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# Effect of Endo-1,4-β-Xylanase Enzyme on Appearance and Physical Properties of Jute Yarn.

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**ABSTRACT**

In this study, jute yarns were treated with endo-1,4-β-xylanase enzyme in different concentrations and times. The results showed an increase in whiteness index and a decrease in yellowness index. However, at high concentration of xylanase a slight decrease in whiteness index and gradual increase in yellowness index were observed. An improvement in the reflectance characteristics of jute fibers was observed as a result of xylanase treatment. The SEM micrographs showed that xylanase treatment leads to a cleaner surface. Furthermore, bio-treated yarns exhibited lower tensile strength than untreated yarn. Fourier transform infrared spectroscopy (FTIR) was used to analyze the effect of xylanase pretreatment on chemical composition of jute fibers. Then, untreated and bio-treated yarns were dyed with reactive dyes. The Color strength values of xylanase treated dyed yarns were higher than untreated dyed yarn.

**Keywords:** Xylanase; Jute fabric, FT-IR spectroscopy; Color spectroscopy; ; Reactive dyes

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## INTRODUCTION

Jute fiber is composed of lignin 11.4-12%, alpha-cellulose 58-63% and hemi-cellulose 21-24% [1, 2]. Chemical scouring solutions attack non-specifically on cellulosic material of fiber while xylanase enzyme specifically degrades the hemicellulosic impurities and efficiently leads to their removal [3].

Xylanases, the xylan hydrolyzing enzymes, can be defined as (EC 3.2.1.55), (EC 3.1.1.6), (EC 3.2.1.8) and (EC 3.2.1.37) [4, 5]. Xylanase (EC 3.2.1.8) degrades the linear polysaccharide beta-1,4-xylan into xylose, [6] thus breaking down hemicellulose. In other words, xylose is the major end product with traces of xylobiose and xylotriose at the beginning of hydrolysis [7].

Enzymatic treatment leads to a significant increase in softness, which is desirable for coarse jute fabric. Furthermore, the removal of hemi-cellulose (the cementing material that bind the ultimate fibers of jute) causes to improve in filamentation and fineness [8].

On the other hand, the pore volume in jute fibers increases by treatment with xylanase enzyme because it can hydrolyze polysaccharide chains and thus reduce cohesion between ultimates with simultaneous proliferation of pores. Moreover, experiments have shown that xylanase pre-treatment can facilitate bleaching [9]. This enzyme is also used for bio-scouring, for instance, alkalo-thermostable xylanase from *Bacillus pumilus* ASH can increase the whiteness and brightness of fabric and decrease the yellowness [10].

In this study the possibility of applying endo-1,4-β-Xylanase from Trichoderma longibrachiatum in acidic pH to jute fiber is evaluated. The aim of present work is to determine whether xylanase enzyme treatment can influence physical appearance and chemical functional groups of the fiber. In this regard, different enzyme concentrations and reaction times were used to compare the efficiency of enzyme.

## EXPERIMENTAL

### Materials

Commercially available tossa jute yarn with a yarn count of 4520 dtex was used throughout the study. Reactive Yellow 176 manufactured by Nordex International D.Z.E Dye Co. in UK was used as a cold reactive dye. Levafix Blue, Blue 29, was purchased from DyStar, Singapore. Buffers were supplied by Scharlau, Spain. The enzyme used in this study is described in Table 1. All chemicals and enzymes were used without any purification.

**Table 1: Xylanase enzyme and its properties.**

| Enzyme | Source | EC number | Opti. temp (°C) for activity | Opti. pH for activity | Activity units/mg | Company cat. number |
|---|---|---|---|---|---|---|
| Endo-1,4-β-xylanase | Trichoderma longibrachiatum | 3.2.1.8 | 30 | 4.5 | 1.0 | Sigma X2629 |

### Enzymatic treatment

Enzymatic treatment of jute yarn was performed for 1-3 h, 40 RPM and liquid ratio 10:1. Xylanase enzyme was applied in its optimum pH and temperature conditions (Table 1). Finally, enzyme inactivation occurred in water bath at a temperature of 98°C.

### Dyeing

The dye bath was prepared by 2% of levafixblue and reactive yellow 176 separately with 80 g/L NaCl salt in a liquor ratio of 30:1. The sample was entered in the dye bath at 40 °C and dyeing was continued at this temperature for 30 min. Then, 10 g/L soda ash was added to the dye bath and the system was kept at that

temperature for 30 min. Afterwards, the dyed sample was rinsed thoroughly in cold and hot water and then dried in ambient temperature.

**Color spectroscopy analysis**

Color strength (K/S) was calculated from the sample reflectance (R):

$$K/S = (1 - R)^2 / 2R \quad (1)$$

The reflectance (R) of dyed sample was measured on a X-Rite Color Eye 7000 A spectrophotometer measurement system (Grand Rapids, MI, USA), at the wavelength of minimum reflectance, under CIE Illumination D65 and d/10° illumination/observation. The whiteness index (WI) and yellowness index (YI) were calculated by the Hunter Lab-scale and ASTM E313-73, respectively.

$$WI=100 - \sqrt{[(100-L)^2 + a^2 + b^2]} \quad (2)$$
$$YI=100 \times [1-(0.847Z/Y)] \quad (3)$$

**Mechanical properties**

Mechanical properties were measured through the material tensile tests (Testometric materials testing machines winTest™ Analysis, UK) according to ASTM D2256-97, with an effective specimen length of 250 mm (clamped distance), a test speed of 300 mm/min, under pretension of 0.5% and at room temperature. The mean values from at least five individual samples are given.

**Scanning electron microscopy (SEM)**

The surface morphology of untreated and treated jute fiber samples was examined using Philips Scanning Electron Microscope (Model: XL30) (Eindhoven, The Netherlands) at an operating voltage of 17 kV, using a magnification of 250X. The fiber samples for the SEM study were prepared with gold-palladium alloy coating.

## RESULTS AND DISCUSSION

**Color spectroscopy analysis**

It is obvious from table 2 that whiteness index of jute yarns can be improved by treatment with xylanase. It can be attributed to the removal of impurities. In other words, the xylanase enzyme is effective in cleaving the xylan backbone into smaller oligosaccharides and causes effective removal of hemicellulosic impurities [3]. The whiteness index was increased by xylanase treatment; however, at higher concentration of xylanase and longer treatment time a slight decrease in whiteness index was observed (table 2).

**Table 2: Effect of xylanase pre-treatment on appearance properties of jute yarn.**

| Enzyme Concentration (g/L) | Treatment Time (minutes) | L* | a * | b* | Whiteness Index | Yellowness Index |
|---|---|---|---|---|---|---|
| 0 (untreated) | - | 64.09 | 5.876 | 20.545 | 58.213 | 53.218 |
| 1.5 | 60 | 63.9 | 4.882 | 16.939 | 59.823 | 45.223 |
| 1.5 | 120 | 63.91 | 5.196 | 18.444 | 59.138 | 48.594 |
| 1.5 | 180 | 63.6 | 5.718 | 17.95 | 59.01 | 48.437 |
| 3 | 60 | 64.15 | 5.981 | 19.125 | 58.926 | 50.717 |
| 3 | 120 | 64.68 | 5.26 | 18.746 | 59.666 | 48.797 |
| 3 | 180 | 63.34 | 5.833 | 18.524 | 58.51 | 49.817 |

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Due to presence of hemicellulose and lignin, jute is more yellowish than cotton [15]. Lignin is linked to xylan by ether bonds [16] or ester linkages [17]. Although xylanases do not affect lignin, they can weaken the hemicelulose/lignin matrix and facilitate lignin removal when xylanase hydrolyses the hemicelluose. Thus, the yellowness index of jute fiber showed a significant decrease from 53.218 to 45.223 after xylanase treatment (table 2).

Figure 1 shows the reflectance characteristics of xylanse treated jute fibers. The reflectance values for xylanase treated samples is slightly higher than untreated sample. Because treatment with xylanase enzyme leads to hydrolysis the hemicellulose which makes the surface of the fibers uniform or smooth.

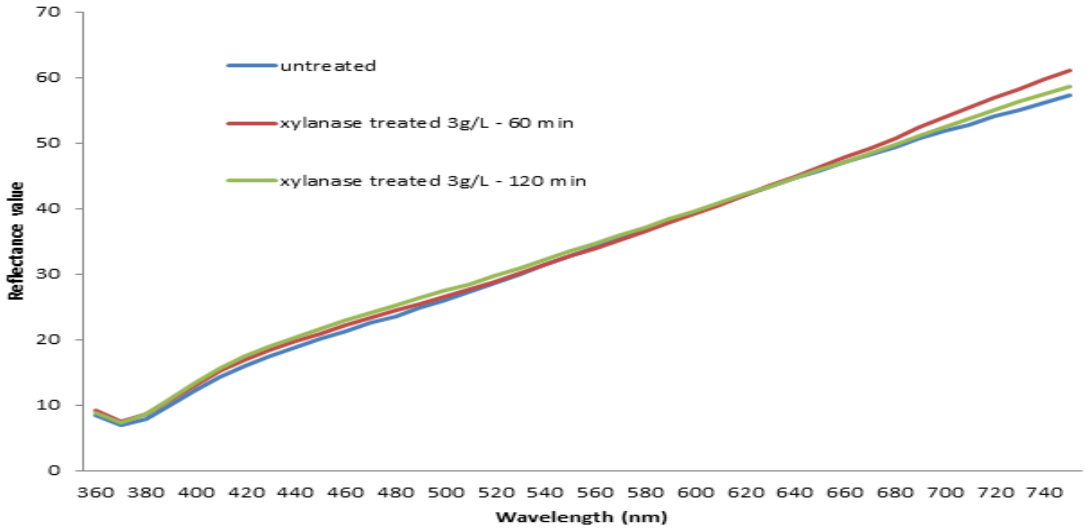


**Figure 1: Reflectance characteristic of untreated and xylanase treated jute fibers.**

**Effect of enzymatic treatment on dyeing of jute yarn with reactive dyes**

The Color strength (K/S) results is shown in figure 2. It demonstrates that K/S values of xylanase treated dyed yarns are higher than untreated dyed yarn. Xylanase improves the fibers' hydration (swelling) and promotes the internal fibrillation and delamination of fiber [18]. Hydrolysis of the xylan influences the fiber swelling [19] which leads to an increase in accessible area. Therefore, xylanase treated fibers have higher absorptive capacity and are more reactive to chemical reagents/dyes [12]. It also can be seen from table 3 that prolonging bio-pretreatment time increases the dye uptake.

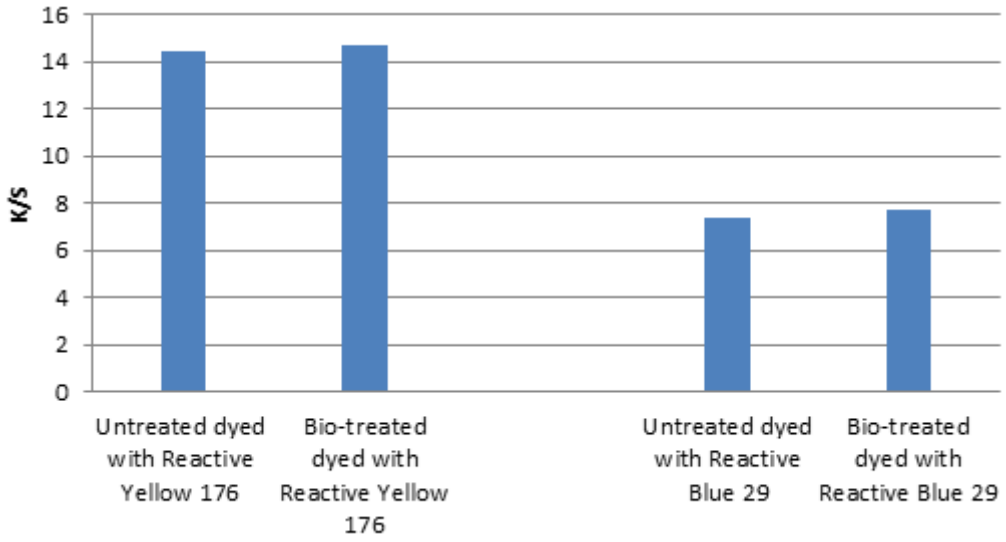


**Figure 2: Dye strength of reactive dyes on pre-treated jute yarns with 3 g/L xylanase for 60 minutes.**

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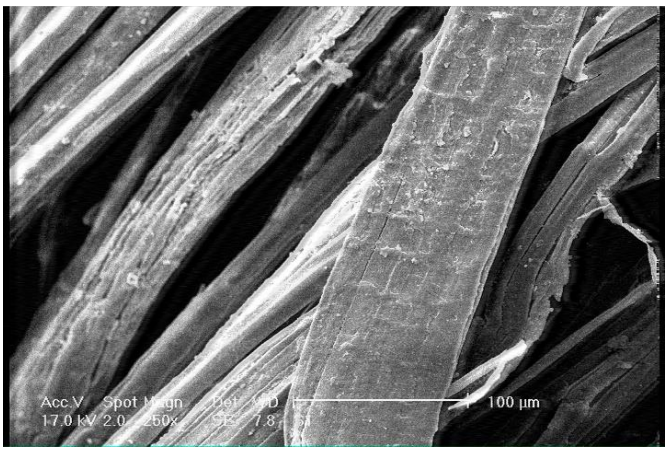
**Table 3: Effect of enzyme concentration and pre-treatment time on sorption of Reactive Yellow 176.**

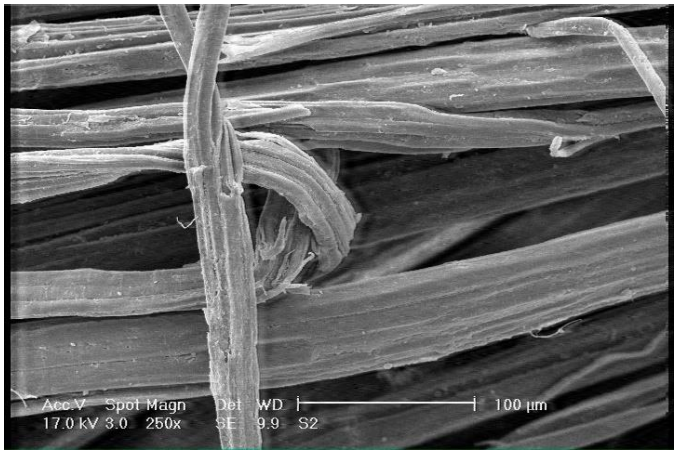
| Enzyme Concentration (g/L) | Treatment Time (minutes) | L* | a * | b* | K/S |
|---|---|---|---|---|---|
| 0 (untreated) | - | 55.064 | 8.899 | 46.424 | 14.410 |
| 1.5 | 60 | 57.073 | 9.661 | 49.555 | 14.685 |
| 1.5 | 180 | 54.844 | 9.143 | 48.031 | 15.631 |
| 3 | 60 | 56.237 | 10.123 | 49.322 | 14.729 |
| 3 | 180 | 58.352 | 9.944 | 52.166 | 14.858 |

**Scanning electron microscopy**

In order to understand the effect of xylanase treatment on appearance of fibers, the fiber surface morphology of the untreated and xylanse treated fibers were studied by SEM. Changes on the surface of the xylanase treated fibers were shown by SEM as a result of xylan hydrolysis (Fig. 3). It can be seen that xylanase treatment leads to a cleaner surface. Although slight fibrillation was observed, xylanase treatment can be safe and quite harmless because there are no severe damage in the surface of fibers.




**(a)**




**(b)**

**Figure 3: SEM micrographs of (a) untreated jute. (b) bio-treated jute fibers with 3 g/L xylanase.**

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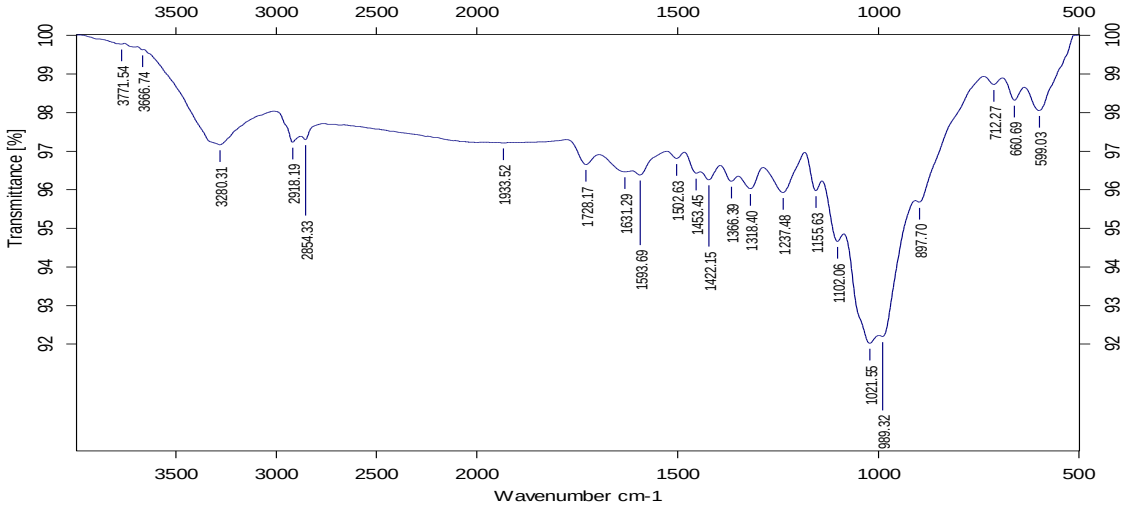
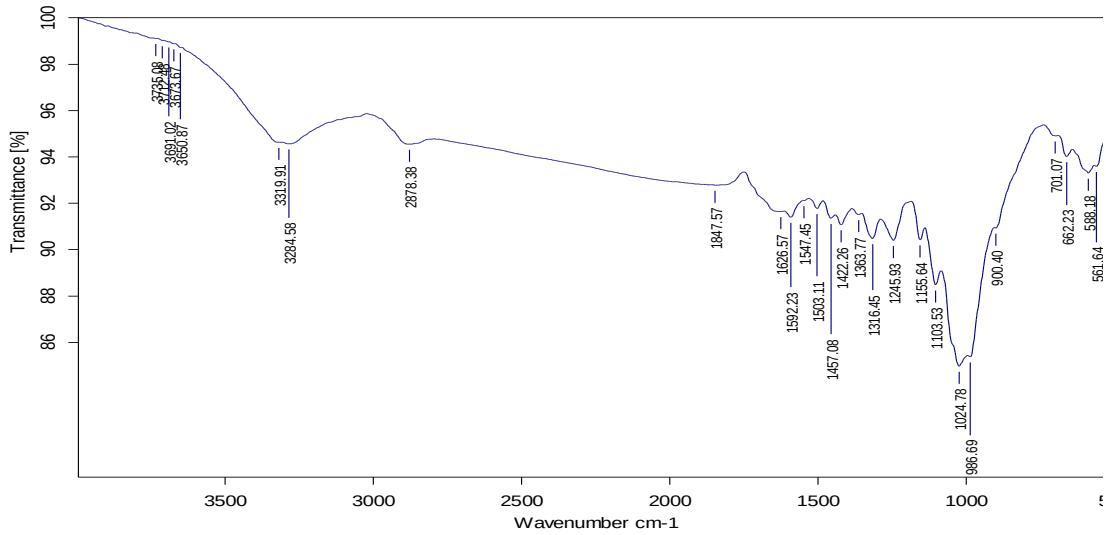
**ATR-FTIR analysis**

The purpose of applying FT-IR is to evaluate the change of surface composition of the fibers after xylanase treatment. Infrared spectra of the untreated and xylanase treated jute fibers are shown in Figure 4.

Almost the same absorption peaks as shown in the untreated jute fibers were observed in the spectrum of the xylanase treated jute fibers. This indicated that the structure of cellulose has not been damaged after xylanase treatment. However, the disappearance of the absorption peak at 1728 $cm^{-1}$ indicated the hemicellulose could be partially removed by xylanase treatment. Because the absorbance at 1728 $cm^{-1}$ is related to the acetyl and uronic ester groups of hemicelluloses [20].




**(a) Untreated jute**




**(b) Xylanase treated jute**

**Figure 4: The FT-IR spectra of (a) Untreated jute (b) Xylanase treated jute.**

**Mechanical properties**

Table 6 shows that enzymatic treatment of jute fibers leads to a decrease in strength of the jute yarns. The cause of the strength loss of enzyme-treated jute yarn is mainly the partial surface hydrolysis of the fibers. Hemi-cellulose (cementing material binding the fiber bundles together) is sensitive to xylanase enzyme

with a deterioration effect. Hence, xylanase pre-treatment can loosen the cement agent in the fiber matrix and lead to a decrease in strength.

**Table 6: Effect of xylanase treatment on mechanical properties of jute yarns.**

| Xylanase concentration (g/L) | Treatment time (minutes) | Initial Modulus (g/dtex) | Force at Rupture (kgf) | Strain at Rupture (%) |
|---|---|---|---|---|
| 0 (Blank) | 0 | 46.018 | 5.748 | 3.620 |
| 1.5 | 60 | 28.922 | 4.292 | 4.015 |
| 1.5 | 120 | 28.139 | 3.792 | 4.199 |
| 1.5 | 180 | 26.114 | 3.775 | 4.075 |
| 3 | 60 | 29.915 | 4.251 | 4.461 |
| 3 | 120 | 25.290 | 3.621 | 4.215 |
| 3 | 180 | 25.286 | 3.481 | 4.119 |

## CONCLUSION

In this study, xylanase enzyme were applied on jute yarn. After bio-pretreatment, some changes were found in carbonyl group of hemi-cellulose. The band near 1728 $cm^{-1}$ disappeared after xylanase pretreatments. Although xylanase treatment led to an increase in whiteness index of jute yarn, at high concentration of xylanase a slight decrease in whiteness index were observed. Enzymatic treatment of the jute fibers led to a decrease in strength of the jute yarns. The K/S values of xylanase treated dyed yarns were higher than untreated dyed yarn. Meanwhile, prolonging bio-pretreatment time led to an increase in the dye uptake.

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