Effect of xylanase enzyme on mechanical properties of fibres extracted from undried and dried corn husks

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Fibres have been extracted from corn husks in undried and dried forms by an alkalization treatment followed by enzymatic process at different concentrations of xylanase enzyme. The effects of drying process and xylanase enzyme concentration on the mechanical and textile properties of corn husk fibres have been investigated. The linear density of extracted fibres decreases with the increase in enzyme concentration and drying. Increasing enzyme concentration increases the breaking tenacity and initial modulus up to a point and then decreases them with any further increase in concentration. Fibres obtained from dried husks show dull colour as compared to those obtained from undried husks. No negative effect of drying process is observed on the performance of corn husk fibres. The highest tensile performance and fineness values are obtained from fibres of dried husks, treated with 0.4% Pentopan® mono BG enzyme, with a breaking tenacity of 9.44 cN/tex, initial modulus value of 282 cN/tex, and a fineness of 21.6 tex.

Keywords: Alkalization treatment, Corn husk, Drying, Fibre, Xylanase enzyme

1 Introduction

Growing awareness of global environmental developments such as depletion of petroleum resources, shortage of landfill spaces, and emissions during incineration, caused by the industrial activities based on conventional synthetic materials has turned the eyes of governments, academia and civil entities once again towards natural materials. The new-generation products produced from natural materials should be based on renewable resources, be recyclable or biodegradable, and should not cause harmful emissions during production, service life or after disposal. They should also sustain their characteristics during use and compete with conventional materials in terms of performance and price1.

Accordingly, conventional natural fibres such as flax, sisal, jute, kenaf and hemp have been the subject of renewed interest since nineties1-7, while unconventional fibre sources such as agricultural byproducts have not been an important research topic until the beginning of the third millennium8-12. It is an undeniable fact that utilizing agricultural byproducts for fibre production would provide significant value addition for the agricultural community. Rice and wheat straws, rice and corn husks, corn and okra stalks and banana bunches can be given as examples of agricultural products as sources of fibres1,9-17.

Very limited research effort has been devoted to corn husks as a fibre resource15-17. Among these few studies, Reddy and Yang15 extracted corn husk fibres via alkalization treatment at a NaOH concentration of 20 g/L for 60 min duration at 95°C. An enzymatic treatment with 5% pulpzyme and cellulase at 50°C for 60 min succeeded the alkalization treatment. The fibres were reported to have moderate strength, high elongation, good pliability and high moisture regain.

Yilmaz16 extracted fibres from corn husks by alkalization at concentrations varying from 2.5 g/L to 10 g/L NaOH for different durations between 30 min and 120 min at boiling temperature. The highest tensile performance was obtained in 5 - 10 g/L NaOH concentration and 60 - 90 min duration ranges. She reported a decrease in moisture content with increasing alkali concentrations and durations.

Yilmaz17 extracted fibres from corn husks by water retting and alkalization processes followed by enzymatic treatments. She reported that enzymatic treatments result in increase in initial moduli and breaking tenacity for alkalized fibres, while resulting in loss of breaking tenacity and elongation in water retted fibres. No significant effect of enzymatic treatment duration was obtained on the mechanical

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properties of corn husk fibres. Alkalized fibres had higher elongation and lower stiffness values compared to water retted ones.

As few studies which include enzymatic treatment of corn husk fibres have been reported in the academic literature\textsuperscript{15-17}, no systematic investigation of the relationship between the enzyme concentration and the characteristics of corn husks fibres is found. Furthermore, no studies have been found that investigate the effects of drying process on the fibre properties, even though drying is a very important process in terms of storing of corn husks.

Despite the fact that corn stock is growing in vast geographical regions, it is not available throughout the year, as being a seasonal plant. Additionally, corn husks start degrading immediately once they have been pulled out from the plant, unless they are dried. This fact brings out the storing problem. Furthermore, drying would lead to a substantial decrease in cornhusk weight, which is a positive fact for transportation. This situation makes the study of the effects of corn husk drying on the fibre characteristics necessary. Accordingly, this study has been carried out to investigate if corn husks can be dried safely without affecting the properties of extracted corn husk fibres negatively.

In this study, fibres have been extracted from corn husks in undried and dried form by an alkalization process, followed by enzymatic treatments at different concentrations. The effects of drying process and xylanase enzyme concentration on the physical and mechanical properties of corn husk fibres have been investigated.

2 Materials and Methods

2.1 Materials

Corn husks were collected from a local farmer’s market in Denizli Province of Turkey in October month. A xylanase enzyme (Pentopan\textregistered Mono BG) was supplied by Novozymes A/S, Denmark. Xylanase enzymes break the covalent bond between lignin and cellulose and depolymerize hemicelluloses in the fibre\textsuperscript{15}. The endohydrolysis of 1,4-\beta-D-xylosidic linkages is catalyzed by Pentopan\textregistered Mono BG\textsuperscript{18}.

2.2 Methods

2.2.1 Sample Preparation

A portion of corn husk fibres was alkalized immediately in undried form. Another portion of corn husk fibres was dried under ambient conditions for several days until they were fully dry before going through alkalization. Undried and dried corn husks were subjected to alkalization treatment at a NaOH concentration of 7.5 g/L for 20 min under boiling temperature in distilled water. The alkalization treatment was carried out at 1:20 liquor ratio. Alkalization was followed by rinsing five times, neutralizing with 10\% acetic acid solution, rinsing and drying under ambient conditions.

Enzyme treatments were carried out in beakers at 50\(^\circ\)C with a liquor ratio of 1:50 in distilled water. Enzyme concentrations were calculated based on the percentage ratio of enzyme mass to the fibre mass. All enzyme treatments were carried out concurrently to avoid any effects of uncontrollable variables. The fibres were left to dry under ambient conditions after being rinsed first in boiling water and then in tap water following the enzymatic treatments.

A 2-factorial experimental design was adopted for the current study with two levels of corn husk forms, namely undried and dried, and four levels of xylanase enzyme concentration such as 0, 0.2, 0.4 and 0.6\% based on fibre weight.

2.2.2 Characterization

Linear density, mechanical properties, moisture content, crimp and morphological structure of the extracted fibres were obtained. Samples were subjected to conditioning at 21\(^\circ\)C and 65\% relative humidity for at least 24 h prior to characterization processes.

Linear density of corn husk fibres was determined according to ASTM D1577-07 standard test methods for linear density of textile fibres\textsuperscript{19}. The fibre lengths were measured using a ruler (mm scale) and the weight was measured to the 0.1 mg accuracy. At least 13 fibres of each batch were measured.

Fibre initial modulus, breaking tenacity and elongation-at-break values were measured according to ASTM D3822 standard test method for single textile fibres\textsuperscript{20}. At least 20 specimens of each fibre set were measured at a crosshead speed of 15 mm/min. Gauge length was set at 2.54 cm. A 10 N load cell was used. The measurement device was Tinius Olsen H10KT(R) Tester with QMat for Textiles(R) software.

The moisture content in the fibres was measured according to ASTM D2495 - 07 standard test method for moisture in cotton by oven-drying\textsuperscript{21}. Fibre bundles which were conditioned for at least 24 h were weighed to the 0.1 mg digit, subjected to oven drying at 105\(^\circ\)C for 16 h, cooled in a desiccator and weighed.
The moisture content was calculated according to the following formula:

$$MC = \frac{OS - OD}{OS} \times 100$$

where $MC$ denotes the moisture content, $OS$ stands for the mass of original (moist) sample, and $OD$ represents the mass of oven dry sample. Three specimens of each sample batch were measured.

The crimp of the fibres was measured by measuring a 10 cm long fibre in its relaxed form to the mm scale and then in its extended form when all waviness of the fibre disappears. The crimp was calculated according to the following formula:

$$C = \frac{EL - RL}{EL} \times 100$$

where $C$ denotes fibre crimp, $EL$ stands for the extended length of the sample, and $RL$ represents the relaxed length of oven dry sample. Ten specimens of each sample batch were measured. The findings of aforementioned characterizations were subjected to 2-factor analysis of variance with $\alpha$ at 0.05 significance level.

A SOIF model microscope was used to investigate the morphological structure of individual fibres. Longitudinal and cross-section images of fibres were recorded by using a Moticam 352 model camera with a Motic Images plus 2.0 ML software.

3 Results and Discussion

The effects of enzymatic treatment on physical and mechanical characteristics of fibres which were extracted from undried and dried corn husks have been investigated and the findings are shown in Table 1.

3.1 Fibre Yield

Upon drying, corn husks show 72% decrease in their initial weight. To obtain 1 g of fibre, 12.66 – 15.43 g of undried corn husks or 3.69 – 4.07 g of dried corn husks are needed. In other words, the mean fibre yield for undried cornhusks is 7.14%, whereas that for dried husks is approximately 25.3%. When the initial undried weight of the dried cornhusks is considered, the yield average becomes 7.13%. This means that the drying process does not cause a difference in the fibre yield, when the starting corn husk material is taken into account.

3.2 Fibre Linear Density

The enzymatic treatment and drying process have affected the fibre linear density ($p$ values $1.0 \times 10^{-2}$ and $3.64 \times 10^{-4}$ respectively) significantly. While linear density values of enzyme treated fibres are consistently lower than their untreated counterparts, a gradual trend cannot be obtained among fibres which have been treated with different concentrations of enzymes. On the other hand, linear density values of dried fibres are lower than undried fibres. Drying might have a maturing effect on fibres such as cotton fibres. This area needs further investigation (Fig. 1). The average lowest linear density (21.6 tex) is achieved at a xylanase concentration of 0.4% for fibre sample obtained from dried corn husks.

3.3 Fibre Moisture Content

A statistically significant effect of drying or enzymatic treatment cannot be obtained with the adopted experimental design ($p$ 0.50). Produced fibre samples give moisture contents which range 7.83 – 11.9% (Fig. 1). Corn husk fibres contain greater amount of moisture compared to cotton, the moisture content of which lies between 6% and 8%.

3.4 Fibre Mechanical Properties

The breaking tenacity values are significantly affected by drying process and enzymatic treatments ($p$ values were $1.96 \times 10^{-2}$ and $5.77 \times 10^{-5}$ respectively). The enzymatic treatment has a stronger effect as evidenced by the $p$ value obtained by the statistical analysis. A more or less similar trend is observed with

<table>
<thead>
<tr>
<th>Corn husks</th>
<th>Xylanase concentration, %</th>
<th>Linear density tex</th>
<th>Moisture content %</th>
<th>Crimp, %</th>
<th>Breaking tenacity cN/tex</th>
<th>Initial modulus cN/tex</th>
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<td>0.0</td>
<td>31.5 ± 5.3</td>
<td>11.9 ± 7.3</td>
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<td>0.2</td>
<td>29.1 ± 7.2</td>
<td>9.9 ± 2.8</td>
<td>7.4 ± 4.6</td>
<td>7.8 ± 3.8</td>
<td>245 ± 63</td>
<td></td>
</tr>
<tr>
<td>0.4</td>
<td>26.4 ± 8.3</td>
<td>8.2 ± 1.1</td>
<td>5.8 ± 3.5</td>
<td>8.1 ± 5.3</td>
<td>278 ± 109</td>
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<td>0.6</td>
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<td>8.9 ± 0.5</td>
<td>5.4 ± 3.0</td>
<td>7.8 ± 4.5</td>
<td>221 ± 83</td>
<td></td>
</tr>
<tr>
<td>Dried</td>
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</tr>
<tr>
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<td>27.4 ± 6.0</td>
<td>10.1 ± 1.4</td>
<td>7.0 ± 3.2</td>
<td>5.7 ± 3.1</td>
<td>198 ± 56</td>
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<td>8.3 ± 0.9</td>
<td>5.8 ± 3.2</td>
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<td>9.4 ± 4.6</td>
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<td>7.8 ± 2.6</td>
<td>6.0 ± 2.1</td>
<td>7.8 ± 3.6</td>
<td>248 ± 75</td>
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</table>
increasing concentration levels of the xylanase enzyme for fibres obtained from undried and dried corn husks (Fig. 1). The enzyme treated fibres show greater breaking tenacity as compared to the untreated fibres. With increasing xylanase concentration, the breaking tenacity value is increased up to a point and then decreased when the concentration level is exceeded 0.4%. The extra-cellulosic material in the fibres cannot contribute to load sharing well, as it has lower strength compared to cellulose. So, its removal by the enzyme enhances breaking tenacity of fibres. Further increase in enzyme concentration leads to decrease in breaking tenacity. This might be because enzyme treatment at higher concentration starts to damage the cellulosic backbone. The highest average breaking tenacity results are achieved by fibres of dried corn husks, treated with 0.4% Pentopan® Mono BG with a value of 9.44 cN/tex. This value is bigger than the greatest value (9.23 cN/tex) achieved by Yilmaz for corn husk fibres extracted by alkalization at 10 g/L NaOH for 90 min.

Initial modulus results (Fig. 1) follow a similar trend to that of breaking tenacity values. Initial modulus behaviour is significantly affected by the drying process and enzymatic treatments (p values $4.64 \times 10^{-3}$ and $2.81 \times 10^{-8}$ respectively). The highest initial average modulus value is achieved at 0.4% xylanase enzyme for fibres obtained from dried corn husks with an average value of 282 cN/tex. This value is followed by the value of fibre sample obtained from undried corn husks, treated at the same enzyme concentration with an average value of 278 cN/tex. These values are more than twice the greatest value achieved by Yilmaz which is 126 cN/tex for corn husk fibres extracted by alkalization at 10 g/L NaOH for 90 min.

The elongation-at-break values of the fibres range between 7.20% and 11.5%. Hence, it can be said that the produced corn husk fibres has slightly higher elongation compared to cotton fibres which break at 5 – 10% elongation.

3.5 Fibre Crimp

Crimp is an important feature of natural fibres which affects fibre cohesion positively. Cohesion is needed during spinning or nonwoven formation. The crimp of produced corn husk fibres varies between 4.48% and 6.28%. Crimp frequency can be given as 0.5-5 crimps/cm. In this sense, corn husk fibres, which may be regarded as leaf fibres, differ from bast fibres which have virtually no crimp at all.
3.6 Fibre Morphology

Similar to other ligno-cellulosic fibres, corn husk fibres consist of many individual fibres held together by extra-cellulosic materials. A lumen takes place in the middle of individual fibres. Corn husk fibres are shaped as flattened cylinders in distorted shapes.

3.7 Fibre Colour

Corn husk fibres extracted in the current study are generally yellowish white. When fibres of undried and dried corn husks are compared, it is found that fibres from un-dried corn husks possess a brighter yellow hue, while dried fibres have a duller creamy colour.

4 Conclusion

It is observed that the linear density of extracted fibres decreases with the increase in enzyme concentration and drying. Increasing enzyme concentration increases the breaking tenacity and initial modulus up to a point and then decreases them with any further increase in concentration. No negative effect of drying process is found on the performance of corn husk fibres. The highest tensile performance and fineness values are obtained from fibres of dried corn husks, treated with 0.4% Pentopan® Mono BG enzyme, with an initial modulus of 282 cN/tex, breaking tenacity value of 9.44 cN/tex, and a fineness of 21.6 tex.

Acknowledgement

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References