with other natural fibers [36], which is a measure of coherency at the related XRD plane (200).

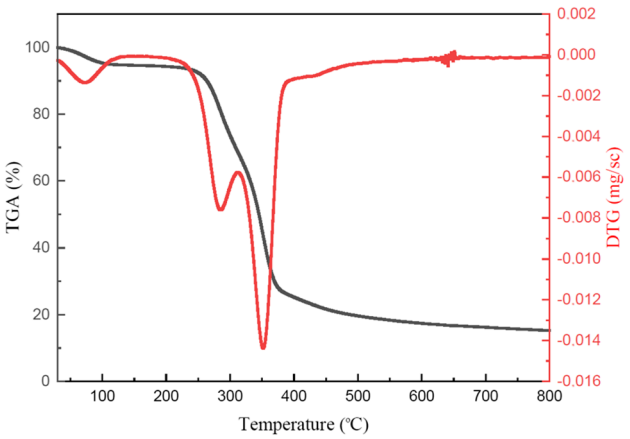

Fig. 3 TGA/DTG curves of the *Carex panicea* fiber

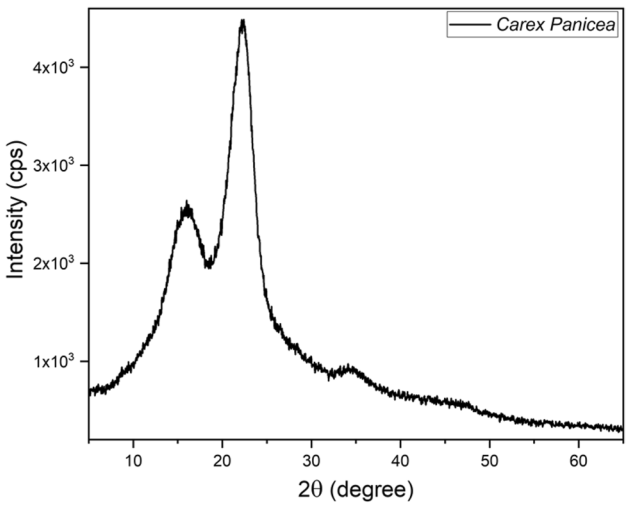

Fig. 4 X-ray diffraction pattern of *Carex panicea* fiber

Table 3 The crystallinity indexes of some common natural fibers with *Carex panicea* fibers

Plant name	Crystallinity index	Reference
Carex panicea	56.42	Present study
Coir	49.60	[100]
Kenaf	62.90	[101]
Sisal	66.95	[100]
Jute	67.69	[100]
Bamboo	78.92	[102]
Hemp	64.87	[103]
Flax	79.13	[100]
Cotton	58.00	[25]

Table 4 O/C ratio of *Carex panicea* and some natural cellulosic fibers

Fiber	O/C	Reference
Carex panicea	0.17	Present study
Conium maculatum	0.22	[25]
Althea officinalis L	0.26	[82]
Ferrula communis	0.27	[106]
Flax	0.28	[100]
Coir	0.29	[107]
Kenaf	0.37	[108]
Chrysanthemum morifolium	0.41	[36]
Hierochloe odarata	0.48	[73]
Centaurea solstitialis	0.54	[54]
Luffa cylindrica	0.61	[109]

4.6 XPS analysis

Atomic concentrations for carbon and oxygen on the surface of *Carex panicea* were obtained to be 81.86 and 14.10%, respectively. The oxygen/carbon ratio of *Carex panicea* was determined as 0.17 which is in comparison with some lignocellulosic fibers (Table 4). It is clear that *Carex panicea* has a relatively hydrophobic surface character presenting dramatically lower O/C as compared with most of commonly used and recently characterized cellulosic fibers as tabulated in Table 4. This finding may indicate its potential as a reinforcement material in highly non-polar polymers considering possible surface compatibility. To determine the functional groups of *Carex panicea*, deconvolution of C1s and O1s peaks was conducted. C1s and O1s spectra of *Carex panicea* are depicted in Fig. 5a and b. The peaks located at 286.5 eV and 532.7 eV can be associated with C–O–C bonds [104]. The ratio of C–C/C-H (284.7 eV) and C–O–C (286.5 eV) groups was calculated as 69.44 and 30.56%, respectively. The major peak at 284.7 in C1s spectra representing C–C/C-H and also C–O–C bonds can affirm the presence of cellulose in fiber [104, 105].

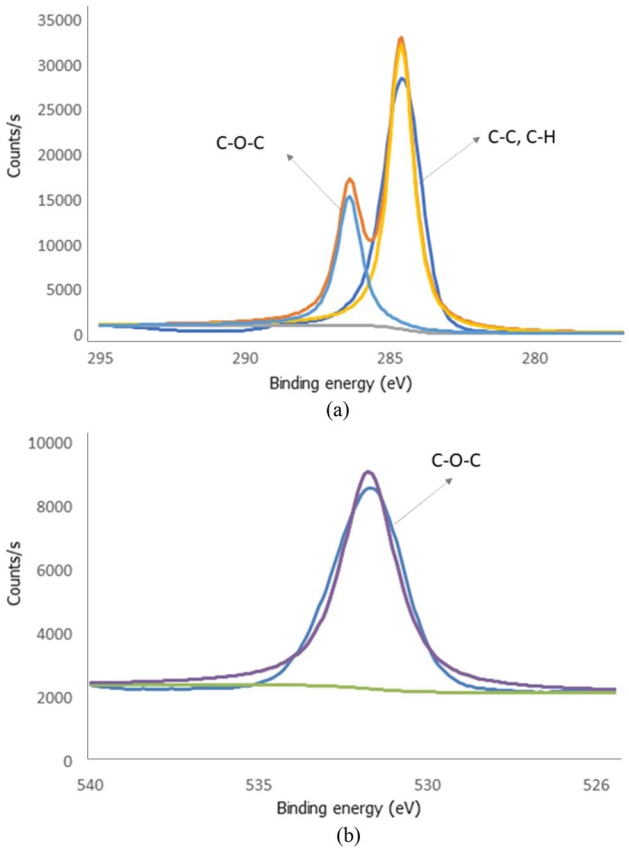

Fig. 5 The high-resolution XPS spectra of **a** C1s and **b** O1s peaks belong to the *Carex panicea* fiber

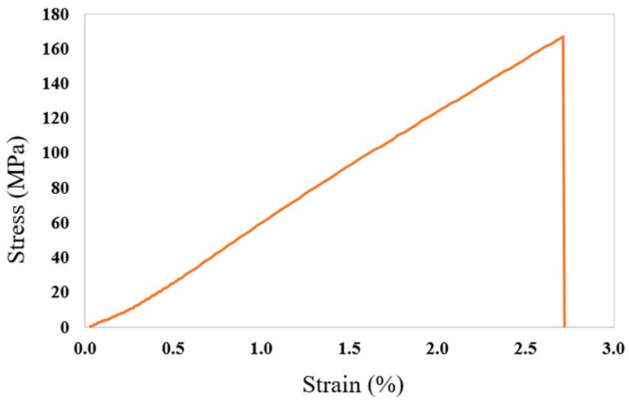

Fig. 6 Stress–strain curve of *Carex panicea* fiber

4.7 Tensile properties

Stress–strain behavior of *Carex panicea* fibers is presented in Fig. 6. Tensile strength, Young's modulus, and elongation at break data are listed and compared with the other natural fibers in Table 5. The standard deviation is quite large which is common for natural cellulosic fibers. As seen in the graph,

Table 5 Mechanical properties of *Carex panicea* fiber and some natural cellulosic fibers

Fiber name	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)	References
Carex panicea	143 ± 41	5.5 ± 1.86	2.738 ± 0.71	Current study
Juncus effuses L	113 ± 36	4.38 ± 1.37	2.75 ± 0.6	[110]
Phoenix dactylifera L	117 ± 35	4.3 ± 1.4	3.13 ± 0.70	[112]
Chrysanthemum morifolium	65.12 ± 25.04	1.55 ± 0.76	4.51 ± 0.95	[36]
Luffa cylindrica	385 ± 10.52	12.2 ± 1.02	2.65 ± 0.05	[109]
Jute	249 ± 89	43.9 ± 12.3	00.6 ± 0.2	[111]
Piassava	131 ± 36	3.8 ± 00.9	11.4 ± 3.6	[111]
Grewia damine	375.6 ± 16.58	126.2 ± 11.93	2.99 ± 0.273	[35]
Albizia amara	640 ± 13.4	-	1.57 ± 0.04	[59]
Heteropogon contortus	476 ± 11.6	48 ± 2.8	1.63 ± 0.06	[60]
Cereus hildmannianus	2897.47 ± 23	2.98 ± 0.2	1.24 ± 0.8	[62]
Piliostigma racemosa	32	2	1.2–3.2	[63]
Coccinia indica	75	-	4.25	[43]
Manau rattan	273 ± 52.88	7.80 ± 1.70	9.40 ± 3.67	[12]

fibers exhibit linear characteristic like other cellulosic natural fibers. On the other hand, a sudden load drop indicates the brittle nature of the fibers. It is clear that *Carex panicea* fiber has higher tensile strength than the *Juncus effuses L.* [110], *Piassava* [111], *Chrysanthemum morifolium* [36], and *Phoenix dactylifera L.* [112]. Young's modulus of fiber (5.5 ± 1.86 GPa) is comparable with other natural fibers such as *Juncus effuses L.* [110], *Phoenix dactylifera L.* [112], and *Piassava* [111]. Due to its linear orientation and high degree of polymerization, cellulose can govern the tensile properties of the fibers (Kathirselvam et al., 2019).

4.8 Morphological characterizations

General and detailed scanning electron microscope images of the *Carex panicea* fiber are presented in Fig. 7 in order

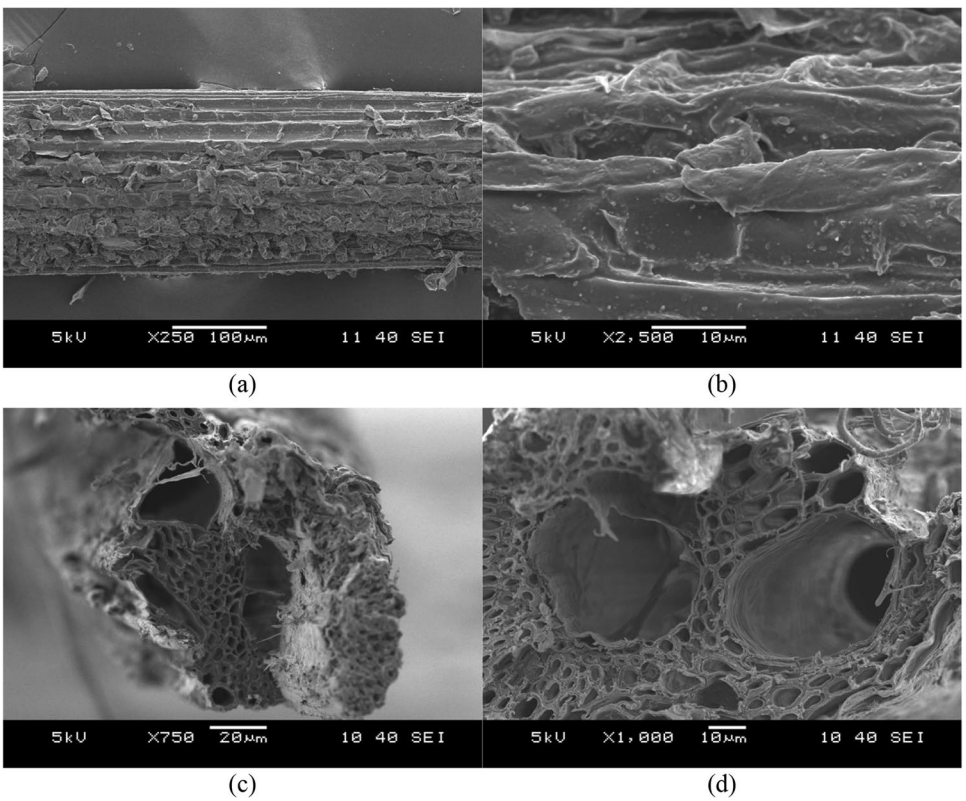

Fig. 7 SEM images of the *Carex panicea* fiber taken from **a** and **b** longitudinal and **c** and **d** cross-sectional surfaces, respectively

to interpret fiber morphology. Both longitudinal and cross-sectional SEM images are presented due to the fact that they provide different information. The fiber sample has about 200-µm diameter according to Fig. 7a. In addition, some impurity residues take attention on *Carex panicea* fiber surface (see Fig. 7a and b). It can be seen that these impurities on the surface cause the formation of an indented surface morphology (Fig. 7b). Thus, *Carex panicea* fiber endorses a high surface area for locking polymer matrix as an additive for composites.

As can be observed from cross-sectional images in Fig. 7c and d, *Carex panicea* fiber consists of many elementary fibers as other natural fibers [25]. The empty nutrition/water channels at the center of elementary fibers are called as lumen [113]. Lumen diameters of the elementary fibers of *Carex panicea* appear to be very variable according to the Fig. 7c and d. It can be concluded from SEM images that *Carex panicea* fibers have about 1–2 µm lumen wall thickness, low lumens about 5 µm lumen diameter while large lumens have a diameter of 50 µm. Combination of the large and small diameters of lumen causes low density, high insulation, and absorbance properties. Among these properties, *Carex panicea* fiber is striking for its very thin wall thickness. For example, *Carex panicea* fiber has lower wall thickness than curaua, jute, sisal, and *Chrysanthemum morifolium* fibers [36, 111].

5 Conclusion

In this study, *Carex panicea* fibers were characterized as new potential natural fiber reinforcement for polymeric composites. The obtained physicochemical characterization results of *Carex panicea* stem fibers have low density, comparable cellulose content and crystallinity index, better surface hydrophobicity, better thermal stability, relatively high tensile properties, and similar surface morphology. According to the results:

- The density of fibers are calculated as 1.247 g/cm^3 which is lower than the most utilized synthetic fibers and this indicate lighter composite production.
- Thermal analysis shows that fibers thermally stable up to 219.4 °C without any degradation. Considering relatively high manufacturing temperature of polymer composites, thermal analysis indicates suitability of *Carex panicea* fibers for polymer composite production.
- XPS results show that with very low O/C ratio (0.17), *Carex panicea* fibers have hydrophobic surface characteristics. Tensile strength and Young's modulus of fibers were determined as 143 ± 41 MPa and 5.5 ± 1.86 GPa, respectively.
- SEM images show that *Carex panicea* fibers consist of many elementary fibers bonded together. According to these results, *Carex panicea* fibers can be a potential natural fiber as an alternative to synthetic fibers, which can be utilized in polymer composites.

Author contribution Ozgur Yasin Keskin: conceptualization, methodology, validation, investigation, writing — original draft, writing — review and editing, and visualization; Serhan Koktas: investigation and writing — review and editing; Yasemin Seki: conceptualization, methodology, validation, investigation, writing — original draft, and writing — review and editing; Ramazan Dalmis: investigation and writing — review and editing; Gonca Balci Kilic: investigation and writing — review and editing; Didem Albayrak: investigation and writing — review and editing.

Data availability Not applicable.

Declarations

Ethical approval Not applicable.

Competing interests The authors declare no competing interests.

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