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# Physical and Chemical Properties of a New Cellulose Fiber Extracted from the *Mentha pulegium L.* (Pennyroyal) Plant's Stem

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## Abstract

Ecological problems, high cost, and non-renewability of petroleum and its derivatives have increased the research on new sustainable natural products. For this purpose, the physical, chemical, and mechanical properties of *Mentha pulegium L.* (MPL) fiber, which may have potential for use in textile and composite sectors, were determined by extraction and characterization. Fiber density, length, and diameter were detected by physical tests. Cellulose, hemicellulose, and lignin ratios of the fiber were obtained by chemical analysis and confirmed by Fourier transform infrared (FTIR) spectroscopic analysis. The surface morphology was identified by scanning electron microscopy (SEM) analysis, and the chemical components on the fiber surface were discovered by X-ray photoelectron spectroscopy (XPS) analysis. Thermal degradation values of the fiber were found by thermogravimetric analysis (TGA), and the fiber's mechanical properties were determined by tensile test. As a result of the tests and analysis, MPL fiber has shown that it has potential for use in textiles and fiber reinforced composites.

**Keywords:** *Mentha pulegium L.*, Sustainability, Natural fiber, Composite, Textile

# Yaban Nanesi (Yarpuz) Bitkisinin Gövdesinden Ekstrakte Edilmiş Yeni Bir Selülozik Lifin Fiziksel ve Kimyasal Özellikleri

## Öz

Petrol ve türevlerinin ekolojik problemleri, yüksek maliyeti ve yenilenebilir olmayışı sürdürülebilir yeni doğal ürünlere olan araştırmaları artırmıştır. Bu amaçla tekstil ve kompozit sektöründe kullanım potansiyeli olabilecek yaban nanesi lifinin eldesi ve karakterizasyonu yapılarak fiziksel, kimyasal ve mekanik özellikleri belirlenmiştir. Fiziksel testler ile lifin yoğunluk değeri, uzunluk ölçümü ve lif çapı ölçülmüştür. Kimyasal analizler ile lifin selüloz, hemiselüloz, lignin oranları tespit edilerek, Fourier dönüşümlü kızılötesi (FTIR) spektroskopik analizi ile doğrulanmıştır. Yüzey morfolojisi taramalı elektron mikroskobu (SEM)

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analizi ile belirlenerek, X-ışını fotoelektron spektroskopi (XPS) analizi ile de lifin yüzeyinde yer alan kimyasal bileşenler saptanmıştır. Yaban nanesi lifine uygulanan termogravimetrik analiz (TGA) ile lifin termal bozunma değerleri, çekme testi ile de mekanik özellikleri tespit edilmiştir. Yapılan testler ve analizlerin sonucunda yaban nanesi lifi tekstilde ve lif takviyeli kompozitlerde kullanım potansiyeli olduğunu göstermiştir.

**Anahtar Kelimeler:** Yaban nanesi, Sürdürülebilirlik, Doğal lif, Kompozit, Tekstil

# 1. INTRODUCTION

In today's world where concepts such as environmentally friendly, renewable, sustainable, etc. are gaining importance, studies on new natural fibers are increasing [1,2]. Natural fibers, utilized in various industrial fields, are preferred as reinforcement elements in composite/bio composite production due to their specific properties, including low density, biodegradability, sourcing from natural origins, high specific strength, and easy availability of raw materials [3-5]. Supplements of natural fibers are derived from the fruits, leaves, and stems of plants [6].

Factors such as geographical growing conditions, extraction method, and maturity of the plants from which natural fibers are obtained, determine the physical structure and chemical composition of the fiber [7,8]. Therefore, in order to develop cellulose-based fiber-reinforced composites with high mechanical properties, it is important to determine the physical and chemical properties of the fiber through characterization processes. Fibers with high cellulose content generally have high strength. At the same time, lightweight composites can be produced with low-density fibers [8,9]. Hemp, flax, and sisal are widely used as natural fiber reinforcements in composite production [10]. However, the need for new natural fibers is increasing with the developing composite industry.

In this section, recent studies on new natural fibers are presented. New cellulose-based fibers such as *Ficus Carica* [11], *Thespesia populnea* [12], *Phaseolus vulgaris* [13], *Arenga Pinnata (Wrumb)* [14], *Cajanus cajan* [15], *Cissus quadrangularis* [16], *Ficus benjamina L.* [17] are presented in the literature as a result of the characterization of fibers obtained from trees and plants and it was concluded that they are suitable for utilization as reinforcing fibers in composite production. Natural fiber research from plants and trees and relevant new studies about them are being added to these examples every day.

The pennyroyal plant, which has the Latin name *Mentha pulegium L.*, is a pungent-smelling perennial herb with pubescent stems and leaves that can reach 10-30 cm in height and grow widely in the Mediterranean region of Turkey [18]. It is harvested on the mountain slopes and along the water banks, in moist and watery areas. Today, MPL is cultivated as a medicinal plant in various regions of Turkey. Both the leaf stalks and the stems of MPL are dried and used for medicinal purposes.

There is no study about its use in fiber-reinforced composite production in the literature. For this reason, in this study, the potential for use in composite or bio-composite production was investigated by characterizing the fibers obtained from the MPL plant's stem.

The physical and chemical properties of MPL fiber were examined through X-ray diffraction (XRD), FTIR, and TGA as well as the chemical structure analysis. The results were then utilized to investigate the applicability of MPL fiber in composite or textile production.

# 2. MATERIAL AND METHOD

The MPL fiber used in this study was collected from the hills of Sarıçam district of Adana province, located in the Mediterranean region of Turkey. In order to obtain the MPL fiber, the leaf parts were cut and removed in the first phase. Secondly, the stem parts of the plant were kept in a bucket of pure water for 10 days to give microorganisms time to act on the shell. After that, the fibers were separated and then dried in daylight for 48 hours. Figure 1 displays the MPL plant (a) along with the fibers extracted from the stem part (b), and, tuft of MPL fiber samples (c).

(a)

(b)

(c)

**Figure 1.** The digital images of (a) MPL plant, (b) obtained MPL stem fibers (c) combed MPL samples

The density of MPL fiber was calculated according to ASTM D8171-18 standard, while its cellulose, hemicellulose, and lignin content was calculated according to Mylsamy and Rajendran method [19].

To determine the mean fiber length, 100 fibers were randomly selected from a tuft of MPL fiber sample. Each fiber was straightened, and its length was measured with a ruler. The mean, standard deviation (SD), and coefficient of variation (CV) were computed using the measured MPL fiber length.

The atomic and chemical components (carbon, oxygen, etc.) of the fiber surface were determined by XPS on a ThermoScientific K-Alpha instrument. In Figure 2, the XPS analysis image of MPL fiber is shown.

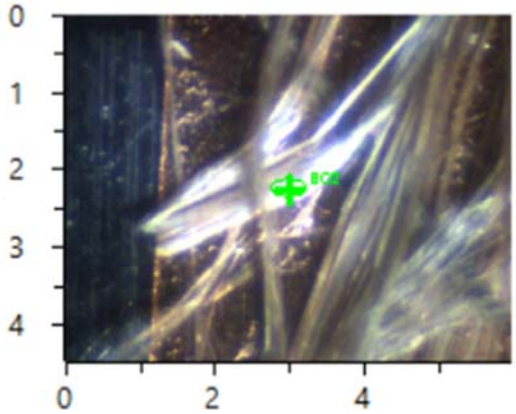


**Figure 2.** XPS surface measurement of MPL fiber

The molecular bond characterization of MPL fiber was performed by using FTIR spectroscopy, and its functional groups were identified between the 610-4000 $cm^{-1}$ wave number range in a Perkin Elmer Spectrum BX X FTIR instrument. The crystallographic properties of the fiber and the relevant phases were determined by using the PANalytical XPert Pro MPD device with a Cu-Kα beam source. The crystallinity index (CI) was deduced from the data generated by the reflection of the XRD pattern. The crystallinity index value was then calculated by using Equation (1) [20].

$$CI\ (\%) = (I_{200} - I_{am}) / I_{200} \quad (1)$$

In equation 1, $I_{2\,0\,0}$ and $I_{am}$, indicate the presence of amorph and crystalline regions in the fiber, respectively, and are used to calculate the crystallinity index percentage [21,22]. To determine the temperature and time-dependent mass loss of the fiber a Seiko SII TG/DTA 7200 device was used for TGA analysis.

The single fiber tensile tests of MPL fiber were performed by calculating the averages and standard deviations of the 30 repetitive tensile tests' results on 10 mm long fibers at a process speed of 0.1 mm/min under 50 N load on an INSTRON universal tester by complying with the ASTM D3822-07 standard. The surface properties such as surface structure, fiber diameter, surface porosity, etc. of MPL fiber were examined in a high-resolution SEM-ZEISS/EVO LS10 device.

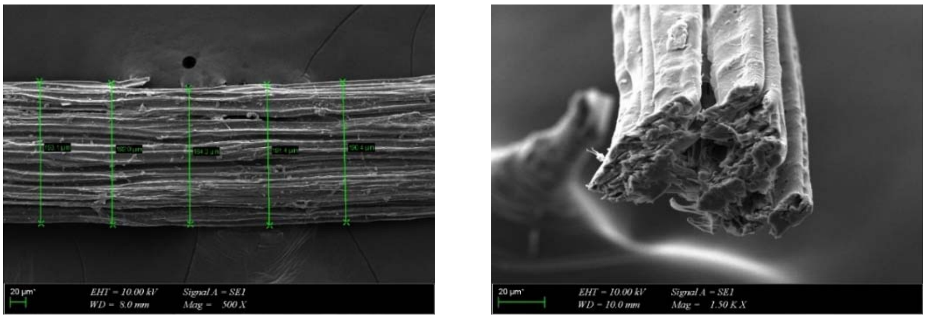
## 3. RESULTS AND DISCUSSION

The density of MPL fiber was determined as 1.4218 g/cm³, which is similar to halfa grass (alpha) fiber [23], and lower than that of sisal, ramie, and palm tree fibers [24]. Since low density (lightweight) is one of the main properties expected in cellulose-based fiber-reinforced composites, MPL fiber may be a good option. In Table 1, the comparison of the mechanical properties of MPL fiber along with other fibers is given.

**Table 1.** Comparison of the mechanical properties of MPL fiber along with other natural plant fibers

| **Fiber name** | **Fiber diameter (µm)** | **Tensile strength (MPa)** | **Tensile modulus (GPa)** | **Elongation (%)** | **Ref** |
|---|---|---|---|---|---|
| *Mentha pulegium L.* | 190.83±5.46 | 84.45±18.28 | 4.26± 0.7 | 3.27±0.64 | This Study |
| Coconut | 380 ±90 | 44±8 | 2.0 ±0.3 | 4.5 ±0.8 | [6] |
| Root | 100-650 | 157 | 6.2 | 3 | [25] |
| Coconut tree leafs heath | - | 119.8 | 18 | 5.5 | [25] |
| Alfa stems | 90-120 | 134-220 | 13-17.8 | - | [6,26,27] |
| Palm Tree Fruit | - | 65.2 | 4.9 | 47.2 | [6,28] |
| Corn husk | 0.186 | 160.49 | 4.57 | 2.08 | [24] |
| Palm Tree | - | 248 | 3.2 | 28 | [24] |
| Rami | 50 | 220-938 | 44-128 | 2-3 | [24] |
| Sisal | 50-300 | 511-635 | 9-22 | 2-2.5 | [24] |

Length and fineness are key characteristics of the fiber's spinnability in yarn production. MPL has an average length of 89.4 mm with a length range of 60 to 120 mm. It can be observed that MPL has an average length in a comparable range to flax fibers (60-90 mm) [29]. The calculated SD and CV of the measured fibers are 16.55 mm and 18.51%, respectively. The mechanical tests of MPL fiber provided similar results to the natural fibers given in Table 1. The mechanical properties of cellulose-based fibers are directly affected by the physical structure as well as the chemical component ratios [6,26,27]. When the literature on natural fibers is investigated, their mechanical properties and values vary greatly. These variations depend on factors such as growing conditions and plant properties, as well as fiber extraction method, harvesting period, and the maturity, etc. [30]. SEM images of the MPL fiber's transverse and longitudinal sections are shown in Figure 3.



(a) (b)

**Figure 3.** SEM images of MPL fiber's (a) longitudinal section, (b) transverse section

When the longitudinal section image given in Figure 3 (a) is examined, the fiber diameter is approximately 190.83 µm. This value was determined by calculating the average of the measurements taken from this image.

The average diameter value of MPL fiber was found to be thicker than that of corn husk, ramie, and halfa grass fibers [24], while thinner than that of root [26], and coconut fibers [6]. It was seen that there are irregularities as well as impurities on the fiber surface (oil, wax, lignin, etc.) [31]. The irregularities seen on the fiber surface increase the surface roughness. It is known that the use of fibers with rough surfaces in composite structures contributes to the bonding and the interfacial compatibility of fiber and the matrix by causing mechanical interlocking between them. Thus, using fibers with surface roughness as reinforcing elements in composites provides a great advantage [22].

The chemical compositions of MPL and other natural plant fibers are given in Table 2. The chemical composition ratios that comprise the fiber structure are affected by the environmental conditions, the extraction methods, and the soil properties where the plant grows [32]. The main chemical components of MPL fiber consist of cellulose (59.1%), hemicellulose (15.93%), and lignin (22.15%).

**Table 2.** The main chemical constituents of MPL and other natural plant fibers

| **Fiber** | **Cellulose (%)** | **Hemicellulose (%)** | **Lignin (%)** | **Ref** |
|---|---|---|---|---|
| *Mentha pulegium L.* | 59.1 | 15.93 | 22.15 | Current study |
| *Acacia nilotica L.* | 56.46 | 14.14 | 8.33 | [32] |
| Alfa stems | 45 | 24 | 24 | [6,26,27] |
| Bagasse | 55 | 17 | 25 | [6] |
| Coir | 26.8 – 43 | 17.2 – 37 | 22.2 – 45 | [6] |
| Bamboo | 26 | 31 | 30 | [33] |
| Corn husk | 46.15 | 33.79 | 3.91 | [24] |
| Ramie | 68.6 – 85 | 13 – 16.7 | 0.5 – 0.7 | [24] |
| Sisal | 60 – 78 | 10 – 14.2 | 8 – 14 | [24] |

It was determined that the MPL fiber has a higher cellulose content compared to that of prickly acacia, halfa grass, sugarcane coconut, bamboo, and the corn stover fibers given in Table 2. The relatively high cellulose content of fiber positively affects its mechanical properties [32], while other properties such as biodegradability, moisture absorption, thermal change, etc. usually vary depending on the fiber's hemicellulose content [33]. Here, 15.93% of the hemicellulose content in MPL fiber is found to be similar to that of prickly acacia [32], coconut [6], ramie [24], and sisal fibers [24]. The ratio of lignin, one of the main chemical components, directly affects both the structure and the surface morphology of the fiber [33]. Here, the lignin content of MPL fiber is found to be higher than that of *Acacia nilotica L.* [32], corn husk [24], and ramie fibers, while lower than that of bagasse [6], coconut [6], and bamboo fibers [33].

Both the chemical structure and the surface formation of MPL fibers were investigated by using the XPS analysis method. According to the as-obtained analysis data, the atomic surface elements detected on the fiber surface are shown in Table 3.

**Table 3.** The atomic surface components of MPL fiber

| Atomic surface elements (%) | | | | |
|---|---|---|---|---|
| **Fiber** | **C1s** | **O1s** | **N1s** | **Si2p** |
| **MPL** | 62.3 | 22.9 | 0.7 | 2.32 |

The elemental composition of MPL fiber surface was determined as 62.3% of carbon (C1s), 22.9% of oxygen (O1s), 0.7% of nitrogen (N1s), and 2.32% of silicium (Si2p). The calculated Oxygen/Carbon (O/C) and Carbon/Oxygen (C/O) ratios of 0.36 and 2.72, respectively, of MPL fiber indicate the hydrophilic or hydrophobic character of its surface. Here, increasing the C/O ratio decreases the hydrophilicity of the fiber surface, and improves the fiber-matrix compatibility in composites [34].

The C1s XPS spectrum curves of MPL fiber are shown in Figure 4. Here, the peak at 284.78 eV (C-C/C/C-H) represents the presence of hemicellulose and lignin, while the peak at 286.48 eV (C=O/O-C-O) represents the carbonyl groups, and the peak at 288.28 eV (O-C=O) represents either ester, carboxylic acid groups or lignin's presence [35].

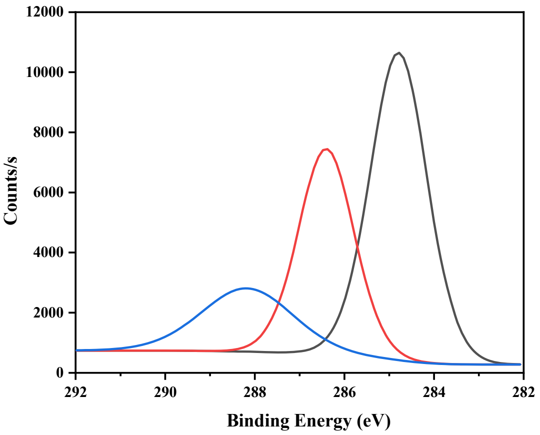


**Figure 4.** C1s XPS spectrum peaks of MPL. fiber

The XRD curves of MPL fiber are given in Figure 5. Here, the two main peaks seen at 18.53°, and 22.39° indicate the basic structure of cellulose I and IV, respectively. At the same time, these two planes indicate $(1\ \bar{1}\ 0\ /\ 1\ 1\ 0)$ and (2 0 0) crystallographic regions, respectively [36].

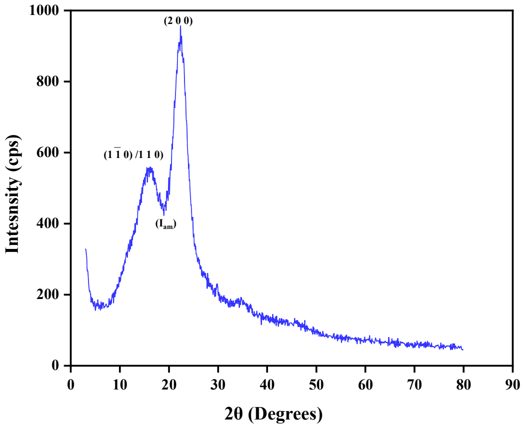


**Figure 5.** XRD curve of MPL fiber

Based on eq. (1), the crystallinity index (CI) value of MPL fiber was calculated as 53.97%. Compared to the CI values of other natural fibers in the literature such as cotton (60%), sisal (71%), jute (71%), flax (80%), and hemp (88%), it shows that the crystallinity of MPL fiber (53%) seems less regular [37].

The FTIR analysis was performed to investigate the chemical structure of MPL fiber, and its spectrum is given in Figure 6. Here, the peak at 3337 cm $^{-1}$ shows the hydrogen bonds and the -OH stretching in the cellulose structure [38]. The CH ($CH_2$ and $CH_3$) vibration of the cellulose and hemicellulose components' alkyl groups generated a peak at 2917 cm$^{-1}$ [39]. The peak at 1737 cm$^{-1}$ corresponds to the C=O tensions in carboxyl acid or acetyl groups, and C=C vibrations in aromatic rings in lignin correspond to the peak at 1527 cm$^{-1}$ [40,41]. The peak at 1396 cm$^{-1}$ is the bending vibration of C-H groups, while the peak at 1285 cm$^{-1}$ indicates C-O tensions in acetyl groups in hemicellulose, and the small peak at 1190 cm$^{-1}$ indicates the C-O-C asymmetric tensions [40–42]. The peak observed at 1022 cm$^{-1}$ indicates the aldehyde/acid/carboxylate functional groups, whereas the amorphous and crystalline cellulosic structures of the fiber can be understood from the peaks at 1464 cm$^{-1}$, and 801 cm$^{-1}$, respectively [41].

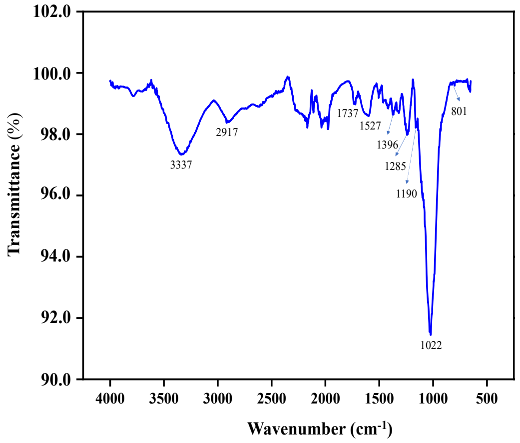


**Figure 6.** FTIR spectrum of MPL fiber

It is important to determine the effect of different temperature values on the fiber. In particular, it is necessary to determine the thermal degradation values of natural fibers that are used in the production of thermoplastic-based composites [22,43,44]. The mass loss of MPL fiber at different temperature values is shown in Figure 7.

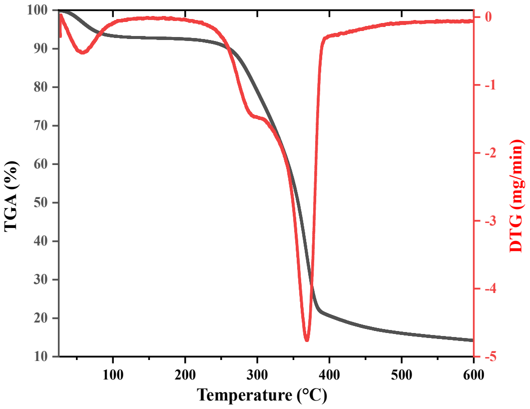


**Figure 7**. TGA-DTG curve of MPL fiber

Here, the subsequent degradation was determined at 290.34°C. This temperature value corresponds to the decomposition of hemicellulose in fiber's chemical structure [45]. The temperature value showing the decomposition of cellulose and lignin in the fiber structure was observed at 369.18 °C [46]. When the studies conducted in the literature were examined, it was seen that the thermal degradation temperature of MPL fiber is similar to that of sisal (340°C), jute (365°C), and flax (345°C) fibers [47].

The thermal analysis conducted between 30-600 °C indicated 85% of mass loss in the fiber structure. As a result of the TGA analysis, it was concluded that MPL fiber can be used as a reinforcing fiber in composite structures under an initial temperature of 290.34 °C [48].

## 4. CONCLUSION

In this study, a cellulose-based fiber extracted from the stem of an MPL plant was characterized, and its potential for use in composites was investigated. The physical, chemical, mechanical, morphological, and the thermal properties are determined by the fiber characterization processes. The MPL fiber's low density value of 1.42 g/cm$^3$ provides an advantage in composite and textile production. MPL fibers are found to have a 59.1% cellulose ratio in their structure, which enhances the mechanical properties of fiber-reinforced composites. Having a 53.97% crystallinity index ratio makes the MPL fiber rich in terms of cellulose. It was also understood from the results that the MPL fiber has better mechanical properties than some other fibers used in composite production with 84.45 MPa of tensile strength, and 4.26 GPa of modulus values. The chemical component details of MPL fiber were determined by FTIR analysis, while the chemical components in its surface structure were determined by XPS surface analysis, and it was understood that MPL fiber has a hydrophobic character compared to other fibers. Both MPL fiber's surface morphology, and its diameter were measured by SEM analysis, and it was seen that this fiber can be used in composites with its current structure. As a result of thermal analysis, it was determined that the fiber structure can remain intact at temperatures below 290°C, and will contribute positively to fiber reinforced composite structures. MPL fiber can also be used in the textile sector in combination with other synthetic fibers to lower the need for petroleum-based fiber production. Furthermore, since MPL's fiber length and fineness are comparable to those of flax, it can be utilized as a blended yarn.

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