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RESEARCH ARTICLE

Effect of enzyme treatments on the performance of fibers obtained from inner and outer corn husk leaves

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Abstract

Fibers were produced from inner and outer corn husk leaves by alkalization followed by enzyme treatment at various concentrations of xylanase. The physical, mechanical, thermal, and chemical properties of the obtained corn husk fibers were studied. The inner and outer corn husk fibers respond differently to enzymatic treatments. In general, the linear density, effective diameter, and water absorptivity of the fibers decreased with enzyme treatment. An increase in xylanase concentration led to a substantial decrease in the elongation of inner corn husk fibers, while leading to an increase in stiffness. The fibers produced from the outer corn husk leaves exhibited higher thermal stability, with greater thermal decomposition stage temperatures and lower mass loss, in comparison to those from the inner husk leaves. Thermogravimetric and FT-IR analyses revealed that the fibers from the inner corn husk leaves contained less noncellulosic content than those from the outer husk leaves.

Keywords: Biodegradable, Corn husk, Enzyme, Fiber, Thermal Stability.

Introduction

The escalating public awareness of environmental issues, including depleting petroleum sources, reducing landfill spaces, and harmful incineration emissions related to industrial practices depending on common synthetic raw materials, prompted public enterprises, research institutes, and civil entities to use bio-based raw materials. Novel products based on natural raw materials should possess renewable resources and exhibit biodegradability and/or recyclability, without leading to detrimental environmental effects during the manufacturing, service, or after-disposal stages. Moreover, their properties should be maintained during storage and use, and their performance and cost should be comparable to those of conventional materials (Bismarck et al., 2005).

In this sense, conventional lignocellulosic fibers, including flax (Rouibah et al., 2020), hemp (Sahi et al., 2022), kenaf (Aggarwal et al., 2022), jute (Tshifularo et al., 2020), sisal (Zhang et al., 2025), and bamboo (Prasad et al., 2022), have received revived interest over the last three decades, while agricultural residues, which are unconventional fiber sources, have not been the focus of intensive research studies until the beginning of the twenty first century (Reddy & Yang, 2005; De Rosa et al., 2010). Using agricultural remains as raw materials has the potential to provide important benefits for the agricultural community and the environment (Çaloğlu & Binay, 2023). Sugarcane bagasse (Foroushani et al., 2025), corn husks (Xing et al., 2025; Reddy &

Yang, 2005), okra stems (Prasanth, 2025), nettle stalks (Büyükkaya, 2025), and banana bracts (Sakthivel et al., 2021) are some agricultural residues investigated as fiber sources.

Until recently, corn husks have received limited research attention as a fiber source. This situation contradicts its abundance worldwide. Corn is the most produced grain (approximately 1.3 billion tonnes) globally, exceeding wheat (~800 million tonnes), rice (over 500 million tonnes), and soybean (~400 million tonnes) in 2021. Corn is produced in diverse regions (OECD, Food and Agriculture Organization of the United Nations, 2022) in over 170 countries (Liu et al., 2025). In the 2023/24 production year, 9 million tons of corn were produced in Türkiye by an estimated 300,000 farmers on an area of 9.1 million decares. While Turkiye's corn production in 2000 was 2.3 million tons, this figure reached 9 million tons in 2023/2024, corresponding to a four-fold increase. Corn, which has been produced for centuries to meet traditional food needs, is now the subject of numerous studies on the production of food components, biofuels, and biodegradable materials, and has great commercial value (NİSAD, n.d.). The remains of corn plants following harvesting can be valorized as fiber sources. The fibers extracted from corn husks have a chemical composition of 57.7% cellulose, 4.46% lignin, 34% hemicelluloses, and 3.77% pectin (Lv et al., 2017).

Enzymes are highly efficient biocatalysts that are widely used in bioengineering procedures (Duman et al., 2020). Enzymes have been used in different application areas, including biomedicine (Akarsu et al., 2025), drug delivery (Aktar et al., 2024), detergents (Hasbek & Yıldırım, 2025), textile processing (Yağcı & Duman, 2021), paper and pulp industry, agriculture (Çaloğlu & Binay, 2023), and food production (Duman et al., 2020). Owing to their cost-effectiveness, harmlessness, and low environmental impact, enzyme treatments are generally considered more advantageous than conventional chemical treatments (Hasbek & Yıldırım, 2025; Akarsu et al., 2025; Arantes et al., 2020).

Enzymes have been used to process lignocellulosic biomass. Different enzymes act on various components. Accordingly, cellulase, laccase, xylanase, and pectinase affect cellulose, lignin, hemicelluloses, and pectin, repectively. Enzymes have been used to extract fibers from agro-residues. A limited number of studies have focused on the use of enzymes on corn husk fibers. Research efforts on the use of enzymes on corn husks have focused on isolating fibers using cellulase and xylanase enzymes. Among these few studies, Reddy & Yang (2005) obtained fibers from corn husk leaves via an alkalization process succeeded by an enzymatic treatment (2005). They reported that the resultant corn husk fibers exhibited medium tenacity, high extension rate, and high moisture content. In such a study, Yılmaz (2013) produced fibers by degumming corn husk leaves in aqua and an alkaline solution, followed by enzyme treatments. She reported that the enzyme processes resulted in stiffer fibers with higher elasticity modulus levels and low breaking elongation rates. Enzyme treatments led to improvements, especially in alkali-retted fibers. Thermal and chemical analyses revealed decreased non-cellulosic components upon cellulase and xylanase treatments, and spectrophotometric testing detected higher whiteness indices for enzyme-treated fibers. In another study, fibers were obtained from undried and dried corn husk leaves via alkalizing and enzyme treatments by the research group. The influence of drying and enzymatic treatments on corn husk fibers was investigated. The drying process reportedly caused no detrimental effects on the mechanical and textile performance. A decrease in linear density was reported as a result of drying and the xylanase enzyme, which was pronounced at higher enzyme concentrations. An increase in enzyme concentration first enhances and then deteriorates the tenacity and stiffness of the fibers (Yılmaz et al., 2014). In a recent study, researchers investigated single-stage enzyme treatments of corn husks to produce soluble and insoluble dietary fibers (Xing et al., 2025). They obtained good thermal durability, low crystallinity, high porosity, and accessible functional sites upon degradation of hemicelluloses and cellulose using *Penicillium oxalicum* cellulases.

In addition to being used as a raw fiber material, cornhusks can be used as feed and bedding (Xing et al., 2025; Reddy & Yang, 2005) or in biofuel production (Nisad, 2025). Therefore, it is important to have knowledge about which part of corn husks is more advantageous in terms of its characteristics and should be allocated for fiber production, while the other sections can be utilized for other purposes. This study was conducted to compare the inner and outer leaves of corn husks in terms of the mechanical, textile, thermal, and chemical characteristics of the extracted fibers, and the effect of environmentally friendly xylanase enzyme treatments on fiber characteristics. Fibers were obtained from the inner and outer corn husks via alkali retting. Enzyme treatments were applied to the extracted fibers at different concentrations, and the obtained fibers were tested to determine their performance characteristics.

Materials and methods

Materials

Corn husks were obtained from Denizli, Turkiye, during the fruiting season. An enzyme with the commercial name Pulpzyme, a xylanase, was supplied by Novozymes A/S, Denmark. Xylanase enzymes act by breaking the covalent bonds between lignin and cellulose and depolymerizing hemicellulose (Yılmaz, 2013).

Sample preparation

The medium and the surrounding leaves of corn husks were taken separately, while the leaves between them were removed to obtain a clear distinction between the former two portions. The medium and the surrounding corn husk leaves were subjected to an alkaline treatment with a 5 g/L NaOH solution for a duration of 30 minutes at 1:20 liquor ratio at boiling temperature in distilled water. The cellulose is generally resistant to alkali, while the non-cellulosic components of the husk including lignin, hemicelluloses, and pectin are not. Thus, alkalization led to separation of the cellulosic fibers by partially removing the non-cellulosic components which cement the fibers together (Lee et al., 2020). After alkalization, fibers were rinsed thoroughly, neutralized using a solution of 10% acetic acid, washed again, and then left to dry in ambient conditions. The photograph of the obtained fibers is shown in Figure 1.

Fibers were treated with Pulpzyme® enzyme solution at a temperature of 50°C with a 1:50 liquor ratio in distilled water at varying xylanase concentrations: 0, 2%, 4% and 6% based on fiber mass. All enzymatic processes were conducted simultaneously to eliminate the effects of uncontrollable parameters. Following the enzymatic treatments, fibers were first washed with boiling water to stop enzyme activity, followingly with tap water, and finally dried in ambient conditions. The experimental design was 2-factorial established with two levels of location of corn husk leaves: inner and outer, and four levels of xylanase concentration: 0, 2%, 4% and 6%.

Figure 1. Photography image of the extracted corn husk fibers.

Characterization

The physical properties (linear density, moisture content, and water absorption), mechanical properties, thermal stability, and chemical structure of the corn husk fibers were investigated. Before conducting the tests, the samples were conditioned at 21 °C and a relative humidity of 65% for at least 24 h.

The linear density of the fibers was tested according to ASTM D 1577-07 (ASTM International [ASTM], 2007). At least 13 measurements were performed for each sample. The measured linear density has the unit tex, which corresponds to the mass (g) of a km-long fiber. The fiber mechanical properties were determined in accordance with ASTM D 3822 (ASTM, 2007). A minimum of 20 measurements of each fiber sample were carried out with a load cell of 10 N at a crosshead speed of 15 mm/min and a gauge length of 2.54 cm. The tests were carried out using a Tinius Olsen H10KT(R) Tester with QMat for Textiles(R) software. The moisture content of the fibers was investigated according to ASTM D 2495-07 (ASTM, 2007). The masses of the conditioned fiber bundles were measured with a precision of 0.1 mg, dried in an oven at 105 °C for 16 h, cooled using a desiccator, and the mass was measured once more. The moisture content was calculated using Equation 1:

$$MC = \frac{OS - OD}{OS} \times 100 \quad (1)$$

where MC is the moisture content, OS is the original sample mass, and OD is the oven-dried sample mass. Three replicates were tested.

The fibers' water absorptive capacity values were determined as per ISO 9073-6.2000 (International Organization for Standardization [ISO], 2000). The fibers were first submerged in distilled water for 1 min at a depth of 2 cm below the water level and removed from the water for two minutes to remove excess water. The fibers were weighed before and after submerging and removing excess water. The water absorption capacity was obtained using Equation 2:

$$WAC = \frac{M_f - M_i}{M_i}. \quad (2)$$

Here, *WAC* symbolizes the water absorptive capacity, and M_i and M_f represent the masses recorded prior to and following submerging and excess water removal, respectively. Three replications were tested.

Thermogravimetric measurements were conducted concurrently using a Shimadzu DTG-60H (Shimadzu Corporation, Kyoto, Japan) Thermal Analyzer in a flowing nitrogen atmosphere (100 ml.min^{-1}). Highly sintered Al_2O_3 was used as the reference material. The ground samples were scanned from 25 to 600 °C at 10°C min^{-1} heating rate. First, the temperature calibration of the thermal analyzer system was performed under the same experimental conditions. For this purpose, the melting points of indium and tin, which were provided by Shimadzu, were checked. All experiments were performed in triplicate to obtain statistically significant results.

Fourier transform infrared (FTIR) spectra of the inner and outer corn husk fibers were obtained using a Perkin Elmer FT-IR Spectrophotometer Spectrum Two, US, in attenuated total reflectance (ATR) mode.

Statistical analysis

The findings of the physical and mechanical testing of the corn husks were statistically tested by a 2-factor ANOVA (a = 0.05), using the Analysis ToolPak Add-in of Microsoft Excel. The degree of freedom in terms of corn husk location was 1, and for enzyme concentration, it was 3. The statistical analyses of the physical and mechanical properties of the fibers are summarized in Tables 1 and 2, respectively.

Table 1. Summary of the statistical analysis of variance (ANOVA) for fibers' physical properties.

Source of variation	SS	df	MS	F	P-value	F crit
Linear density analysis						
Husk location	3252.077	1	3252.077	41.63975	4.46E-09	3.940163
Enzyme concentration	1327.133	3	442.3777	5.664226	0.001291	2.699393
Interaction	384.7039	3	128.2346	1.641923	0.184805	2.699393
Within	7497.629	96	78.1003			
Total	12461.54	103				
Moisture content analysis						
Husk location	9.378707	1	9.378707	2.827685	0.112064	4.493998
Enzyme concentration	7.554598	3	2.518199	0.759238	0.533204	3.238872
Interaction	2.151694	3	0.717231	0.216246	0.883679	3.238872
Within	53.0679	16	3.316744			
Total	72.1529	23				
Water absorption analysis						
Husk location	0.084078	1	0.084078	0.765739	0.394485	4.493998
Enzyme concentration	3.872064	3	1.290688	11.75496	0.000255	3.238872
Interaction	0.135875	3	0.045292	0.412494	0.746288	3.238872
Within	1.756791	16	0.109799			
Total	5.848807	23				

Results and discussion

The physical, mechanical, thermal, and chemical properties of the obtained corn husk fibers were studied. The fiber yield of the inner and outer corn husk leaves was determined.

Fiber yield

The yield of the outer husk fibers, which was 9.58%, was greater than that of the inner husk fibers, which was 8.20%. The yield percentage values were determined based on the undried husk weights. There is some evidence that the difference is statistically significant ($p = 7.04\times10^{-2}$). The obtained fiber yield is slightly higher than that of Yılmaz et al. (2014), who obtained 7.13-7.14% by extracting corn husk fibers via alkalization. Their alkali concentration (7.5 g/L NaOH) was higher than that used in the current study (5 g/L NaOH), which might have led to greater non-cellulosic component removal.

Fiber linear density

The effects of enzyme treatment and the location of the cornhusk leaves on the fiber linear density values are shown in Figure 2. The xylanase treatment and the husk leaf location significantly influenced the fiber linear density (p values 1.29×10^{-3} and 4.46×10^{-9}, respectively). The fibers obtained from the surrounding corn husk leaves were found to be more mature and presented a coarser structure than those obtained from the inner husk leaves. During maturation, plant cells exhibit deposition of higher lignin content, which may be associated with coarseness (Rencoret et al., 2011). The surrounding corn husk leaf fibers exhibited linear density levels higher than those of the inner corn husks. This might be explained by the increase in diameter that occurs with maturation. However, no significant difference was observed in the

Table 2. Summary of the statistical analysis of variance (ANOVA) for fibers' mechanical properties

Source of variation	SS	df	MS	F	P-value	F crit
Initial modulus analysis.						
Husk location	4569.178	1	4569.178	0.489471	0.485233	3.903366
Enzyme concentration	61036.69	3	20345.56	2.17951	0.09276	2.664107
Interaction	31269.77	3	10423.26	1.116587	0.344284	2.664107
Within	1418909	152	9334.926			
Total	1515784	159				
Breaking force analysis						
Husk location	736901	1	736901	40.32666	2.36E-09	3.903366
Enzyme concentration	95263.17	3	31754.39	1.737748	0.161648	2.664107
Interaction	12982.4	3	4327.468	0.236819	0.870584	2.664107
Within	2777541	152	18273.29			
Total	3622687	159				
Breaking tenacity analysis						
Husk location	106.0688	1	106.0688	4.49715	0.035574	3.903366
Enzyme concentration	26.93404	3	8.978015	0.380654	0.767085	2.664107
Interaction	48.24416	3	16.08139	0.681826	0.564456	2.664107
Within	3585.038	152	23.58577			
Total	3766.285	159				
Breaking tenacity analysis						
Husk location	0.158131	1	0.158131	0.011577	0.914458	3.903366
Enzyme concentration	505.2628	3	168.4209	12.33018	2.9E-07	2.664107
Interaction	298.7969	3	99.59898	7.29169	0.000133	2.664107
Within	2076.205	152	13.65924			
Total	2880.423	159				

fibers subjected to enzyme treatments at different concentrations. This suggests that the fibers were saturated by xylanase enzymes at 2% concentration in terms of non-cellulosic material removal. The lowest linear density (19.9 tex) was obtained from the 6%-xylanase-treated fiber sample obtained from the inner corn husks, corresponding to the finest fibers obtained in this study. This linear density is finer than that of the fiber sample with the lowest linear density, 21.6 tex, attained by Yılmaz et al. for a 0.4% Pentopan BG treated fiber sample obtained from dried corn husks (2014).

Owing to the highly irregular and substantially variable cross-sections of natural fibers, including corn husk fibers, it is not practical or accurate to measure the cross-sectional area to calculate the diameter of the fibers in this study. Thus, the obtained fiber linear density value was used to obtain the effective fiber diameters for a more dependable solution via the following equation:

$$D = 2\sqrt{\frac{L}{\pi\rho \times 10^6}}, \quad (4)$$

where, D represents the effective fiber diameter in μm, L stands for the linear density in tex ($g{\cdot}km^{-1}$), and, ρ is the fiber density in $kg{\cdot}m^{-3}$. Here, the corn husk fiber density was adopted as 1480 $kg{\cdot}m^{-3}$, as reported by Pratheesh et al. (2022). The effects of corn husk location and enzyme concentration on fiber diameter are presented in Figure 3. The fiber diameter exhibited a trend similar to that of the fiber linear density. The obtained fiber diameters are generally higher than those used in textile applications, such as flax, hemp, and ramie (Lee et al., 2020).

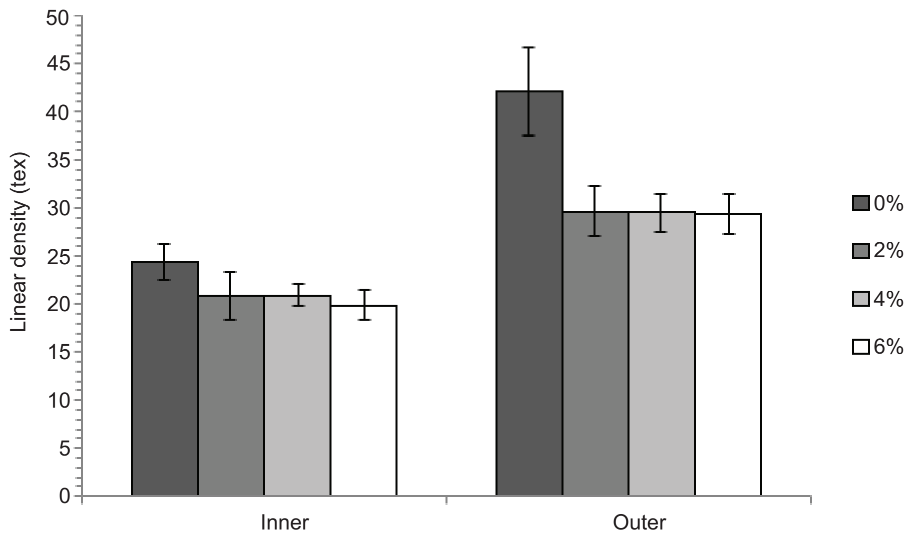

Figure 2. The effect of the enzymatic treatments on the linear density (tex) of the fibers extracted from the inner and outer corn husk leaves. The legend stands for the enzyme concentrations. Note: All error bars in the images represent standard errors.

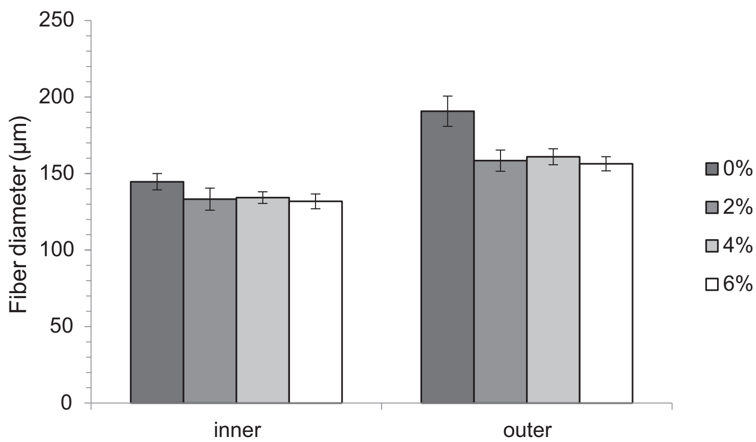

Figure 3. The effect of the enzymatic treatments on the effective diameters (μm) of the fibers extracted from the inner and outer corn husk leaves. The legend stands for the enzyme concentrations.

Fiber moisture content

Figure 4 shows the influence of enzyme concentration on the moisture content of the fibers from the inner and outer corn husks. Although the xylanase concentration did not significantly affect the moisture content ($p=1.3 \times 10^{-1}$), there was some evidence that the location of corn husk leaves significantly affected the moisture content ($p=0.11$). The outer husk fibers generally exhibited a higher moisture content than the inner husk fibers. In general, the obtained fibers exhibited moisture content between 6.89 – 9.98 %. This is similar to that of hemp fibers with a moisture content in the range of 6 – 12% (Lee et al., 2020).

Fiber water absorption

Corn husk fibers absorb 2–3.5 times more water than their mass. Figure 5 shows the effect of the enzymatic treatment parameters on the water absorption of the corn husk fiber.

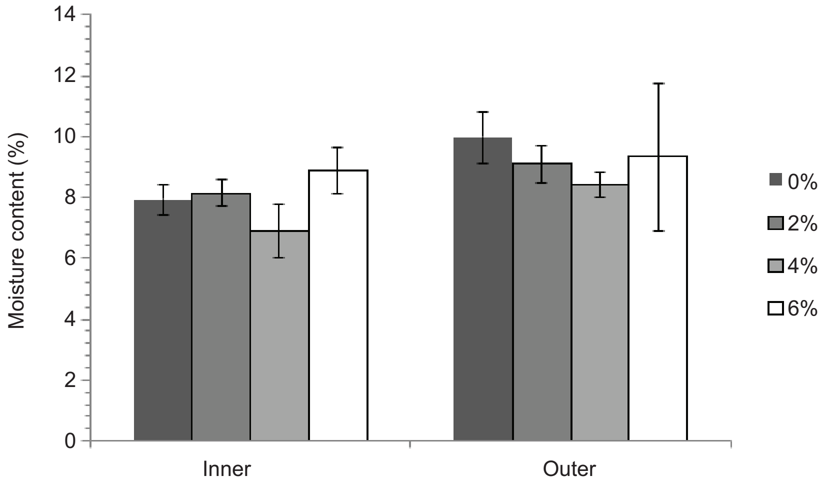

Figure 4. The effect of the enzymatic treatments on the moisture content (%) of the fibers extracted from the inner and outer corn husk leaves. The legend stands for the enzyme concentrations.

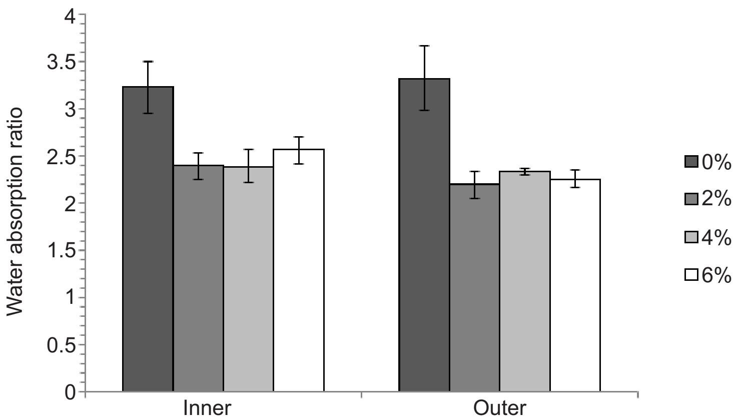

Figure 5. The effect of the enzymatic treatments on the water absorption (%) of the fibers extracted from the inner and outer corn husk leaves. The legend stands for the enzyme concentrations.

Whereas the xylanase treatment strongly affected water absorption ($p=2.55\times10^{-4}$), the husk leaf location did not have a statistically significant effect. The decrease in water absorption can be elucidated by the decrease in the water-absorbent hemicellulose content with degradation due to the bioactivity of the xylanase enzymes. The increase in xylanase concentration absorption to levels higher than 2% did not have a notable effect on water absorption. This finding supports the hypothesis that all available sites of the corn-husk fibers have already been occupied at 2% xylanase ratio.

Fiber mechanical properties

The mechanical properties of the corn husk fibers are shown in Figures 6–9. The highest initial modulus of 280.46 $cN\cdot tex^{-1}$ was attained by the 6%-xylanase-treated inner cornhusk fiber, the peak breaking force, 333.04 cN, by the outer cornhusk fiber untreated with the enzymes, and the greatest breaking tenacity, 9.92 $cN\cdot tex^{-1}$, was achieved by the 6%-xylanase-treated outer cornhusk fibers. The highest elongation-at-break rate was achieved by the untreated inner cornhusk fiber at 13.59%. The attained mechanical properties were higher than those reported by Yılmaz (2013).

The enzyme treatments showed some evidence of affecting the initial moduli ($p= 9.27\times10^{-2}$), whereas the husk location did not ($p=0.48$). The inner and outer corn husk leaf fibers exhibited a general increase in the initial modulus with an increase in enzyme concentration, as shown in Figure 6. Yılmaz (2013) also reported that the enzymatic treatment of corn husk fibers resulted in the production of stiffer fibers.

There was insufficient evidence that the breaking force of the inner and outer fibers was significantly affected by xylanase concentration (p=0.16). On the other hand, the husk location had an immense effect on the breaking force of fibers (p value 2.36×10^{-9}). The coarser outer fibers exhibited greater breaking force than the inner corn husk fibers (Figure 7). The breaking tenacity values were influenced by the husk fiber location (p value 3.56×10^{-2}), but not by the xylanase concentration (p=0.77). The breaking tenacity of the corn husk fibers is close to that of low-density polyethylene fibers.

The elongation at break ratio was affected by the xylanase concentration ($p = 2.9 \times 10^{-7}$), but not by the husk location (p = 0.91). The effect of xylanase concentration differed between the inner fresh and outer mature corn husk fibers ($p = 1.33 \times 10^{-4}$). When the trends in Figure 9 are investigated, a clearer trend can be observed in the inner fresh corn husk fibers, where the elongation decreases with an increase in the xylanase content. Similar to this finding, Yılmaz (2013) reported decreased elongation upon enzyme treatment of corn husk fibers. The decrease in elongation may be attributed to the removal of non-cellulosic components. These components form a network structure among the fibrils, preventing their breaking at high extension rates, similar to elastane.

When the trends in the figures were investigated, a clean trend was observed in the inner cornhusk leaf fibers, whereas it was difficult to detect in the outer corn husk fibers. This may be interpreted as the inner young cornhusk fibers' being more vulnerable to enzymatic treatment

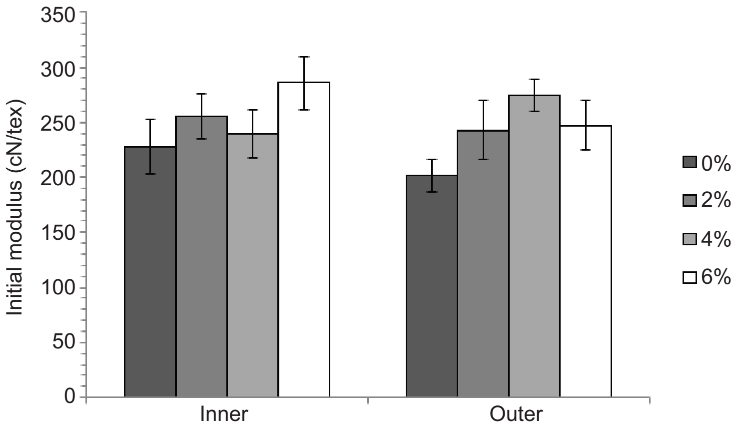

Figure 6. The effect of the enzymatic treatments on the initial modulus (cN/tex) of the fibers extracted from the inner and outer corn husk leaves. The legend stands for the enzyme concentrations.

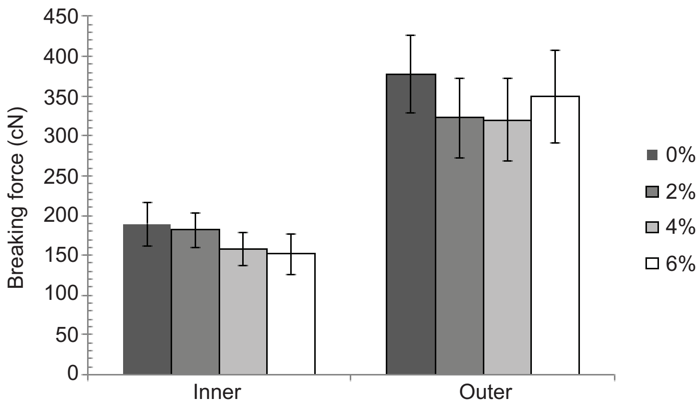

Figure 7. The effect of the enzymatic treatments on the breaking force (cN) of the fibers extracted from the inner and outer corn husk leaves. The legend stands for the enzyme concentrations.

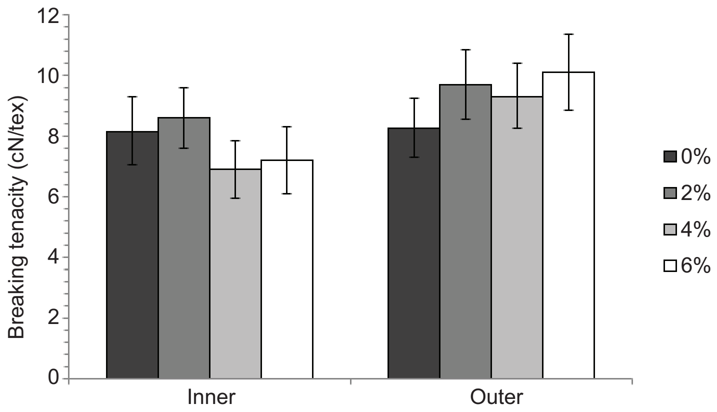

Figure 8. The effect of the enzymatic treatments on the breaking tenacity (cN/tex) of the fibers extracted from the inner and outer corn husk leaves. The legend stands for the enzyme concentrations.

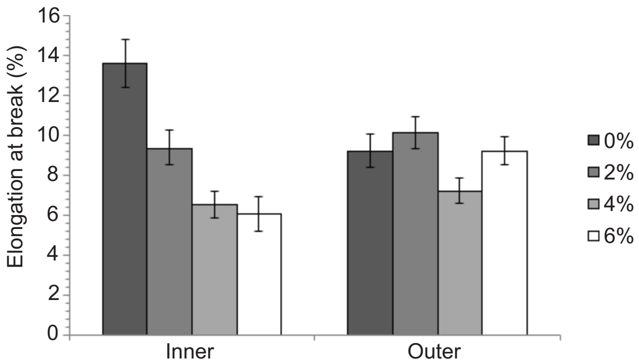

Figure 9. The effect of the enzymatic treatments on the elongation at break (%) of the fibers extracted from the inner and outer corn husk leaves. The legend stands for the enzyme concentrations.

than the outer mature cornhusks. Maturity is associated with lignification. Lignin which is a complex biopolymer that is hard to break might have formed a protective layer on the mature corn husk fibers which hinders enzyme activity.

The studied corn husk fibers exhibited tensile properties comparable to those of conventional natural and manmade fibers. The breaking elongation rates of the corn husk fibers lie between those of cotton and polyester fibers, with values of 7% and 15%, respectively. The initial modulus and breaking tenacity values of the cornhusk fibers were comparable to those of wool fibers (Gupta, 2008). With tensile performance in the range of common fibers, corn husk fibers may be utilized in various application fields.

Thermal analysis

Thermogravimetric analyses (TG) were conducted to scrutinize the thermal degradation characteristics of the inner and surrounding corn husk fibers. The thermal degradation characteristics of plant fibers are important for determining their suitable processing conditions and application areas (Mudoi and Sinha, 2024). The TG, DTG, and DTA curves of the fibers extracted from the inner and outer husk leaves are shown in Figure 10. The decomposition routes of the fibers, which occurred in three stages, were similar. The first stages are related to the dehydration of moisture and are realized with an average mass loss of 9.972%. The second stage corresponds to the depolymerization of cellulose and hemicellulose groups. The decomposition of lignin groups started very slowly in the second decomposition stage. Actual lignin degradation occurs during the third stage. Additionally, the third stage indicates the degradation of residual a-cellulose and ash groups. These reactions are in accordance with the findings of De Rosa et al. (2010).

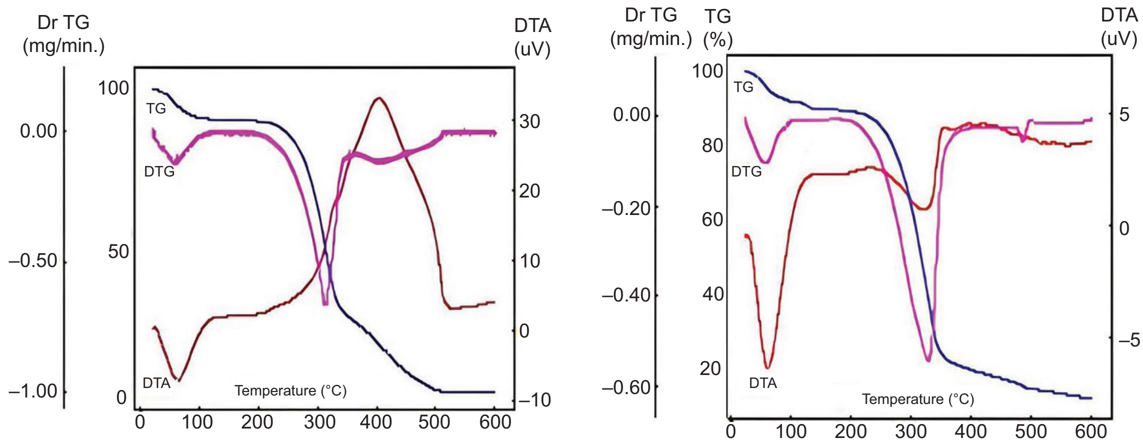

Figure 10. TG, DTG and DTA curves of the inner (left) and outer (right) fibers.

In the first decomposition stage, the mass loss due to the water vaporization of the inner fiber (9.728 %) is lower than that of the outer fibers (10.215%), in accordance with the moisture content analysis. The dehydration reaction of the inner fiber was completed at 105°C; however, this temperature shifted to 134°C for the outer fiber. The removal of excess water adsorbed on the outer fiber takes more time and occurs at partially higher temperatures.

The anhydrous fibers extracted from the inner and outer husk fibers show thermal stability up to 204 and 208°C, respectively. This result agrees with the thermal degradation temperatures of many plant fibers reported in the literature (De Rosa et al., 2010; Mothe & Miranda 2009; Yao et al., 2008; Thomason & Rudeiros-Fernández 2021; Mudoi & Sinha 2024). The thermal stability of the outer fiber was slightly higher than that of the inner fiber, which might be due to the maturity of the outer fibers. The maturation process increases the intermolecular attraction forces of the cellulose fibers. The density of the hydrogen bonds increased. This improves the thermal stability of the cellulose molecules, shifting the thermal degradation to a slightly higher temperature. Furthermore, the presence of a thicker lignin layer surrounding the cellulose in the outer fibers protects the cellulose molecules. Asim et al. reviewed all previous studies on the effect of temperature on the thermal stability and weight loss of natural fibers. In continuation of this study, different polymer composites were produced from natural fibers, and their thermal properties were re-examined. Researchers have emphasized that the thermogravimetric behavior of natural fibers is directly dependent on their chemical composition and the intensity of their intermolecular interactions. As the intermolecular interactions increase, the thermal decomposition shifts to higher temperatures. This has been confirmed for different fibers and polymer composites of the same fiber (Asim et al., 2020). The temperature values corresponding to the thermal stabilities of the selected plant fibers are listed and compared with those of the husk fibers in Table 3.

Table 3. Temperature values which correspond to thermal stabilities of selected plant fibers.

Plant fiber	T (°C)	Reference
Cornhusk	206 (avg)	Present study
Coir	200	Thomason & Rudeiros-Fernández, 2021
Date palm	197	Thomason & Rudeiros-Fernández, 2021
Sugarcane	200	Mothe & Miranda, 2009
Okra	236	Karateke & Yılmaz, 2025
Kenaf	219	Yao et al., 2008
Grewia optiva	200	Mudoi & Sinha, 2024

The cellulose and hemicellulose groups in the inner fibers were not shielded by lignin as effectively as in the case of the outer fibers. Therefore, the inner fibers exhibited lower thermal stability than the outer fibers. The depolymerization of these groups started at 204°C and continued until 345°C. The reaction occurs rapidly, spontaneously, and exothermically in the inner fiber. The total mass loss was 63.3%. Ariffuzaman Khan et al. (2012) reported similar exothermic peaks for the thermal decomposition reaction of coconut husk fibers.

The cellulose and hemicellulose groups in the outer fibers were preserved by lignin groups. Thus, the depolymerization of these groups requires heat. Therefore, an endothermic peak was observed in the DTA curve. The reaction started at 208°C and finished at 355°C. The mass loss was 68.851%, which was higher than that of the inner fiber. The mass loss was relatively high because the lignin surrounding the cellulose and hemicellulose groups as a protective barrier also decomposed.

The peak temperature values of the last decomposition stage, which correspond to the degradation of the residual a-cellulose, lignin groups, and ash, are 404.25 °C and 471.23 °C for the inner and outer fibers, respectively. The reaction completes with 26.265% mass loss for the inner fibers and 11.669% mass loss for the outer fibers. The reason for the lower mass loss for the outer fibers might be that the cellulose in its structure is shielded more effectively with lignin, which decomposes slowly in the whole temperature range, as reported by De Rosa et al. (2010). Similarly, Yılmaz observed greater mass loss in the third thermal degradation step for corn husks, including a lower amount of non-cellulosic materials (2013). This reaction occurs consecutively after the second reaction.

The total mass loss as a result of the three decomposition stages was 99.213% and 90.735% for the inner and outer fibers, respectively. The reason for the greater residual amount for the outer fibers is that they include constituents such as lignin and ash, which have not completed their decomposition in the temperature range studied, as can be seen in Figure 8, where the declining trend of lignin still continues at 600°C, whereas that for the inner fiber finishes at 512°C and follows a horizontal line thereafter. Accordingly, Yılmaz obtained a narrower third decomposition temperature range and greater total thermal decomposition mass loss for corn husk fibers with a lower amount of non-cellulosic materials (2013). All the thermoanalytical results are summarized in Table 4.

Chemical structure (FTIR spectroscopy)

The FTIR spectra of the fibers produced from the inner and outer husk leaves are presented in Figure 11. A wide band in the 3200-3600 cm^{-1} range is due to the axial deformation of the O-H groups (Mothe & Miranda, 2009). This peak appeared at 3325 and 3321 cm^{-1} for the inner and outer fibers, respectively. The peaks at 2877 cm^{-1} (inner fiber) and 2873 cm^{-1} (outer fiber) correspond to C-H stretching. The carbonyl groups of the hemicellulose in the corn husk may give signals at 1633 and 1637 cm^{-1} for the inner and outer fibers, respectively. The peak at 1368 cm^{-1} is attributed to the aromatic ring C-H bending vibration in lignin (Karateke & Yılmaz, 2025). The peak at 1157 cm^{-1} corresponds to C-O-C anti-symmetrical deformation in hemicelluloses

Table 4. The thermoanalytical results obtained from TG, DTA and DTG curves.

Fiber	Temperature range (°C)	DTA peak temperature (°C)	DTG peak temperature (°C)	Mass loss (%)
Inner	21–105	63.23(endo)	58.20	9.728
	204–345	332.97(exo)	313.47	63.300
	345–512	404.25(exo)	412.59	26.265
Outer	24–134	62.37(endo)	56.63	10.215
	208–355	318.60(endo)	328.64	68.851
	355–601	471.93(endo)	486.05	11.669

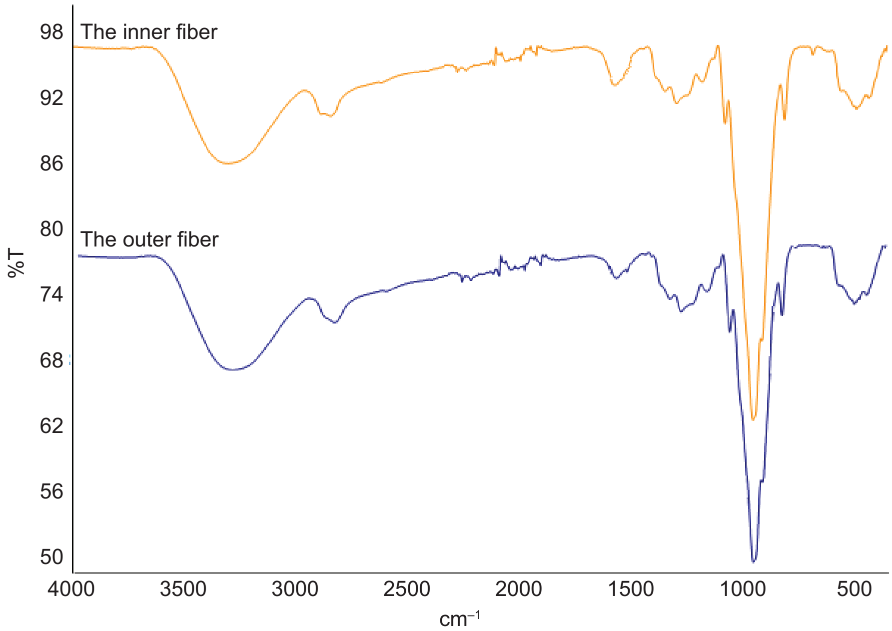

Figure 11. The FTIR spectra of the inner and outer fibers.

and cellulose (Mothe & Miranda, 2009). The intense peak at 1035 cm^{-1} is attributed to the stretching vibrations of C-O and O-H in cellulose. The peak at 894 cm^{-1} is ascribed to b-glycosidic linkages between the monosaccharides (De Rosa et al., 2010).

The physical, mechanical, thermal, and chemical properties of corn husk fibers are comparable to those of common fibers and can be utilized in similar application fields. The diameters obtained in this study are higher than those of conventional fibers used in textiles; therefore, technical uses are more suitable. In the literature, corn husk fibers have been reported for use in composites in the automotive industry, sound insulation, and hygiene product manufacturing. The use of these fibers is sustainable and promising in reducing harmful environmental impacts (Chokshi et al., 2025, Rastogi et al., 2021; Chun et al., 2020).

Conclusions

Fibers were produced from inner and outer corn husk leaves by alkalization succeeded by enzyme treatments at various concentration levels of a xylanase enzyme. The physical, mechanical, thermal and chemical properties of the obtained corn husk fibers were studied. The linear density of the fibers decreased with enzyme treatment. An increase in xylanase concentration led to an increase in fiber stiffness and strength to a certain extent, whereas a further increase in concentration resulted in the deterioration of the mechanical properties. Fibers produced from inner husk leaves are finer and more sensitive to changes in enzyme concentration than those produced from outer leaves. Enzyme treatment resulted in a decrease in water absorption and breaking force and an increase in the initial modulus of corn husk leaves. Fibers extracted from the outer leaves exhibited higher thermal stability, with greater thermal decomposition stage temperatures and lower mass loss, compared to those extracted from the inner husk leaves. Fibers from inner husk leaves contain less noncellulosic content than those from outer husk leaves. The physical characteristics of the obtained fibers were linear density (19.9–42 tex), moisture content (6.89–9.98%), and water absorption (2.0–3.5 g/g). The highest initial modulus (280.46 cN tex^{-1}) was attained by the 6%-xylanase treated inner cornhusk fiber, the highest breaking force (333.04 cN) was obtained for the outer cornhusk fiber untreated with enzymes, the highest breaking tenacity (9.92 cN tex^{-1}) was obtained for the 6% xylanase treated outer cornhusk fiber, and the highest elongation-at-break rate (13.59%) was obtained for the untreated inner cornhusk fiber.

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Conflict of interest

The authors declare no conflict of interest.

Data availability statement

Data can be obtained from the corresponding author upon a reasonable request.

Ethics committee approval

Ethics committee approval is not required for this study.

Authors' contribution statement

The authors acknowledge their contributions to this paper as follows: **Study conception and design**: N.D.Y,; **Data collection**: N.D.Y, G.K.Ç,; **Analysis and interpretation of results**: N.D.Y, G.K.Ç,; **Manuscript draft preparation**: N.D.Y, G.K.Ç,; Both of authors reviewed the results and approved the final version of the manuscript.

Use of Artificial Intelligence

No artificial intelligence-based tools or applications were used in the preparation of this study. The entire content of the study was produced by the author(s) in accordance with scientific research methods and academic ethical principles.

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