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Helianthus Annuus L. Comparison of The Properties of Fibers Obtained From The Plant By Methods of Decortication and Retting

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Abstract

A large amount of sunflower production is carried out in our country. The 2,500,000 tons of sunflower stalks that appeared after production pose a problem for our farmer. In order to clean up this environmental problem from the field, sunflower stalks are destroyed by burning to warm up in winter, broken down and mixed into the soil, or burned after harvesting. It is thought that by obtaining qualified, ecological and naturally decomposing sunflower fiber from the stem of the sunflower plant, which is an agricultural waste, it can increase the added value of agricultural products and contribute to the protection of the environment. In this study, the anatomical characteristics of the stem of the sunflower plant were determined, and natural lignocellulosic fibers were obtained from the sunflower stem by retting and decortication methods (fresh stem, dried stem). Various physical, chemical and mechanical properties of these fibers have been measured. For this purpose, FTIR (Fourier Transform Infrared Spectroscopy) analysis, XRD (X-Ray Diffractometer) analysis and SEM (Scanning Electron Microscope) analysis were applied to the fibers obtained by different methods. Thermal analyses were performed by TG-DTA (Thermogravimetric) analysis. In addition, fiber strength, fiber fineness, fiber length and color measurements were made. The chemical content of the obtained fibers (pectin, lignin, cellulose, hemicellulose) was determined. The properties of the fibers were compared using the obtained data. As a result of the study, it has been seen that the characteristic properties of the sunflower fibers obtained by the retting method are better. It has been determined that the elemental, thermal and crystal structures of the fibers obtained by different methods are similar. It was concluded that sunflower fiber will not be spun as a yarn, but can be used as a natural polymeric composite reinforcement material.

Keywords: *Helianthus annuus* L., Sunflower stem, Bast fibers, Retting, Decortication, Fiber qualities

1. Introduction

A large amount of waste is generated in the post-harvest production area of most cultivated plants. The wastes of plants such as flax, hemp, ramie and jute contained in cellulosic bast fibers are an important source of raw materials for the textile, composite and paper sector. By evaluating these wastes, it is thought that by obtaining qualified, ecological and naturally decomposing sunflower fiber from the stem of the sunflower plant, which is an agricultural waste, it can increase the added value of agricultural products and contribute to the protection of the environment. With the increase in population in our world, natural cellulosic raw material resources are gradually decreasing due to the fact that agricultural areas are used more for the food sector [1].

The sunflower plant (*H. annuus* L.), which belongs to the Asteraceae family, is an annual plant belonging to the Helianthus genus in the Asterales order [2]. 136 genera and 1345 species from the Asteraceae family grow in our country. The largest number of endemic species are in the Asteraceae family. It is one of the richest families in terms of species and genera [3]. The sunflower plant, which belongs to the Asteraceae family, is an annual plant belonging to the Asterales order, *Helianthus* genus and *annuus* species. The genus *Helianthus* has 51 species and 19 subspecies in the world. 14 of these species are annual and 37 are perennial [2]. There are two important species of the Helianthus genus (*Helianthus tuberosus* L., *Helianthus anuus* L.) in our country. In our country, it is known as gündöndü, günebakan and günçiçeği [4].

Sunflower grows in subtropical and temperate climate conditions in July-August. It is a plant species that loves light and sun. It grows in moist, organic matter-rich, well-drained, neutral pH (6.5-7.5) soils with high water holding capacity and mostly in arid and semi-arid climates [2,5]. Sunflower, which has single and large-headed capitula, contains a large number of achene. Sunflower does not consist of a single flower, but consists of 1,000 to 2,000 individual flowers connected to the table base. It has a strong tap root structure that spreads deep and wide. It can use water from 3 meters deep with its root structure [2,6]. Although the sunflower plant has many uses, it is an important raw material source especially for animal feed, fertilizer, soap, chemistry, cosmetics, paint, biodiesel, plastic and food industries. Large amounts of oil sunflower are produced in our country [6]. Sunflower stalks left in the field after the sunflower plants produced in our country are collected cause problems. The stems are collected from the field and destroyed by burning to warm up in the winter months, they are mixed into the soil by breaking up or they are burned after harvesting [7].

In the current study, it is aimed to determine the morphological properties of the sunflower stem and to obtain natural lignocellulosic fiber from the sunflower stem. In addition, examining the chemical, physical and mechanical properties of the obtained fibers and the details obtained has become another target. Two different methods were used to obtain sunflower fiber: retting and decortication of fresh and dried stems. The usability of previously broken sunflower fiber as a polymeric composite reinforcement material, its rights were obtained, and the usability of these fibers in textiles was emphasized. In addition, we will attach great importance to increasing the recognition of fibers and disseminating plant fibers obtained from management waste for the future of many industries, especially textile. It is believed that the research outputs will contribute to the literature on reducing environmental pollution produced by plant waste and producing soluble fiber that meets the need for natural cellulose from the stems of the sunflower plant, which are agricultural residues.

2. Materials and Methods

To obtain fiber from the stem of the sunflower plant, 75 kg of sunflower plants were collected from the Altınova district of Balıkesir province in August 2022. Since the sunflower is harvested in August, this month was chosen as the harvest time. 75 kg of plants were collected in 1 hour. Samples to be used in anatomical studies were stored in 70% ethanol during fieldwork.

2.1. Anatomical Properties

The stem of the sunflower plant examined in the study was kept in glycerin for 15 days to soften. Transverse and longitudinal sections were taken as thin layers from the stored sunflower stem with the help of a razor blade. The obtained sections were examined and photographed using a LEICA DM 750 camera microscope.

2.2. Obtaining Fibers

In order for the fibers to be used for different purposes after the harvest of the sunflower plant, first of all, the fibers must be separated from the stems. The process of separating the fibers from the stems was done by retting, decortication (fresh stem, dried stem) methods. The purpose of retting and decortication (dried stem, fresh stem) methods is to easily separate the woody parts of the stems and to expose the fibers [8].

2.3. Combing of Fibers

The resulting sunflower stems were combed to separate the woody parts. Three different combs were used in the study. First, the stems were passed through an 8 cm wide, 2-row, 36-tooth comb. Thus, the fiber was separated from the woody regions (lignin). Afterwards, the fibers were cleaned by combing again with two different combs with finer and denser teeth (79 teeth and 124 teeth), thus achieving a homogeneous and parallel appearance.

2.4. Characterization Analyzes

Various measurements were made to determine the physical, chemical and mechanical properties of the fiber obtained by different methods. The breaking strength, breaking elongation, color properties, fiber fineness and fiber length of the obtained fibers were examined. Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and Scanning Electron Microscope (SEM) analyzes were applied for structural characterization. Thermal analyzes were carried out with TG-DTA (Thermogravimetric) analysis. The breaking strength, elongation at break, color properties, fiber fineness and fiber length of the fibers kept for at least 24 hours in the laboratory under standard atmospheric conditions (20 °C ± 2 °C temperature and 65% ± 4% relative humidity) were examined.

2.4.1. Chemical Content of Fiber

The determination of the chemical structure of sunflower fibers was based on the "Chinese Textile Industry Standard" in the study of Zhang et al. (2013) [9].
Sunflower fibers were dried in an oven at 100 °C for 4 hours to reach constant weight (W0), then washed with 70% ethanol solution and dried. To determine the pectin content, it was mixed with 0.5% EDTA (ethylene diamine tetra acetic acid) solution in a magnetic heater for 30 minutes. After being rinsed with pure water and filtered, it was brought to a constant weight in the oven at 100 °C for 4 hours and measured (W1). Pectin content (%) was calculated using the formula below.

$$\% \text{ Pektin} = \frac{W_0 - W_1}{W_0} * 100 \qquad (2.1)$$

To determine the hemicellulose content of the fibers, in the next step, the pectin-free samples were washed with acetone, rinsed with pure water, dried and mixed with 0.5 M HCl at boiling point with a magnetic heater for 60 minutes. After being rinsed with pure water and filtered, it was dried in an oven at 100 °C for 4 hours and its weight was measured (W2). Hemicellulose content (%) was calculated using the formula below.

$$\% \text{ Hemiselüloz} = \frac{W_1 - W_2}{W_0} * 100 \qquad (2.2)$$

The fibers, which were kept in 72% sulfuric acid solution for 24 hours at room temperature to determine their cellulose content, were then vacuum filtered and rinsed using a sintered glass filter funnel (por 3). The fibers were dried in the oven and their weight was measured (W3). The amount of Klason lignin was also calculated by sulfuric acid treatment. Calculations (% Cellulose, % Lignin) were made using the formulas below.

$$\% \text{ Selüloz} = \frac{W_2 - W_3}{W_0} * 100 \qquad (2.3)$$

$$\% \text{ Lignin} = \frac{W_3}{W_0} * 100 \qquad (2.4)$$

2.4.2. Fiber Length

Sunflower fibers were combed and cleaned, and after making them homogeneous and parallel, the length of the resulting fibers was measured with the help of a ruler.

2.4.3. Fiber Fineness

The Metric System (Nm) was used to determine the fiber fineness. It was calculated by finding the length value in meters of a textile material weighing 1 gram. Since the thickness of the upper and lower parts of the plant's stem are different, the weight of 5 fibers, 8 cm long, was measured from both the lower and upper parts. This measurement was made twice and the weight average was taken.

2.4.4. Color Measurement

“Hunter Lab UltraScan PRO" brand color measurement device was used to determine the color changes in the fiber. L*, a* and b* color values of sunflower fibers obtained by three different methods were obtained. The L* value is in the range of 0-100 and 0 indicates black and 100 indicates white. In addition, a* and b* color values are used for the determination of darkness [10].

2.4.5. Fiber Strength

Strength measurement was carried out with testXpert II software on the “Zwick Roell Z010” device. Jaw gap was 100 mm/min, head speed was 100 mm/min and no pre-tension was applied. By making 10 measurements from each production method, the breaking strength (cN/dtex) of sunflower fiber and the standard deviation and coefficients of variation (%CV) of these values were obtained.

2.4.6. XRD (X-Ray Diffractometer) Analysis

XRD analysis was performed to determine the crystal structure of fibers obtained from sunflower stalks by different methods. The fibers are ground and ground into powder. Approximately 2 g of sample was performed on a Panalytical/ Empyrean model (Ni filtered CuKα beam source) X-ray diffraction device that can operate at 40 kV and 40 mA. Scanning was performed between angles of 10°-80° at a speed of 2°/sec.

2.4.7. FTIR (Fourier Transform Infrared Spectroscopy) Analysis

FTIR analysis was performed to determine the molecular structure of sunflower fibers. It was carried out with a Perkin Elmer Spectrum1400 brand spectrometer in the scanning range of 4000-650 cm-1 and at room temperature. The test was performed at 4 cm^{-1} resolution.

2.4.8. TG-DTA (Thermogravimetric) Analysis

TGA analysis was performed to determine thermal and gravimetric changes of sunflower fibers. It was carried out on EXSTAR TG/DTA7300 brand thermo gravimetric analyzer in nitrogen environment between 25-600ºC temperatures at a heating rate of 10ºC/min. The analysis result was calculated by taking as reference the curve of change of mass against time and temperature.

2.4.9. SEM (Scanning Electron Microscope) Analysis

In the SEM analysis, the fibers obtained by different methods and the transverse and longitudinal sections of the sunflower plant stem were placed appropriately on double-sided adhesive carbon tapes.

3. Results and Discussion

3.1. Anatomical Properties

The fibers in dicotyledonous sunflower plant stems, which have a secondary structure, are divided into three groups: primary and secondary bast fibers and xylem fibers [11]. The development of these three structures is independent of each other. Primary fibers are longer and of higher quality than secondary fibers and xylem fibers [12]. When viewed in cross section, it consists of protective tissue, cortex and central cylinder parts. The cortex is divided into two: primary cortex and secondary cortex. The primary cortex consists of primary phloem. There are sclerenchyma fibers in this part and these fibers

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are also called primary bast fibers. Secondary cortex consists of secondary phloem. There are phloem scleranchyma cells in the secondary phloem. The fibers formed in the phloem sclerenchyma are called secondary bast fibers. These fibers provide support to the phloem [13].

It consists of the central cylinder, secondary xylem and soft core. The soft core consists of spongy parenchymatic cells. There are sclerenchyma cells in the secondary xylem and therefore they are called xylem fibers [13].

Anatomical findings of the sunflower fibers examined in this study are given in Figures 1. and Figure 2. Transverse and longitudinal microscopic images of the sunflower stem.

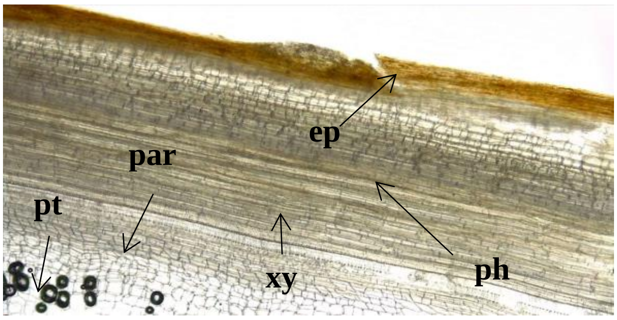

Figure 1. *Helianthus annuus L.* 10x magnified longitudinal sections of the stem of the plant (ep: epidermis, xy: xylem, ph: phloem, par: parenchyma, pt: pith)

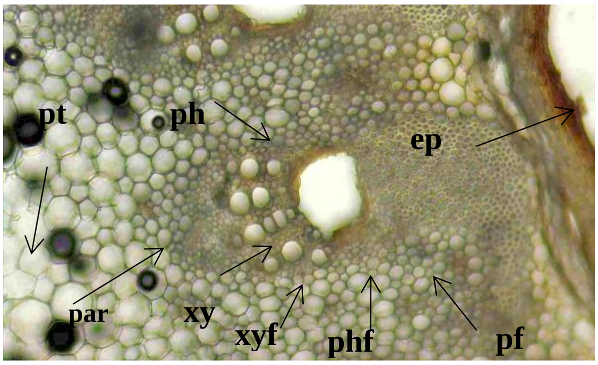

Figure 2. *Helianthus annuus* L. 10x magnified cross-section of the stem of the plant (ep: epidermis, xy: xylem, xyf: xylem fibers, ph: phloem, phf: phloem fibers, pf: primary fibers, par: parenchyma, pt: pith)

3.2. Obtaining Fibers

Since the fibers are very fragile, few and short fibers were obtained during the scans performed in the decortication (dried stem) method. In the retting method, less brittle and longer fibers were obtained compared to the decortication method (fresh stem, dried stem).

In this study, since the retting method produces less brittle and longer fibers than other methods, fiber was obtained by the retting method twice to calculate the fiber yield. As a result, 92 grams of fiber was obtained from 832 grams of sunflower stalks, yielding 11.6% efficiency.

3.3. Chemical Content of Fiber

In experimental studies conducted to determine the chemical content of sunflower fiber;
In the fibers obtained by the retting method, the cellulose rate was found to be 47.7%, the hemicellulose rate was 13.1%, the lignin rate was 31.1%, and the pectin rate was 12%. In the fibers obtained by the decortication (fresh stem) method, the cellulose rate was found to be 48.8%, the hemicellulose rate was 16.4%, the lignin rate was 21.7%, and the pectin rate was 13.7%. With the method obtained by decortication (dried stem), the cellulose rate in the fibers was found to be 55.5%, the hemicellulose rate was 19%, the lignin rate was 19.8%, and the pectin rate was 10.5%.

Fibers obtained from sun-dried stems were found to have a higher cellulose content than fibers obtained by other methods. Fibers obtained by retting method were found to have higher lignin content than fibers obtained by other methods. In addition, the cellulose ratio was low because the lignin was not removed sufficiently in the retting method. Chemical structure; Harvest time may vary depending on the climatic conditions in which the plant is grown, soil structure and the methods in which the fibers are obtained.

3.4. Fiber Length

The longest fibers were obtained by the retting method (16-37 cm). During scanning, it was determined that the shortest fibers were obtained by the decortication (dried stem) method (6-15 cm) since the fibers were fragile.

3.5. Fiber Fineness

According to the findings, the thickest fibers were obtained by the retting method and the thinnest fibers were obtained by the decortication (dried stem) method (Table 1.). It has been determined that it consists of thinner fibers than the upper part of the stem and thicker fibers than the lower part of the stem.

Table 1. Fiber fineness results according to the fiber obtaining method

Method	Upper part of the stem (Nm)	Lower part of the stem (Nm)
Retting	~ 4,2	~ 3,7
Decortication (fresh stem)	~ 10,5	~ 4.5
Decortication (dried stem)	~ 14,8	~ 5,9

3.6. Color Measurement

In this study, the a * value in the color system is between 4 and 7; It was determined that the b * value varied between 19 and 30 and all a * and b * values obtained as a result of the measurement were positive (Table 2). Accordingly, the fibers obtained by decortication (dried stem) method are closer to red tones than the fibers obtained by retting and decortication (fresh stem); It is concluded that the fibers obtained by decortication (fresh stem) are closer to yellow tones than the fibers obtained by retting and decortication (dried stem). It is seen that the illuminance value (L*), which varies between 0 and 100, varies between 56 and 67 according to this study. According to the L* values determined as a result of the measurements of the fibers obtained by all three methods, the fibers obtained by the retting method were lighter in color than the fibers obtained by the decortication (fresh stem) and decortication (dried stem) methods. It was observed that it was darker in color than the fibers obtained by decortication (fresh stem) methods.

Table 2. Color measurement results of sunflower fibers according to the method of obtaining the fiber

Method	L*	a*	b*
Retting	67,11	4,15	19,18
Decortication (fresh stem)	64,55	7,07	30,24
Decortication (dried stem)	56,88	7,95	23,08

3.7. Strength Property

It is observed that the variation of the values obtained for the breaking strength is high. The reason for its high variation is that the thickness of the upper and lower parts of the sunflower plant's stem is different and the fibers are not uniform and homogeneous along their length. Fibers obtained by retting method have higher breaking strength values. Fibers obtained by decortication (dried stem) method were found to have the lowest breaking strength.

Table 3. Strength results of sunflower fibers

Method	Breaking strength (cN/tex)		
	Average	Standard deviation	% CV
Retting	6048	1715,68	28,37
Decortication (fresh stem)	2699,90	814,81	30,18
Decortication (dried stem)	811,70	339,98	41,88

3.8. XRD (X-ray diffractometer) Analysis

XRD analysis curves applied to examine and compare the effect of the internal structure of fibers obtained from sunflower stems by 3 different methods are shown in Figure 3. The intensities of the peaks showing (110) and (002) crystal structures varied between methods. When the peak intensities of sunflower fibers were examined, it was determined that the highest intensity was in the fibers obtained by the retting method, and the lowest intensity was in the fibers obtained by the decortication (dried stem) method. XRD measurements showed that sunflower fibers have similar crystal structures between the production methods.

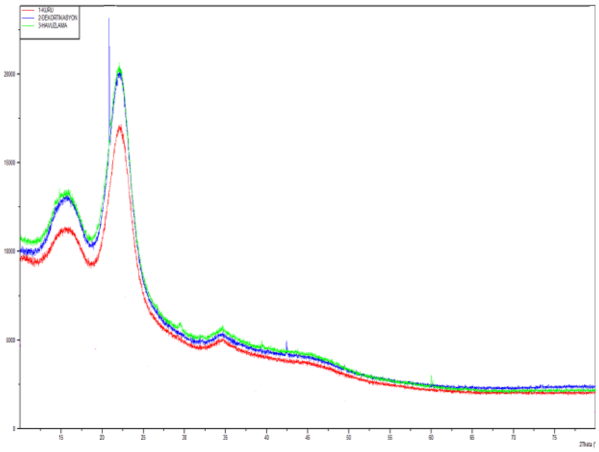

Figure 3. XRD curves according to the methods of obtaining sunflower fibers (Red- Retting, Blue- Decortication (fresh stem), Green- Decortication (dried stem)

3.9. FTIR (Fourier Transform Infrared Spectroscopy) Analysis

FTIR spectra of sunflower fibers obtained by 3 different methods are seen in Figure 4. The peaks in the FTIR spectrum show the functional groups of the basic components of cellulosic fibers such as cellulose, hemicellulose, lignin and pectin [14].

The peaks formed in the absorption bands between 3287.3-3319.5 cm^{-1} in the FTIR spectra show O-H voltage Decibels and alcohol groups. The presence of C-H group tension or CH_2 and CH_3 vibrations [15]. in cellulose and hemicellulose components was observed to occur in the absorption band of 2898.8; 2876.6-2953.1; 2852.5-2912.9. There has been a change in the fibers obtained by decortication (dried stem) method in these bonds. This is because weight loss occurs due to the removal of water. The absorption between 1729 and 1733.1 cm^{-1} belongs to the ester group in hemicellulose or the carbonyl (C=O) group of the bond vibration of carboxyl acid (R-COOH) in lignin [16]. This bond was broken down by the effect of microorganisms during the retting method. The peaks seen in the other absorption band showed that they had similar elemental properties according to different production methods.

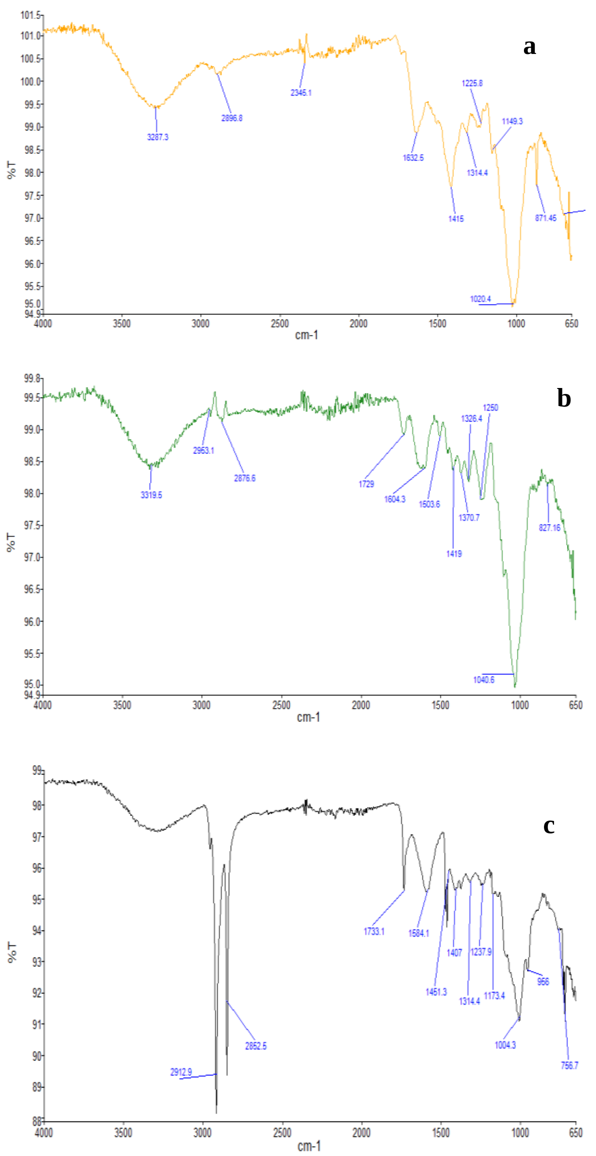

Figure 4. FTIR spectra according to the methods of obtaining sunflower fibers (a-Retting b- Decortication (fresh stem) c-Decortication (dried stem))

3.10. TG-DTA Analysis

The graphs obtained as a result of TG-DTG analyzes performed to determine the temperature resistance of fibers are given in Figure 5. The weight loss of fibers obtained by 3 different methods occurs as a result of multi-step reactions. Differences in the number of deteriorations are due to dehydration, decomposition events and material removal in the fibers. It is understood that this weight loss continues continuously with increasing heating rate and this process continues until the final temperature of 600°C.

When the basic decomposition temperature was compared, it was seen that it started and ended at approximately the same temperatures for all fibers.

The greatest weight loss is very slow up to 200°C, the slope between 250°C and 350°C indicates very rapid weight loss. However, as a result of the analysis, it is seen that the weight losses in the fibers are approximately the same. As a result, it was found that the thermal behavior and stability of fibers obtained by 3 different methods were similar.

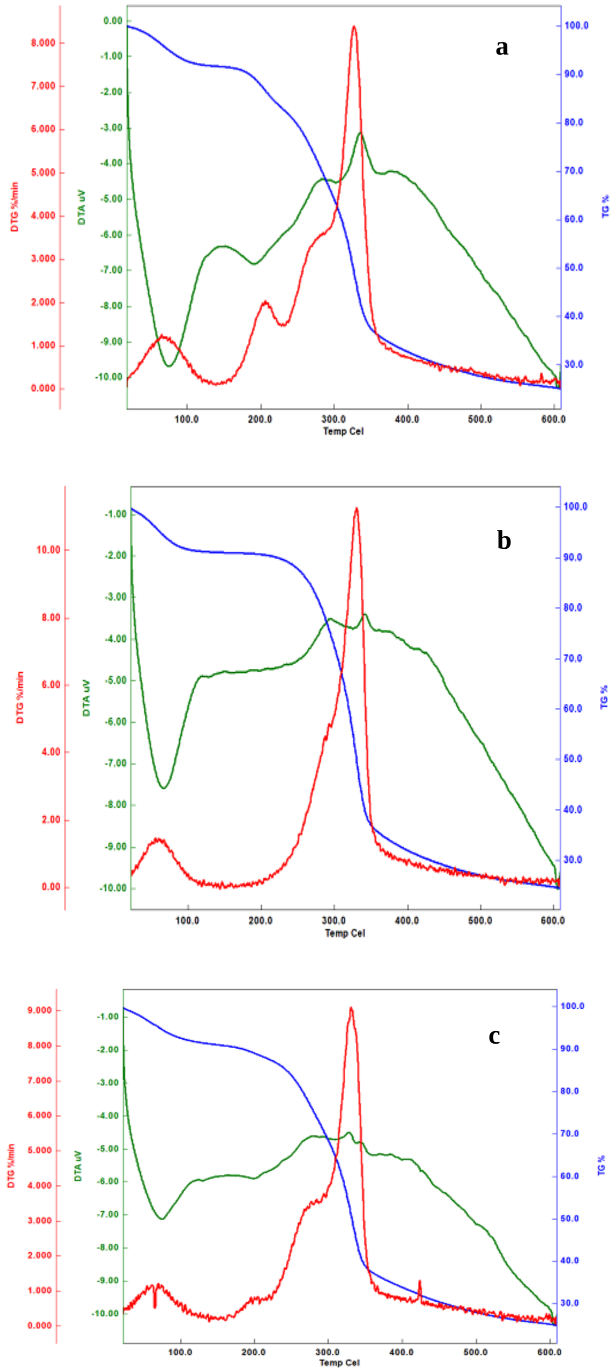

Figure 5. TG graphs according to the methods of obtaining sunflower fibers (a- Retting b- Decortication (fresh stem) c -Decortication (dried stem))

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3.11. SEM (Scanning Electron Microscope) Analysis

SEM microscopic images of transverse and longitudinal sections of the fibers are shown in Figure 6-7-8-9. Accordingly, the fiber thickness of sunflower fibers varies along the fiber. It contains many fibrils in the fiber and it is seen that the fibers are arranged in a parallel way and there is no space between them. The fiber is in the shape of a polygonal cavity (lumen) in the cell center. It was determined that there are many spherical pits on the surface of the fibers. Looking at the sunflower fiber, it is seen that the rough fiber surface and its surface are covered with impurities. Since the surface of the sunflower fibers obtained by retting method is covered with more substances compared to the fibers obtained by decortication (dried stem) and decortication (fresh stem) methods, the visibility of the pores has decreased.

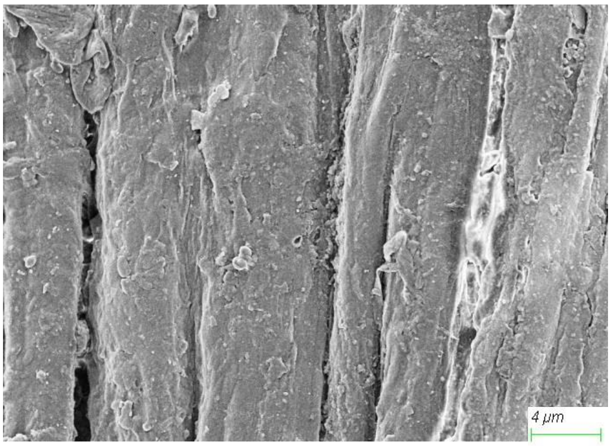

Figure 6. Longitudinal section SEM images of the fiber obtained by retting method from the stem of *Helianthus annuus* L. plant

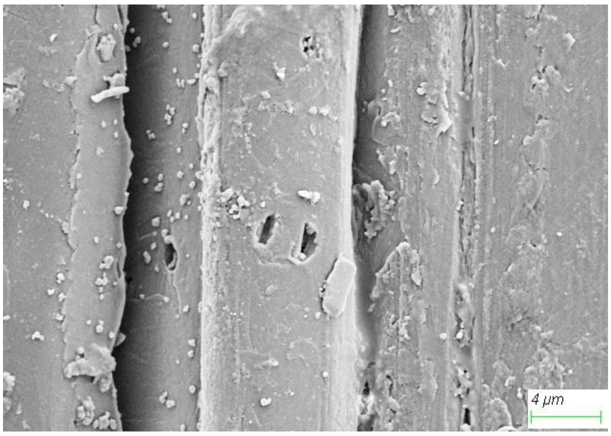

Figure 7. Longitudinal section SEM images of the fiber obtained by decortication (fresh stem) method from the stem of the *Helianthus annuus* L. plant

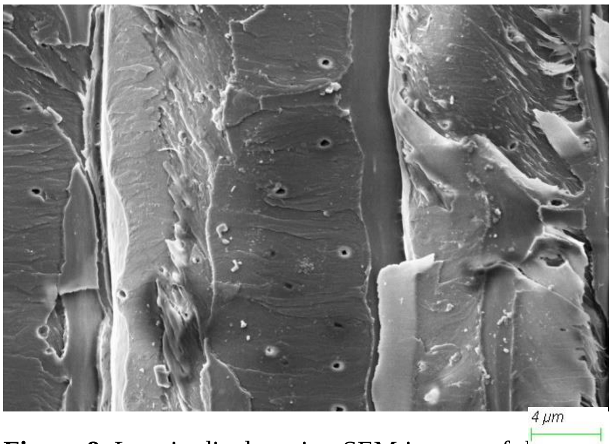

Figure 8. Longitudinal section SEM images of the fiber obtained by the decortication (dried stem) method from the stem of *Helianthus annuus* L.

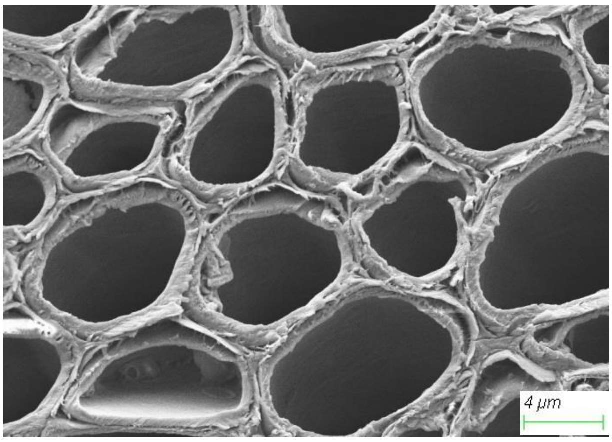

Figure 9. Cross-sectional SEM images of the fiber obtained from the stem of the *Helianthus annuus* L. plant

4. Conclusion

As a result of the study, when the properties of natural lignocellulosic sunflower fiber were compared, it was seen that the characteristic properties of the fibers obtained by the retting method were better. It has been determined that the elemental, thermal and crystalline structures of the fibers obtained by different methods are similar.

The purpose of cultivation of plants such as flax, hemp and jute is important. Farmers cultivate for their intended use. Sunflower is not used as a fiber plant, it is generally cultivated for oil production. The primary aim of farmers is to ensure oil yield. If fiber production is targeted, the added value of the fiber must be revealed and its economic study must be carried out. Additionally, farmers should be informed about the main and secondary uses of the fibre.

Planting and harvesting times should be planned according to the intended use. At harvest time, the fibers obtained from the stems of the sunflower plant are thick and hard due to the high amount of lignin. It is thought that when sunflower stalks are collected earlier than the harvest time, thinner, spinnable fibers can be obtained and these fibers can be used in the textile industry. Since the sunflower fiber obtained at harvest time will not be spun into yarn, it can be used as a natural polymeric composite reinforcement material. Nowadays, the use of natural fibers of plant origin in composite materials is increasing. The use and superior properties of composite materials in many areas come to the fore. The qualified, ecological and biodegradable properties of sunflower fiber will contribute to increasing the added value of agricultural products and protecting the environment.

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Author's Contributions

Nilsu Ataman: Performed the experiments and results analysis.
Levent Şık: Prepared experimental method, supervised the experiment's progress, result in interpretation and helped in manuscript preparation.

Ethics

There are no ethical issues after the publication of this manuscript.

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