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POSİDONİA OCEANİCA ATIKLARINDAN ELDE EDİLEN DOĞAL SELÜLOZİK ELYAF ÜZERİNE ÇALIŞMA: KARAKTERİZASYON VE ANALİZ

STUDY ON NATURAL CELLULOSIC FIBER FROM POSIDONIA OCEANICA WASTE: CHARACTERIZATION AND ANALYSIS

Yasemin SEKİ[1]
Havva Nur ÖZDEMİR[2]*
Gizem Ceylan TÜRKOĞLU[1]
Barış AKÇALI[3]
Gökdeniz NEŞER[3]

[1]Dokuz Eylul University, Department of Textile Engineering, Tınaztepe Campus, Izmir 35390, Turkey
[2]Dokuz Eylul University, Department of Chemistry, Tınaztepe Campus, Izmir 35390, Turkey
[3]Dokuz Eylul University, Institute of Marine Sciences and Technology, Inciraltı, Izmir 35330, Turkey

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STUDY ON NATURAL CELLULOSIC FIBER FROM *POSIDONIA OCEANICA* WASTE: CHARACTERIZATION AND ANALYSIS

Yasemin SEKİ[1]
Havva Nur ÖZDEMİR[2]*
Gizem Ceylan TÜRKOĞLU[1]
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[1]Dokuz Eylul University, Department of Textile Engineering, Tınaztepe Campus, Izmir 35390, Turkey
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[3]Dokuz Eylul University, Institute of Marine Sciences and Technology, Inciraltı, Izmir 35330, Turkey

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ABSTRACT: This current research reports the characterization of fibers from *Posidonia oceanica L. (P. oceanica)* for the interest in usability as additive biomaterial in polymer-based materials. The results revealed that cellulose content, density, and average fiber diameter of the fibers from *P. oceanica* were 45.35%, 1.15 g/cm^3 and 238.41 µm, respectively. The maximum degradation temperature, char yield and activations energy were found to be 318.8 ˚C, 31.82% 600 ºC and 49.36 kJ/mol, respectively. Considering the output of this current research, the fibers from *P. oceanica* can be employed as reinforcement or additive for polymeric based materials for potential applications.

Keywords*: Posidonia oceanica (L.),* cellulose, fiber, surface morphology, characterization

POSİDONİA OCEANİCA ATIKLARINDAN ELDE EDİLEN DOĞAL SELÜLOZİK ELYAF ÜZERİNE ÇALIŞMA: KARAKTERİZASYON VE ANALİZ

ÖZ: Bu araştırma, *Posidonia oceanica L.* (*P. oceanica*) liflerinin polimer bazlı malzemelerde katkı biyomalzemesi olarak kullanılabilirliğine yönelik karakterizasyonunu rapor etmektedir. Sonuçlar, *P. oceanica* liflerinin selüloz içeriği, yoğunluğu ve ortalama lif çapının sırasıyla %45,35, 1,15 g/cm^3 ve 238,41 µm olduğunu ortaya koymuştur. Maksimum bozunma sıcaklığı, kömür verimi ve aktivasyon enerjisi sırasıyla 318.8 ºC, %31.82 600 ºC ve 49.36 kJ/mol olarak belirlenmiştir. Bu mevcut araştırmanın çıktıları göz önüne alındığında, *P. oceanica*'dan elde edilen lifler, potansiyel uygulamalar için polimerik esaslı malzemeler için takviye veya katkı maddesi olarak kullanılabilir.

Anahtar Kelimeler: *Posidonia oceanica* (*L.*), selüloz, lif, yüzey morfolojisi, karakterizasyon

Sorumlu Yazarlar/Corresponding Author: *havanurozdemir818@gmail.com*
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1. INTRODUCTION

P. oceanica (Linnaeus) Delile, which is a Mediterranean endemic seagrass, starts in shallow waters on the Mediterranean coasts [1]. It has great economical and ecological importance for the marine environment, as it prevents erosion in the sea with its roots, creates an oxygen source in the marine environment with photosynthesis. It creates a suitable habitat for fish and other species for shelter, feed, and reproduction, and also forms the first link of the food chain [2]. The economic benefits of seagrass meadows to the ecosystem have been determined and the contribution of these species is 15837 Euro ha^{-1} $year^{-1}$, which is more than the agricultural areas provide [3]. *P. oceanica*, which has an equivalent function to the forests on land with these listed ecological and biological features, constitutes the source of life in the sea [4]. As in many parts of the Mediterranean, the marine ecosystem on Turkish coasts is affected by domestic and industrial wastes, marine transportation, intensive coastal use, excessive hunting, and aquaculture activities by bottom dredging methods. Due to these activities, the distribution areas of marine plants, especially *P. oceanica*, are limited and their sexual reproduction is ceased [5]. *P. oceanica*, can be found in salty and clean waters between 11 ºC – 29 ºC in the Mediterranean. It may create meadows from the surface to a depth of more than 40 meters. *P. oceanica* leaves form banks on the shore with currents and waves when they completed their life. These structures protect the coastline, prevent erosion, and enable many sea creatures to survive. In recent years, many investigations were performed on the usability of fibers from *P. oceanica* as an adsorbent for dyes or as a source of lignocellulosic fibers for the production of pulp and paper or reinforcement for polymeric composite materials [1].

The manufacturing of polymeric composite with good mechanical performance and desired properties requires awareness and a search for novel cellulosic natural fibers. In related literature, there are many papers focused on the characterization and extraction of natural fibers like *Hierochloe Odarata [6], Chrysanthemum morifolium [7], Centaurea Solstitialis [8], Piliostigma Racemosa [9], Opuntia ficus [10], Zmioculus Zamiifolia* [11]. To meet the demands for biodegradable, abundant, and ecologically friendly fibers, fibers from *P. oceanica* were characterized as a potential cellulose resource for many applications. In the literature, very limited research has been done on the usability in composite production and absorption performance of *P. oceanica*. In the current study, physical, chemical, morphological and crystallographic structure of *P. oceanica* fiber were analyzed by using x-ray diffraction (XRD), fourier transform-infrared spectroscopy (FT-IR), thermogravimetric analysis (TGA), chemical analysis, and density measurement, scanning electron microscope images (SEM).

2. MATERIAL AND METHOD

2.1 Material

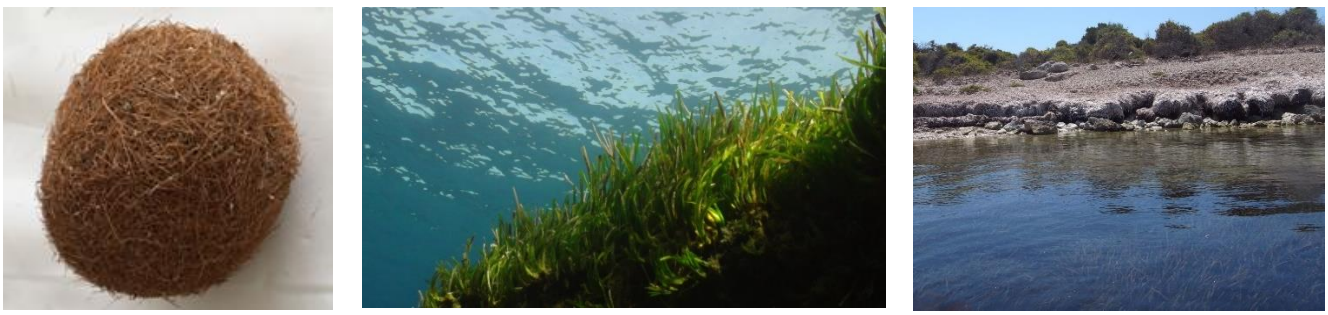
The fibers from *P. oceanica* are collected from Ayvalık beaches where summer activities are very intensive. The chemical agents such as sodium hydroxide, sulphuric acid, ammonia, acetic acid, sodium chlorite, and EDTA were purchased from Sigma-Aldrich for the chemical composition analyses. Figure 1 presents the image of *P. oceanica* fibers.

2.2 Method

2.2.1. Determination of fiber density

The density (d) measurement of the fibers from *P. oceanica* was used by Archimedes' Law (according to ASTM D8171-18 Method B). For accurate outcomes, the density measurements were performed in 3 replicates. Each sample was oven-dried at 105 °C for 4 h and then weighed. Afterward, the samples were immersed in boiling water for 24 h of water absorption. Finally, the submerged weight of the samples was taken after 24 h of boiling in deionized water. The density measurement of the fibers was calculated using the formula given in Eq.1,

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



(a) **(b)** **(c)**

Figure 1. (a) The fibers from *P. oceanica* ball, **(b)** meadows of *P. oceanica* under the sea and **(c)** the coastline where *P. oceanica collected*

Here, d indicates the density, W_d and W_s are the dry weight and the actual mass of fibers submerged in the deionized water, respectively [6, 12].

2.2.2. Determination of fiber chemical composition

The fibers from *P. oceanica* were dried at 105°C in an oven and then fibers were kept in a desiccator prior to analysis to avoid humidity. All experiments were carried out in triplicate.

The brief procedure of the processes is as follows. For the determination of cellulose, one gram of fiber was put in 1.72% $NaClO_2$ in acidic medium adjusted with few drops of H_2SO_4 (pH 4). After an hour of refluxing at boiling temperature, the solution was filtered to eliminate the solution. The residue was washed with concentrated ammonia solution and rinsed with distilled water. After drying at 105 °C, the residue's final weight was determined as cellulose content [6,13].

For the determination of hemicellulose, one gram of fiber was put in 10% NaOH for 1.5h at room temperature and followed by 5% HCl treatment at room temperature using mechanical stirring. The solution was filtered and the resultant sample was treated in a laboratory oven at 105 °C for 1 h. The amount of hemicellulose was reported by the difference between the initial and final weights [6,13].

For the determination of lignin, one gram of fiber was immersed in 12.5 mL of 72% sulfuric acid for 1 h at room temperature. Afterward, 300 mL of distilled water was added, and the mixture was left without agitation for 2 h. The solution is filtered and the remaining is dried at 105 °C and the weight is noted as lignin content [6,13].

For the determination of pectin, the fibers were put in 0.5% w/v EDTA solution for 30 min using a magnetic stirrer at boiling temperature. After the process was completed, the fibers were rinsed with deionized water and dried in an oven at 105 °C for 4 h. The amount of hemicellulose was determined by the difference between the initial and final weights. [6,13].

2.2.3. FT-IR analysis

FT-IR spectrum of *P. oceanica* fibers was obtained using a Perkin Elmer FT-IR spectrometer (Spectrum BX). The measurements were recorded in the range of 4000 – 400 cm^{-1}. The spectrum of the sample was obtained from 20 scans and the spectral resolution was 2 cm^{-1}.

2.2.4. XRD analysis

XRD pattern of *P. oceanica* fibers was obtained from Rigaku Ultima 3 device. A copper X-ray tube was used as the radiation source (λ-Cu-Kα1 = 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. The crystallinity index (CI) was calculated by using the empirical formula as given in Equation 2 [8].

$$\text{Crystalline index (CI) (\%)} = \frac{(I_{200} - I_{am})}{I_{200}} \times 100 \qquad (2)$$

where I_{200} represents the peak with the highest intensity that relates to the lattice plane (200) at 22.32°, and I_{am} represents the minimum intensity near 18.1° [14].

Scherrer's equation was applied to the XRD pattern after baseline correction by employing OriginPro software to estimate crystalline features (Eq.3).

$$D = \frac{K \times \lambda}{\beta \times \cos\theta} \qquad (3)$$

2.2.5. TGA analysis

TGA was conducted by using a Shimadzu DTG-60H instrument. The investigation was performed in the range of room temperature to 600 ºC at a rate of 10 °C/min. To prevent oxidation, the analysis was conducted under a nitrogen atmosphere.

2.2.6. SEM observation

The morphological structure of *P. oceanica* fibers was examined by FESEM FEI Nova Nanosem 650 model scanning electron microscope (SEM). SEM images were taken with an accelerating voltage of 5 kV. Prior to the examination, *P. oceanica* fibers were coated with Au–Pd alloy by sputter coating. The average fiber and fibril diameters were calculated from 15 random measurements using ImageJ software on SEM micrographs.

3. RESULTS AND DISCUSSION

3.1 Fiber density

According to the result obtained from the Archimedes principle, the density of the fibers from *P. oceanica* was calculated as 1.15 g/cm^3. The density values of the fibers from *P. oceanica* and some other plant fibers are given in Table 1. The density of this fiber is slightly lower than these fibers and this property can provide its usability in lightweight applications.

Table 1. The density values of some natural fibers

Fiber name	Fiber density (g/cm^3)	References
P. oceanica	1.14	Current study
Jute	1.30	[15]
Kenaf	1.45	[16]
Hemp	1.47	[15]
Flax	1.50	[17]

3.2. Chemical composition

The chemical composition is highly effective in thermal decomposition, crystalline structure, mechanical performance, and some other physical properties [18]. The hemicellulose, pectin, cellulose, and lignin contents are determined as 8.02,15.39, 44.56 and 32.03%, respectively. In accordance with related literature, percentage of cellulose is less but lignin is quite high as compared with similar plant fibers such as *Centaurea solstitialis* [8], *Piliostigma Racemosa* [9], *Corypha taliera* fruit [19] and cellulose fraction of the fibers from *P. oceanica* is comparable

with *Pennisetum purpureum* (47.1%), *Phoenix Dactylifera L.* (43.0%) and *Conium Maculatum* (49.5%) fibers [20-23].

3.3. FT-IR analysis

Figure 2 presents the FT-IR spectrum of the fibers of *P. oceanica*. FT-IR spectra of the lignocellulosic fibers exhibits chemical functional groups of cellulose, hemicellulose, and lignin. The first broad absorption peak located at 3422 cm^{-1} indicates the OH stretching. The next peak at 2924 cm^{-1} presents the asymmetric and symmetric stretching vibrations of acetyl groups in C–H. The peak at 1635 cm^{-1} can be related to OH bond vibrations including intermolecular hydrogen bonds between polysaccharide chains [24]. The peaks at 1428 cm^{-1} and 1513 cm^{-1} indicate the presence of C=C stretching of aromatic lignin and C–H symmetric bending of cellulose, respectively [6]. The peaks around at 1400 cm^{-1} and 1250 cm^{-1} can be associated with the presence of lignin and hemicellulose through the stretching of CO of the acetyl groups. The peak at 1113 cm^{-1} can be attributed to the C-H in-plane deformation of the syringyl unit of lignin [25]. The strongest peak at 1035 cm^{-1} appeared due to C–O and O–H stretching in the fiber [7].

3.4. XRD analysis

XRD analysis was conducted to learn information about the crystalline structure of *P. oceanica* (Figure 3). The typical peaks of *P. oceanica* are known to be 2θ = 3°–30° which can be attributed to the major components of the fiber such as cellulose, hemicelluloses, and lignin [26, 27]. The structure of *P. oceanica* presented three cellulose Iβ crystalline allomorphs diffraction peaks at 15.4°, 22.0°, and 34.8°, with ascribed planes of 1-1 0, 2 0 0, and 004, respectively [28]. *P. oceanica* fiber also presented a peak located at 2θ = 29.1°, which is detected possibly due to the existence of lignin [29]. In addition, there is a small peak recorded at 34.8°, which might correspond to the presence of sodium metal [30]. XRD pattern of *P. oceanica* showed an amorphous nature of the biomass [31]. According to Scherrer's equation, full width at half maximum (FWHM) and crystallite size (L) were calculated as, 3.50 and 22.86 respectively, where k is 0.89.

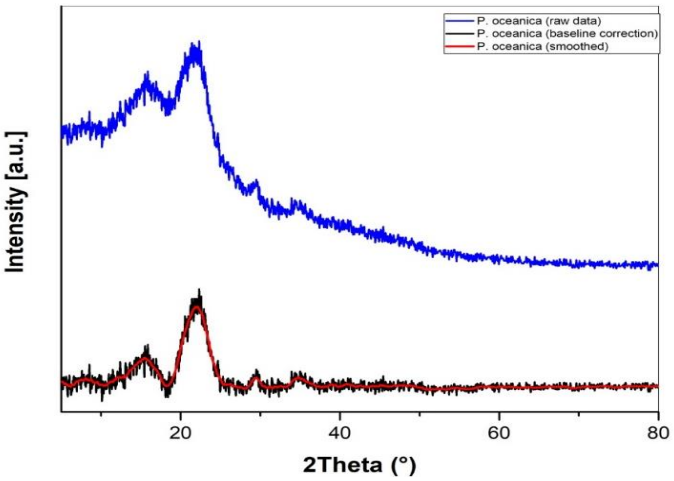


Figure 3. XRD spectra of *P. oceanica*

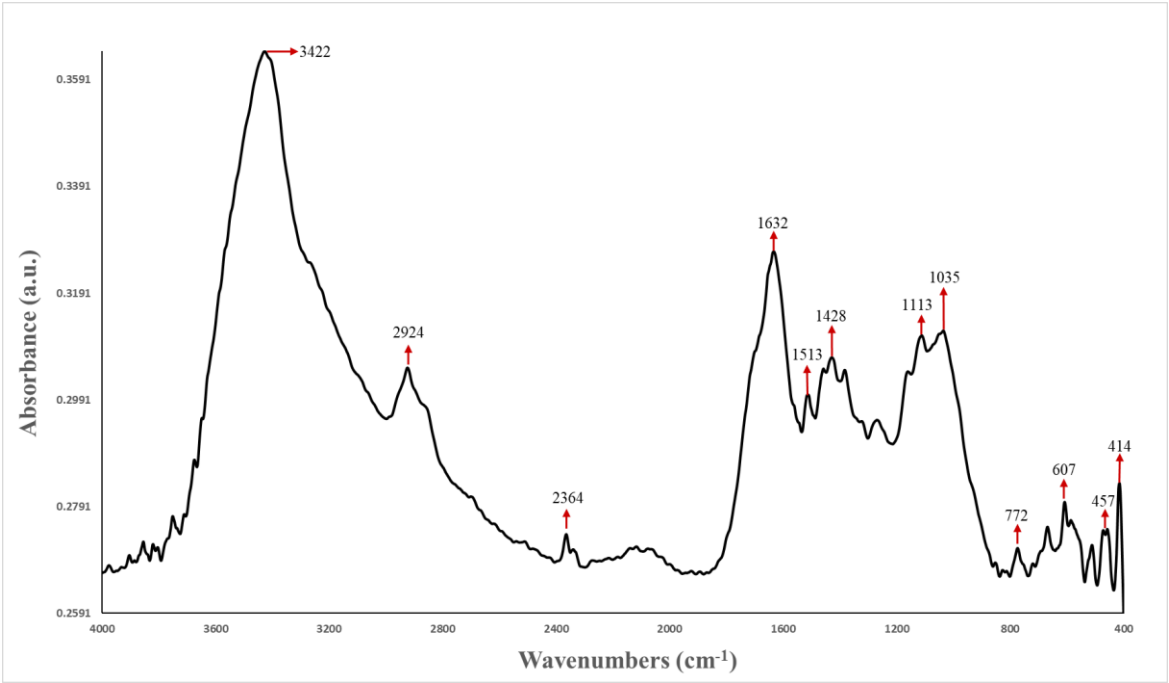


Figure 2. FTIR spectra of *P. Oceanica*

3.5. TGA analysis

The thermal decomposition of the fibers from *P. oceanica* was examined by TGA-DTG. The data and the graphs are presented in Table 2 and Figure 4, respectively. The first mass loss till 200 ºC which is recorded as about 11% can be attributed to the evaporation of water present in the fibers [6]. The onset degradation temperature is detected as 173.2 ºC with 11.3% mass loss which can be also correspond to the decomposition of hemicelluloses in the fiber [8]. The maximum degradation temperature was stated as 318.8 ºC with about 40% mass loss that can be attributed to the degradation of α-cellulose and lignin. The researchers found similar decomposition temperatures for *P. oceanica* as compared with *Azadirachta indica* (321.2 ºC), *Chloris barbata* (324.6 ºC), *Cannabis sativa* (Hemp) (329 ºC), and *Citrullus Lanatus* (325 ºC) fibers [32-35]. 50% mass loss occurred at 350.37 ºC. The char yield value, which is the remaining residue after completing TGA analysis at 600 ºC, is 31.82% for the fiber from *P. oceanica*.

Table 2. Thermal decomposition data of *P. oceanica*

Parameter	Value
$T_{1\%\ mass\ loss}$ (ºC)	34.97
$T_{5\%\ mass\ loss}$ (ºC)	58.69
$T_{50\%\ mass\ loss}$ (ºC)	350.37
T_{onset} (ºC)	173.20
T_{max} (ºC)	318.83
Mass loss at T_{max} (%)	39.77
Char yield (%)	31.82

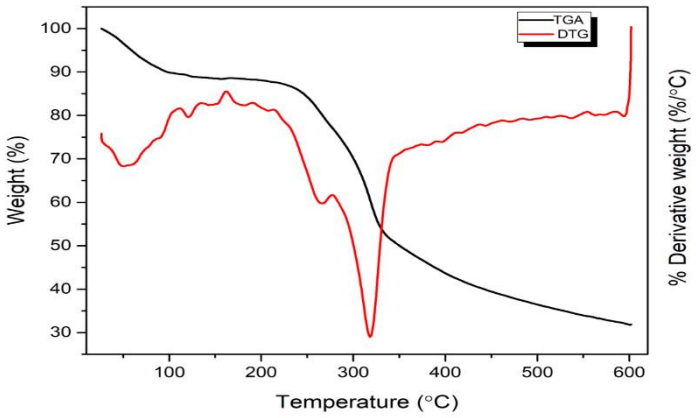


Figure 4. TGA-DTG graph of *P. oceanica*

The kinetic activation energy (E_a) of the fiber from *P. oceanica* was determined by using Broido's plot (Figure 5) as 49.36 kJ/mol. This value is slightly lower than recently characterized cellulose fibers such as *Atriplex Halimus* (56-72 kJ/mol) [36], *Trachelospermum jasminoides* (64.16 kJ/mol) [37], and *Coccinia grandis* (67.02 kJ/mol) [38].

3.6. SEM observation

Figure 6 displays SEM images of the fibers from *P. oceanica* taken at ×250 and ×500 magnifications. Photomicrographs revealed that the fibers resemble an irregular shaped cylinder and are assembled into cellulose fibrils. The average diameter of the fiber was 238.41 µm with a standard deviation of 6.80 µm. The diameter of the fibrils varied between 20.51 µm and 54.01 µm. The mean diameter of the fibrils was calculated as 36.56 µm with a standard deviation of 9.40 µm. *P. oceanica* consists of many several elementary fibers bound together by pectin or other non-cellulosic constituents like the other natural cellulosic fibers [39]. It was observed that there were some impurities which might be saline and sand particles [40]. The fiber presents rough surface because of the impurities and cementing materials like lignin and waxy materials. The surface roughness can be beneficial when utilized as reinforcement material in composite manufacturing by enhancing mechanical interlocking between fiber and polymer in a composite system [7].

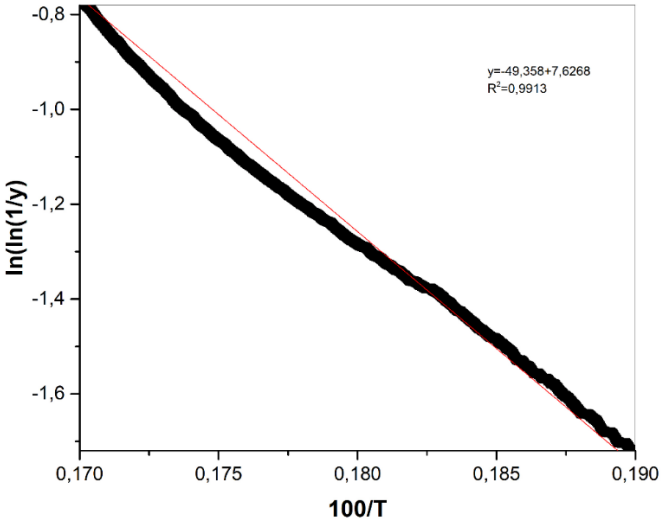
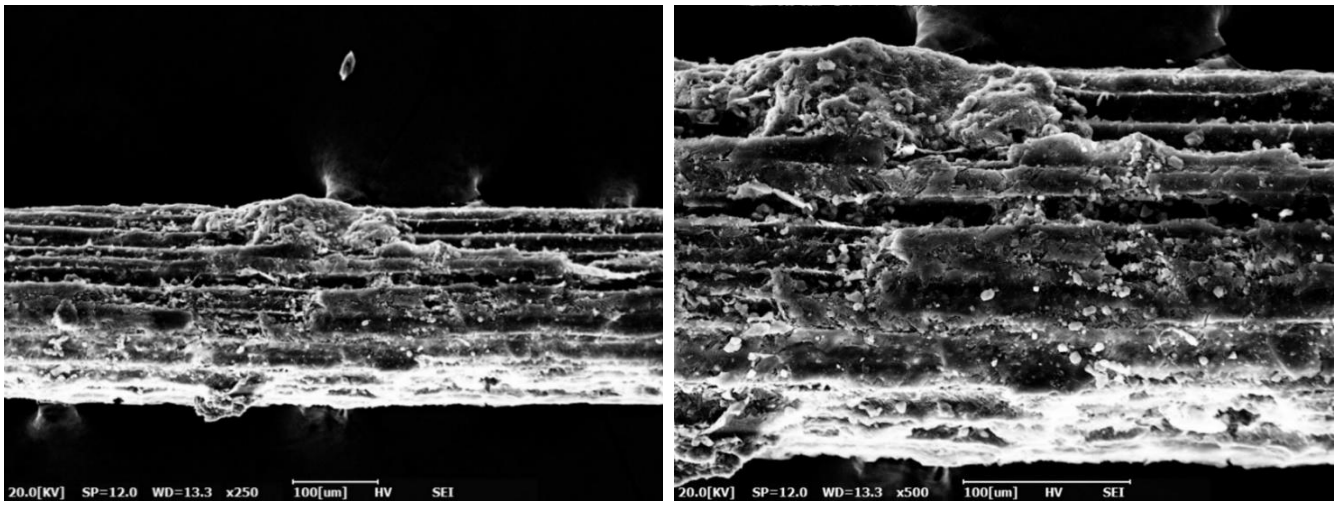


Figure 5. Broido's plot of *P. Oceanica*

4. CONCLUSION

The chemical, physical, thermal, structural, and morphological properties of the fibers from *P. oceanica* were characterized in this current study. The morphological studies indicated that the fibers from *P. oceanica* consisted of several elementary fibers bonded together with non-cellulosic components. There were also impurities on the surface of the fibers which can lead to surface roughness. The average diameter of the *P. oceanica* was estimated to be 238 ± 6.80 µm, and the density of *P. oceanica* was 1.15 g/cm^3. The cellulose and hemicellulose contents were determimed as 45.35% and 8.70%, respectively. The *P. oceanica* had a maximal degradation which can indicate that these fibers could be used for industrial applications in which injection and extrusion processes take place. In conclusion, the fibers from *P. oceanica* was found as an appropriate material for reinforcement in the fabrication of polymer matrix-based composites.



(a) (b)

Figure 6. SEM micrographs of *P. oceanica*

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