

# **Effect of enzymatic bio-scouring on the dyeability, physicochemical and mechanical properties of jute fabrics**

**Mohammad Mahbubul Hassan<sup>1,\*</sup> Khaled Saifullah<sup>2</sup>**

*<sup>1</sup> Food & Biobased Products Group, AgResearch Limited, Cnr Springs Road and Gerald Street, Lincoln, Christchurch 7647, New Zealand.*

*<sup>2</sup> Jute Processing Group, Bangladesh Jute Research Institute, Manik Miah Avenue, Dhaka-1207, Bangladesh.*

## **Abstract**

Scouring is necessary for the loom state jute fabrics in order to make them suitable for dyeing and finishing by improving their wettability by removing non-cellulosic matters from the fibre. Scouring is carried out at the boil with a cocktail of caustic soda, detergent, and wetting agent, and the process is energy-intensive. In this work, bio-scouring of jute fabrics was carried out using four kinds of enzymes, Esperase 4.0T (protease), xylanase, and Alcalase 2.5 L (alkaline protease) in a combination with cellulase. The performance of scouring of jute fabrics scoured with various enzymes was evaluated by measuring their whiteness index, hydrophilicity, wettability, and dyeability, and was compared with jute fabrics scoured by the traditional alkali scouring method. The effect of bio-scouring on the weight loss and tensile strength loss was assessed. Of the enzymes investigated, the combined treatment with 50/50 mixture of Alcalase and cellulase provided the best performance in terms of improvement in whiteness index, hydrophilicity, wettability, and dyeability, but also caused the highest loss in

---

\* Corresponding author. Tel.: +64 3 3 321 8755; fax: +64 3 321 8811  
E-mail: [Mahbubul.hassan@agresearch.co.nz](mailto:Mahbubul.hassan@agresearch.co.nz) (M. M. Hassan)

tensile strength. The conventional alkali scouring caused higher weight and tensile strength loss compared to enzymatic scouring treatments investigated, but produced poor whiteness index and also poor colour strength when the treated jute fabrics were dyed with a direct dye and two reactive dyes. The developed treatment could be used in industry as an eco-friendly bio-scouring process.

**Keywords:** jute fibre; bio-scouring; delignification; dyeability; tensile strength

## **Introduction**

Jute, a natural lignocellulosic fibre, is known as the ‘golden fibre’ in Bangladesh, one of the largest jute fibre producing countries in the world. In the 1970s, jute industry was the largest export-earning sector in Bangladesh, which is now replaced by the textile and apparel industries. The demand for jute in the world market severely declined in the past decades as it lost its market to cheaper synthetic fibres. However, due to the recent change in consumer choice to bio-based products, there is optimism that jute may recover its past glory and will be a major textile fibre in the near future. Jute fibres are mainly used in the manufacturing of sacking and coarse cloths, carpets, and carpet backings. A small portion of jute fibres is used in apparel making fabrics, usually blended with cotton fibres that provides cotton fabrics high moisture absorbency as jute is one of the highest hygroscopic natural fibres [1].

Jute fibre falls under the bast fibre category as the fibres are collected from the bast or skin of the plant. The jute fibres are located between the epidermises and an inner woody core of the jute plant. Within the stem, there are a number of fibre bundles, each containing individual fibre cells or filaments, which are made up of cellulose and hemicellulose. The filaments are bonded together by a matrix made of lignin and pectin. The pectin is removed

during the retting process to separate individual fibres from fibre bundles. A single jute fibre consists of many cells averaging 2 to 6 mm in length and they are called 'ultimate' cells. These cells are bonded together by lignin, which protects the fibre from biological attacks and provide stiffness. Jute fibres are composed of 59–72%  $\alpha$ -cellulose, 12–15.9% hemicelluloses, 11.8–14.2% lignin, 0.2–0.5% pectin, 0.3–0.5% waxes, 0.8–1.5% protein and 0.6–1.2% mineral matters and nitrogenous substances [2, 3]. The hemicellulose component is a mixture of pentosan (xylan: 12–14%), polyuronide (4–5%) and contains acetyl groups (3.2–3.5%) [4]. The non-cellulosic parts are considered as impurities that impart an undesirable colour, and hydrophobic waxes hinder their dye absorption during the dyeing stage. The residual batching oils (mainly hydrocarbon oils) that are used to make the fibre soft remain in the loom-state woven fabrics. Some of the jute batching oils used in jute industry, such as kerosene oil, could be environmentally harmful, and their removal is essential. To make jute fibres absorbent and easily wetted with water and or other aqueous solutions, a process called scouring is carried which removes these impurities other than lignin. It is known that alkaline scouring treatment of jute can remove some levels of lignin depending on the reaction conditions [6].

The scouring process is necessary to remove the non-cellulosic matters and batching oils from jute fibre. The success of the subsequent chemical processes such as bleaching, dyeing and or printing, and finishing very much depends on the efficiency of the scouring process. Traditionally scouring is carried out at the boil in an aqueous solution of caustic soda, detergent, and wetting agent, but at milder conditions compared to cotton fabrics. The traditional scouring process causes a high loss of tensile strength of the fibre. Moreover, it produces highly alkaline effluent that needs to be neutralised before discharging to watercourses. A large volume of water and acetic acid is used to neutralise the alkali-scoured fabric. To comply with the increasingly stringent environmental regulations and also to save

energy, water, and chemicals, it is necessary to look for an eco-friendly scouring process. Pectinase has been widely investigated for the bio-scouring of various cellulosic fibres as it is capable of degrading the pectin to low molecular weight water-soluble products without affecting the cellulose [5, 7–9]. The most popular pectinase for the delignification of jute fibres is polygalacturonase [10] and lyase [11]. It was found that bio-scouring of jute not only improved its wettability but also improved the whiteness of the scoured fibres [12, 13]. Lyase catalyses the breakdown of  $\alpha$ -1,4 glycosidic linkage in pectin in jute fibres. Xylanase has been investigated for scouring of ramie stem to liberate the fibres and also in the bleaching of pulp [14, 15]. Protease has been frequently investigated to remove unbound and bound lipids from wool fibre surface to ease polymeric coating and also to make them shrink-resist [16, 17]. Protease has also been investigated for the scouring of cotton fabrics [18] and degumming of silk fabrics [19]. Bio-scouring of jute fabric with cellulase-free xylanase from *Bacillus pumilus* ASH, which is stable at wide pHs and temperatures, has also been investigated [20]. It was found that two hours of incubation with that enzyme at 55 °C improved the whiteness of jute fabrics up to 3.93%. The addition of ethylenediaminetetraacetic acid and Tween 20 to the bio-scouring process slightly improved the whiteness [20]. Protease can degrade proteins but xylanase is capable of degrading beta-1,4-xylan. The alkaline cellulase is used in the laundry detergents to enhance their cleaning activity in the cleaning of cellulosic fabrics [21], but it is also known that lignin has an inhibitory effect on the cellulose enzyme [22]. As jute fibre contains some proteins, it is anticipated that protease enzymes, especially alkaline protease, may enhance scouring of jute by degrading the proteins, and, in combination with cellulase, it may show some synergistic effect by enhancing the overall scouring performance. However, cellulase in combination with protease was never investigated for the bio-scouring of jute fabric.

In this work, scouring of jute fabrics was carried out using xylanase, protease, and alkaline protease in combination with cellulase. The effect of enzymatic treatments on whiteness, hydrophilicity, tensile strength and dyeability of jute fabrics was investigated. The performance of enzymatic bio-scouring treatments was being compared with the traditional alkali scoured jute fabrics to assess their effectiveness and benefits over traditional alkali scouring process.

## **Materials and methods**

### **Materials**

The jute fabric used was a hessian cloth with specifications of 23 ends/cm, 13 picks/cm, and 300 g/m<sup>2</sup>. The jute fabric was supplied by Latif Bawani Jute Mills Ltd (Dhaka, Bangladesh). The enzymes investigated were a proteolytic enzyme (Esperase 4.0 T), an endo-1,4- $\beta$ -D-xylanase (Dyadic Xylanase 2XP), and a combination of alkaline cellulase and an alkaline protease (Alcalase 2.5 L). The protease enzymes were purchased from Novozymes (Norway), and xylanase from Dyadic International Inc. (USA). The alkaline cellulase was purchased from Creative Enzymes (USA). Two reactive dyes, Rifacion Red H-E3B (C.I. Reactive Red 120) and Rifazol Brilliant Orange 3R (C.I. Reactive Orange 18) were supplied by Rifa Industrial Co., Ltd. (Korea). The selected direct dye was Solophenyl Violet 4BLE (200%), supplied by Huntsman Chemicals (USA). Caustic soda, acetic acid, sodium carbonate, and sodium sulphate were purchased from Sigma-Aldrich and were of analytical reagent grade. Sandopan DTC, a non-ionic detergent, Antimussol SF, a defoaming agent, and Hostapal MRN, a non-ionic wetting agent, were procured from Clariant (Singapore) Pty Ltd, Singapore.

## **Enzymatic scouring of jute fabrics**

All treatments were carried out in a Roaches Pyrotec sample dyeing machine (Roaches International Ltd, Birstall, UK) using tap water and 1:30 materials to liquor ratio. The bio-scouring treatment of hessian jute fabric was carried out with 1, 2, 3, and 4% on the weight of fibre (owf) Esperase, xylanase and cellulase/Alcalase (50%/50%) at pHs 9, 6.5 and 8.0, respectively. The treatment conditions (pH, time and temperature) for protease [23], cellulase, xylanase [24] and alkaline protease [25] were selected from published literature as various researchers found that these enzymes exhibited maximal activity at these conditions. The bath was set at the appropriate pH with sodium acetate and acetic acid for pHs 6.5 and 6.7, and HCl/sodium borate for pH 8 and sodium carbonate for 9.0. The required amount of water was taken in the dyeing pot, 0.5 ml/l Hostapal MRN and 1 ml/l Antimussol SF were added, and jute fabric samples were then introduced into the dyeing pot. The temperature of the bath was then raised to 58 °C at 2 °C/min and held for 120 min. After completion of the scouring treatment, the bath was cooled to 45 °C at 2 °C/min and the liquor drained. The samples were washed twice with hot and cold water. The control scouring was carried out with 3 g/l sodium carbonate, 1 g/l Sandopan DTC, and 0.25 g/l Hostapal MRN. All of the scoured fabrics were neutralised. Albelgal POC (a dye levelling agent) was purchased from BASF Chemicals (Germany).

## **Fourier-transform infrared spectroscopy (FTIR)**

Any change in lignin content in the fabric after enzymatic processes was assessed by using a Shimadzu FT-IR (Model Prestige 21, Shimadzu Corporation, Japan) with an attenuated total reflectance (ATR) attachment at a resolution of 4 cm<sup>-1</sup> for 32 scans in the range from 650 to 4000 cm<sup>-1</sup>. The KRS-5 crystal was used to take ATR-FTIR spectra. A qualitative measurement of change in the lignin content of the fabric after the various enzymatic

treatments were carried out by measuring the peak height at 1418, 1457 and 1506  $\text{cm}^{-1}$ . The decrease in peak height indicates the decrease in lignin content of the fibre.

### **Bleaching with hydrogen peroxide**

Bleaching was also carried out in the same machine used for the scouring. All the samples were bleached with 2 % owf hydrogen peroxide (50%). The dye pot was filled with sufficient water and 1 ml/l Sandopan DTC, 0.5 ml/l Hostapal MRN, 1 ml/l Antimussol SF, 1 g/l EDTA, and 6 g/l sodium silicate were added. The solid compounds were pre-dissolved in water before adding to the bath. A jute fabric sample was introduced into dyeing pot and the pH was set to 10 with sodium carbonate. The required quantity of hydrogen peroxide was added and the pH was again adjusted to 10. The temperature was then raised to 98 °C at 2 °C/min and held for 60 min. The bath was then cooled to 45 °C at 2 °C/min, and the samples were cold washed and hot washed with 2 ml/l Basopal PK to destroy residual peroxide. The samples were then cold and hot and again cold washed, neutralised with an acetic acid solution and dried. The samples are now ready for dyeing.

### **Dyeing of jute with reactive dyes**

All dyeings were carried out in the same laboratory dyeing machine that was used for carrying out scouring and bleaching treatments using tap water and materials to liquor ratio 1:30. The dyeing pot was filled with sufficient water, and 0.5 g/l Antimussol SI. The electrolyte used was sodium sulphate at 60 g/l and sodium carbonate as a dye fixing agent at 20 g/l. The machine was then run for 20 min and 1/3 of sodium sulphate was added. The machine was then run for another 10 min and the second 1/3 of the salt was added. The machine was then run again for 10 min and the last 1/3 of sodium sulphate was added. The temperature was then raised to 80 °C (60 °C for the Rifazol Brilliant Orange 3R) and held for

10 min. The required quantity of sodium carbonate was added and held for another 60 min. The dye bath was then cooled to 45 °C, the liquor drained and the dyed samples were rinsed with cold water. The fabric samples were then hot washed with 1 ml/l Sandophan DTC at 60 °C for 15 min and again cold washed and hot washed. The samples were then dried at 60 °C for 30 min.

### **Dyeing of jute with direct dye**

The dyeing with the direct dye was also carried out in the same laboratory dyeing machine using tap water and materials to liquor ratio 1:30. The dyeing pot was filled with sufficient water, dye (2% owf) and 0.2 g/l Albegal POC. The machine was then run for 20 min and 20 g/l sodium sulphate was added as an electrolyte and the machine was run for another 10 min. The temperature was then raised to 98 °C and held for 60 min. The dye bath was then cooled to 45 °C and the liquor was drained and the dyed samples were rinsed with cold water and also hot washed. The samples were then dried at 60 °C for 30 min.

### **Evaluation of fabric properties**

The weight loss, whiteness index and wettability of jute fabrics after bio-scouring and bleaching stages were assessed. The assessment of weight loss of jute fabric during bio-scouring and also during bleaching was carried out by the gravimetric method. The oven dried (at 105 °C) jute fabric sample was weighed before and after the bio-scouring and bleaching processes. The weight loss was calculated according to the following equation:

$$\text{Weight loss} = \frac{W_1 - W_2}{W_1} \times 100 \quad (1)$$

where  $W_1$  and  $W_2$  are the oven dry weight of jute fabric before and after the treatment respectively [25].



The improvement in hydrophilicity of the jute fabrics by enzymatic treatments was assessed by the contact angle measurement. The contact angle was measured by using a Ramé-Hart Contact Angle Measurement Apparatus (Model 190, Ramé-Hart Instrument Co., Succasunna, USA). For each sample, the contact angle was measured at six places and the average contact angle was reported. For each sample, the first measurement was taken immediately after placing the drop of water and then at 15 s interval measurements were taken until 45 s. To assess the bio-scouring performance, the wettability of the fabric was also measured by means of the drop test before and after the bio-scouring process according to the *AATCC Test Method 39-1998: Evaluation of wettability* at room temperature [26]. In this method, a water droplet is placed on the fabric and the time taken between the contact of a water droplet with the fabric and the complete disappearance of the water droplet into the fabric was counted as the wetting time. Average values of five readings were reported here.

The  $L^*$ ,  $a^*$ ,  $b^*$  values were measured by a hand-held X-Rite spectrophotometer (X-Rite International, USA). The assessment of whiteness was then deduced by the following equation [27]:

$$\text{Whiteness index} = 100 - [(100 - L^*)^2 + (a^*)^2 + (b^*)^2]^{0.5} \quad (2)$$

The colour strength of the control scoured and the bio-scoured jute fabric samples dyed with direct and reactive dyes were measured using a Datacolor Spectraflash 600 reflectance spectrophotometer, interfaced to a personal computer by measuring the reflectance values at the appropriate wavelength of maximum absorption for each dyeing [28]. Samples were measured under illuminant D65, using a 10<sup>0</sup> standard observer with UV component excluded and specular included. The colour strength or K/S value was calculated by using the following Kubelka-Munk equation [29]:

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \quad (3)$$

where K is the absorption coefficient of the substrate, S is the scattering coefficient of the substrate and R is the reflectance value of the dyed samples at the wavelength of maximum absorption. For each sample, the measurements were made at four positions of each fabric sample and the average value is reported.

The tensile strength of the treated wool fabrics were assessed by using an Instron Tensile Strength tester (Model 4204, Instron Corporation, Norwood, USA) at  $20\pm 2$  °C and  $65\pm\%$  relative humidity according to the ASTM Test Method D5035-06: Standard Test Method for Breaking Force and Elongation of Textile Fabrics (Strip Method) [30]. The sample size was  $25.4\times 152.4$  mm, the gauge length was 80 mm and the traversing speed was 50 mm/min. The samples were conditioned at the above-mentioned temperature and humidity for at least 2 days. At least 10 samples were measured for each treatment and averages are reported here. The tensile strength was measured in both warp and weft both directions. The characterisation of the micro-level surface texture of various enzyme treated jute fabrics was carried out by scanning electron microscopy (JEOL JSM 6700F, JEOL Corporation, Japan) after sputter coating with gold to make the surface electroconductive. The surfaces of various scoured jute fabrics were also evaluated by a Karl Zeiss optical microscope (Karl Zeiss GmbH, Germany) with an Olympus DP70 digital microscope camera interfaced to a personal computer.

## **Results and discussion**

### **Characterisation of jute fibres treated with various enzymes**

#### **FT-IR analysis**

The FT-IR spectra of jute fibres treated with various enzymes are shown in Figure 1. The spectra showed prominent peaks in the fingerprint regions of 650 to 1520  $\text{cm}^{-1}$ . The spectrum of untreated jute is a typical spectrum of jute fibre containing lignin and hemicellulose. The peak at 1506, 1458 and 1418  $\text{cm}^{-1}$  represent aromatic skeletal in lignin, C-H deformation and C-H deformation in polysaccharides, respectively [31,32]. The peak at 1233  $\text{cm}^{-1}$  could be related to the -OH groups and the peak at 1158  $\text{cm}^{-1}$  could be attributed to C-O-C vibration in the polysaccharide. The other peaks are shown at 1052 and 896  $\text{cm}^{-1}$  could be attributed to C-O stretch in polysaccharide and C-H deformation in cellulose out of plane bending, respectively [33].

Insert Figure 1 here

We used FTIR data to qualitatively measure the lignin content of the various treated jute fabrics as published literature showed that FTIR could be used to determine lignin content of wood and jute fibres [32,34]. We measured the peak height at 1506, 1457 and 1418  $\text{cm}^{-1}$  as the intensity of these peaks is dependent on the lignin content [35]. It is evident that the height of these peaks decreased with a decrease in the lignin content of the fibre. It can be seen that the height of these peaks for the control scoured and the bio-scoured jute fibres with xylanase are almost similar. The height of these peaks decreased for the jute fibres bio-scoured with Esperase but the peak height considerably decreased in the case of jute fibres bio-scoured with combined cellulase and Alcalase (50/50 mixture). The decrease in the height of peaks at 1506, 1457 and 1418  $\text{cm}^{-1}$  clearly shows that the combined Alcalase and cellulase considerably increased the removal of lignin.

Insert Table 1 here

## Contact angle

The contact angle of a surface is related to its hydrophobicity; the higher the contact angle the higher the hydrophobicity. The contact angle of jute fabrics treated with various enzymes is shown in Table 1. The untreated jute fabric showed quite poor hydrophilicity and wettability as the contact angle shown at 0 s was 78.5 and after 45 s it decreased to only 58.5. Of the scoured jute fabrics, the highest contact angle at 0 s was shown by the alkali-scoured jute fabric but after 15 s the contact angle became 0. However, in the case of jute fabric treated with xylanase, the contact angle at 0 s was 69.4° but after 15 s the contact angle was still 57.7°. Table 1 also shows the photographs of water droplets on the surface of jute fabrics treated with various enzymes at 4% owf level. It can be seen that the surface of untreated jute fabric was quite hydrophobic as the droplet of water was not soaked by the fabric even after 45 s. Of the enzymatic treatments investigated, the xylanase-treated jute fabric showed the poorest wettability as the water droplet was absorbed after 25 s. In the case of the treatment with Esperase, the water droplet was absorbed after 15 s but for other treatments, the water droplet was soaked by the treated fabrics within 15 s. The alkali treated jute fabric also showed quite good hydrophilicity but the combined enzymatic treatment showed the best hydrophilicity as the water droplet was soaked by the fabric immediately after placing it on the fabric and it was not possible to measure the contact angle.

Table 1 also shows the wettability of the surface of jute fabrics treated with various enzymes measured by the AATCC Test Method 39-1998. It is evident that all the treatments considerably improved the wettability of jute fabrics compared to the untreated control fabric. The highest wettability was shown by the jute fabric treated with the combined enzymatic method (Alcalase + cellulase) as the water droplet was soaked by the fabric immediately after placing the water droplet on the fabric. The second best treatment in terms of wettability was

shown by the fabric treated by the traditional scouring treatment with alkali, which soaked the fabric by  $11 \pm 2$  s and the next best one was the jute fabric bio-scoured with Esperase, which took only  $15 \pm 2$  s to completely absorb the water droplet. On the other hand, the untreated jute fabric took quite a long time to wet (249 s) as the fibre surface was coated with cross-linked lignin, oil, and fat. Of the various treatments investigated, the treatment with xylanase showed the worst result but still increased the wettability of the fabric compared to its untreated jute fabric. The wettability and the contact angle data show that the Alcalase/cellulase treatment provided the best scouring performance for jute fabrics.

Insert Table 2 here

### **Effect on weight loss**

The effect of various enzymatic treatments on the weight loss of jute was shown in Table 2. The weight loss was occurred not only by the removal of hemicellulose, proteins and fatty matters available in jute fibres but also by the partial removal of lignin [36]. It can be seen that alkaline bio-scouring treatment resulted in the highest weight loss. However, the weight loss in the case of bio-scouring with various enzymes was considerably less than the weight loss occurred in the case of the alkali treatment. Of the enzymes investigated, xylanase caused the lowest weight loss and Alcalase/cellulase combined treatment caused the highest weight loss. The weight loss by the treatment with protease (Esperase) showed weight loss higher than the xylanase treatment but lower than the combined treatment. All the enzymes investigated here caused lower weight loss for the jute fabrics compared to the alkali scouring. However, the strength loss of jute fabrics increased with an increase in applied dose of the enzymes. Of the enzymes investigated, Alcalase in combination with cellulase at 4% owf showed the highest loss in tensile strength in both warp and weft directions. In the case

of alkali scouring the weight loss was 6.36%, which increased to 7.66% after the bleaching treatment with hydrogen peroxide. On the hand, in the case of combined Alcalase/cellulase scouring, the weight loss was 4.85% which increased to 6.16% after the bleaching treatment.

The addition of alkaline protease enhanced removal of insoluble lignin as well as the soluble lignin. The weight loss was further increased during bleaching but the trend was similar to the weight loss occurred in the bio-scouring process. It is evident that there is a correlation between the whiteness index and the weight loss data for the various enzymatic treatments.

Insert Figure 2 here

### **Effect of on mechanical properties**

The effect of various enzymatic bio-scouring treatments on the tensile strength of the jute fabric in both the warp and the weft directions is shown in Figure 2. The tensile strength of the control treated and various treated fabrics in the weft direction were lower than the tensile strength in the warp direction. Normally weft yarns have a lower twist compared to the warp yarns and as a result, they are weaker than the warp yarns. It is evident that the fabric treated with xylanase and the traditional alkali scouring caused the lowest and the highest tensile strength loss in both the warp and the weft directions. The tensile strength of alkali treated wool fabric was 39.8 and 31.1 MPa in the direction of warp and weft respectively. On the other hand, the jute fabric treated by the combined Alcalase and cellulase at 4% owf showed the tensile strength of 42.09 and 33.5 MPa respectively. Cellulase is know to have cellulose degrading capability but the lignin present in jute fibre seems inhibited the degrading power of cellulose and therefore limited the degradation of cellulose resulting in only a small loss in tensile strength.

## **Scanning electron microscopy**

The optical microscopic and SEM images of the surface of jute fabrics scoured with alkali and also with various enzymes at 4% owf are shown in Figure 3. The smoothness of the surface of jute fibre increased with various treatments in the order of alkali treatment>xylanase>protease>xylanase in combination with protease. In the case of alkali-scoured jute fabric, debris of impurities and particulates on the fibre surface are visible with partial removal of gummy substances from the fibre surface (Figure 3). These are non-cellulosic cementing materials (e.g. lignin) dislodged by alkali treatment that bind the ultimate cells together and they are responsible for providing high tensile strength to the untreated fibres. Therefore, the alkali scoured fabric showed quite low tensile strength. However, the surface of jute fibres scoured with various enzymes is relatively clean. It is evident that the Alcalase/cellulase combined treatment caused the formation of grooves on the fibre surface because of partial removal of lignin. The removal of fat, hemicellulose and the degradation of cellulase at the fibre surface by the combined action of Alcalase and cellulase dislodged some of the cross-linked lignin and therefore increased the hydrophilicity of the fibre surface as the time taken for complete wetting of the fibre surface was considerably decreased. The contact angle measurement results showed that the surface of fabric treated by the combined enzymatic treatment exhibited super-hydrophilicity. On the other hand, xylanase did not cause any fibre breakage and the surface of the fibres was comparatively clean without any fragments.

Insert Figure 3 here

## **Colour and Whiteness Index**

The *CIE L\*a\*b\** values and whiteness index of jute fabrics after the bio-scouring treatment as well as after the bleaching treatment are shown in Table 3. It is evident that all the bio-scouring treatments improved the whiteness index of the treated fabrics, which is due to partial removal of lignin from the jute fabrics because of the enzymatic hydrolysis [36]. The value of  $L^*$  for the bio-scouring treatment with Alcalase/cellulase at 4% owf was the highest (55.07) and the lowest was for the Esperase at 1% owf (51.06).

Insert Table 3 here

The whiteness index of the jute fabrics treated with alkali and enzymes alone was not high but its value considerably improved after the bleaching treatment with hydrogen peroxide. Enzyme treatment carried out prior to bleaching increased the pore volume of jute fibre as well as increased the amount of exposed lignin to the bleaching agent (hydrogen peroxide) causing an increase in bleaching performance and also an increase in whiteness of the fabric [10,37]. The whiteness index of the alkali treated jute fabric improved to 67.12 after the bleaching treatment with hydrogen peroxide and the maximum whiteness index (71.7) was shown by the jute fabric treated with combined Alcalase and cellulase. It is evident that all the enzymes investigated showed whiteness index better than the whiteness index achieved for the alkali treated jute fabric, especially at concentrations higher than 2% owf after the bio-scouring treatment and also after the bleaching treatment with hydrogen peroxide. For all the enzyme-treated jute fabrics, the whiteness index improved with an increase in the applied dosage of enzymes except for xylanase, for which the maximum whiteness index was achieved for the 3% owf. The whiteness index of the various enzyme-treated fabrics was further increase after the bleaching treatment with hydrogen peroxide. The best results were shown by the combined treatment with Alcalase and cellulase. The whiteness index of



various treated jute fabrics was consistent with the bio-scouring performance assessed by the ATR-FTIR spectroscopy and with the wettability data.

### **Effect on dyeability**

The dyeability not only depends on the functional groups of the fibre by ionic interactions but also improved with an increase in wettability. Fabrics with poor wettability may produce a poor depth of shade and also uneven dyeing. All the fabrics treated produced even dyeing indicating overall good scouring by all the treatments. The effect of various enzymatic treatments on the colour strength of the jute fabric dyed with two types of reactive dyes and one direct dye is shown in Table 4. It is evident that there is a correlation between the

Insert Table 4 here

whiteness index and the colour strength of the dyed fabric. The dyed jute fabric scoured with traditional alkali scouring produced poor colour strength (only 10.87, 21.23 and 32.1 for Rifazol Brilliant Orange 3R, Rifacion Red H-E3B, and Solophenyl Violet 4BLE, respectively). Of the two reactive dyes investigated, Rifacion Red H-E3B produced higher colour strength than the colour strength produced by the jute fabrics dyed with Rifazol Brilliant Orange 3R dye because of their different tinctorial strength. All the bio-scoured jute fabric samples at 1% owf various enzymes produced colour strength lower than the colour strength produced by the alkali-scoured jute fabric. With an increase in applied dosage of the enzymes, colour strength of the dyed fabrics considerably increased (such as the colour strength increased to 15.59, 26.33 and 35.66 for the jute fabrics treated with 4% owf combined Alcalase/cellulase dyed with Rifazol Brilliant Orange 3R, Rifacion Red H-E3B, and Solophenyl Violet 4BLE, respectively). The second best results were shown by the jute

fabrics treated with Esperase at 4% owf. The colour strength produced by the jute fabric scoured with the combined Alcalase/cellulase and dyed with reactive and direct dyes showed considerably higher compared to the alkali-scoured jute fabric dyed with the same dyes at the same depth of shade.

## **Discussion**

The complex scouring treatments used to treat jute fibres and fabrics are primarily to remove residual jute batching oil, hemicellulose, and also fatty matters. Lignin binds jute fibre cells together and provides mechanical load-bearing strength. Therefore, complete removal of lignin will disintegrate the fibres to cells and only a small percentage of lignin is removed to improve the exhaustion of dyes into the fibre and to provide uniform dyeability but a high level of bleaching is used to destroy their natural brown colour. Of the bio-scouring treatments investigated here, Alcalase in combination with cellulase provided the highest improvement in whiteness index and xylanase the poorest. The second best treatment in terms of improvement in whiteness was the scouring treatment with Esperase. The xylanase-treated jute fabrics showed comparatively poor whiteness compared to the treatments with other enzymes. The maximum whiteness index shown by xylanase and Esperase was 68.20 and 68.75 which increased to 71.7 for the treatment with Alcalase in combination with cellulase. Of the enzymatic bio-scouring treatments investigated, the alkaline protease in combination with xylanase-treated jute fabric showed the highest wettability and the lowest was shown by the xylanase-treated jute fabric. It indicates that xylanase in combination with alkaline protease facilitated bleaching of lignin during bleaching of jute fabrics with hydrogen peroxide. Alkaline protease in combination with cellulase showed the higher removal of lignin compared to the lignin removed by the treatment with xylanase as high alkaline conditions used in the case alkaline protease helped

in the removal of some loosely held lignin. In combination with cellulase, alkaline protease shows the synergistic effect and therefore not only wettability but also whiteness index increases. However, the actual mechanism of the enhanced removal of lignin by the combination enzymes is unknown.

From the wettability results shown in Table 1, it is evident that xylanase is not very effective in releasing lignin from jute fibre during the bio-scouring treatment, but it has the ability to degrade hemicellulose. Therefore, the whiteness of the fabric considerably improves after the bleaching treatment with hydrogen peroxide compared to the other enzymatic treatments. After dyeing with two reactive dyes and one direct dye, we found that the combined treatment (alkaline protease with cellulase) produced the highest colour strength, whereas xylanase-treated fabric, as well as the alkaline scoured fabric, produced poor colour strength.

## **5. Conclusion**

We demonstrated that the combined alkaline protease and cellulase-based enzymatic bio-scouring treatment could be a viable alternative to the traditional alkali-based bio-scouring treatment for jute fabrics. Of the enzymes investigated for the bio-scouring, Alcalase 2.5L in combination with cellulase at 50:50 ratio at 4% owf showed the best results in terms of the improvement in whiteness index, hydrophilicity, and wettability resulting in increased absorption of reactive and direct dyes. Control alkaline scouring showed quite high weight loss and also a loss in tensile strength but the whiteness produced was poorer compared to enzymatic scouring. The dyeability was also poor compared to jute fabrics bio-scoured with enzymes. Of the enzymes investigated, xylanase alone showed the poorest performance while the combined Alcalase/cellulase treatment showed the best performance. All of the treatments

made the fibre surface hydrophilic but the combined Alcalase/cellulase treatment showed the best hydrophilicity. Xylanase treated fabric showed the poorest hydrophilicity but the control alkaline scouring was only better than the bio-scouring treatment with xylanase. The FTIR spectra show that the combined enzymatic treatment and the alkali scouring removed some lignin from jute fibre but Esperase and xylanase hardly removed any lignin. The developed treatment could be an eco-friendly alternative to the alkaline bio-scouring of jute fabrics.


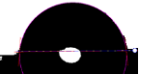











## References

1. G. Basu and A. N. Roy, *J. Natur. Fiber*, **4**, 13-29 (2008).
2. G. Buschle-Diller, Y. El Mogahzy, M. Inglesby and S. Zeronian, *Text. Res. J.*, **68**, 920–929 (1998).
3. C. Roul, “The International Jute Commodity System”, p. 22, The Northern Book Centre, New Delhi, India, 2009.
4. N.N. Das, S.C. Das and A.K. Mukherjee, *Text. Res. J.*, **54**, 166–171 (1984).
5. R. Agarwal, Y. Shukla, S. Kumar and N. K. Mehrotra, *Arch. Toxicol.*, **62**, 406–410 (1988).
6. K Wunna, K. Nagasaki, J.L. Auresenia, L. C. Abella and P. D. Gaspillo, *Chem. Eng. Trans. (Italy)*, **56**, 1831–1836 (2017).
7. E. Choe, C. Nam, S. Kook, C. Chung and A. Cavaco-Paulo, *Biocat. Biotransf.*, **22**, 375–382 (2004).
8. P. Agrawal, V. Nierstrasz and M. Warmoeskerken, *Enzyme Microb. Technol.*, **42**, 473–82 (2008).
9. M. Calafell and P. Garriga, *Enzyme Microb. Technol.*, **34**, 326–331 (2004).
10. R. Kozłowski, J. Batog, W. Konczewicz, M.M. Talarczyk, M. Muzyczek, N. Sedelnik and B. Tanska, *Biotechnol. Lett.*, **28**, 761–765 (2006).

11. P. Yuan, K. Meng, P. Shi, H. Luo, H. Huang, T. Tu, P. Yang and B. Yao, *J. Ind. Microbiol. Biotechnol.*, **39**, 909–915 (2012).
12. M.M. El-Zawahry, H.L. Helmi and A. Abou-Okeil, *Res. J. Textile Apparel*, **13**, 34–44 (2009).
13. N.A. Ibrahim, A.R. Abdel-Ghani, A.A. Gouda, I.H.M. Hanafi and M.Y. Hussein, *J. Natural Fiber*, **8**, 205–239 (2011).
14. F. Brühlmann, M. Leupin, K.H. Erismann and A. Fiechter, *J. Biotechnol.*, **76**, 43–50 (2000).
15. L.P. Christov, G. Szakacs and H. Balakrishnan, *Process Biochem.*, **34**, 511–517 (1999).
16. H.B.M. Lenting, M. Schroeder, G.M. Guebitz, A. Cavaco-Paolo and J. Shen, *Biotechnol. Lett.*, **28**, 711–716 (2006).
17. J. Shen, M. Rushforth, A. Cavaco-Paolo, G.M. Guebitz and H. Lenting, *Enzyme Microbial. Technol.*, **40**, 1656–1661 (2007).
18. J. Buchert, J. Pere, A. Puolakka and P. Nousiainen, *Text. Chem. Color. & Am. Dyestuff Rep.*, **32**, 48–52 (2000).
19. G. Freddi, R. Mossotti and R. Innocenti, *J. Biotechnol.*, **106**, 101–112 (2004).
20. G. Garg, S.S. Dhiman, R. Gautam, R. Mahajan, A.K. Patra and J. Sharma, *J. Mol. Cat. B: Enzymatic*, **85–86**, 43–48 (2013).
21. S. Ito, S. Shikata, K. Ozaki, S. Kawai, K. Okamoto, S. Inoue, A. Takei, Y. Ohta and T. Satoh, *Agric. Biol. Chem.*, **53**, 1275–1281 (1989).
22. R. H. Newman, A. A. Vaidya and S. H. Campion, *Bioresour. Technol.*, **130**, 757–762 (2013).
23. B. Bhunia, D. Dutta and S. Chaudhuri, *Eng. Life Sci.*, **11**, 207–215 (2011).

24. E. Csiszár, K. Urbánszki and G. Szakács, *J. Mol. Cat. B: Enzymatic*, **11**, 1065–1072 (2011).
25. A. Sellami-Kamoun, A. Haddar, N.E. Ali, B. Ghorbel-Frikha, S. Kanoun and M. Nasri, *Microbiol. Res.*, **163**, 299–306 (2008).
26. AATCC Test Method 39-1980: Evaluation of wettability, *Technical manual of the American Association of Textile Chemists and Colorists*, Vol. 57, Research Triangle Park, North Carolina, USA, 1981/1982, p. 290.
27. E. Aguayo, A. Allende and F. Artés, *Eur. Food. Res. Technol.*, **216**, 494–499 (2003).
28. M.M. Hassan and C.J. Hawkyard, *Col. Technol.*, **118**, 104–111 (2002).
29. P. Kubelka and F. Munk, *Z. Tech. Phys. (Leipzig)*, **12**, 593–601 (1931).
30. ASTM Test Method D5035-06: Standard Test Method for Breaking Force and Elongation of Textile Fabrics (Strip Method), *American Society for Testing Material*, Conshohocken, PA, USA, 2006.
31. W. Collier, T. Schultz and V. Kalasinsky, *Holzforschung*, **46**, 523–528 (1992).
32. B. N. Bandyopadhyay, A. Venkataraman and A. V. Gore, *Indian J. Fibre Text. Res.*, **24**, 156–159 (1999).
33. A. Mitchell, A. Watson and H. Higgins, *Tappi J.*, **48**, 520–533 (1965).
34. G. Zhou, G. Taylor and A. Polle, *Plant Methods*, **7**, 9 (2011).
35. J. Rodriguez, O. Faix and H. Pereira, *Holzforschung*, **52**, 46–50 (1998).
36. C. Vigneswaran and J. Jayapriya, *J. Text. Inst.*, **101**, 506–513 (2010).
37. A. Gessesse, R. Hatti-Kaul, B.A. Gashe and B. Mattiasson, *Enzyme Microbial. Technol.*, **32**, 519–524 (2003).

**Table 1.** Dynamic contact angle of alkali scoured jute fabric and also jute fabric bio-scoured with various enzymes at 4% owf.

Samples ID.	Average contact angle (°) at				Wettability (sec)
	0 s	15 s	30 s	45 s	
Untreated control					
	78.5±2.6	71.0±2.3	61.1±1.8	58.5±2.5	249±5
Xylanase					
	69.4±3.2	57.7±2.7	0	0	25±3
Esperase					
	0	0	0	0	15±2
Alcalase/cellulase					
	0	0	0	0	7±3
Scoured with alkali					
	55.4±3.0	0	0	0	11±2

**Table 2.** Weight loss (%) of jute fabrics during bio-scouring and bleaching treatments.

Applied dosage of enzymes (% owf)	Weight loss (%)			
	Control (alkali scoured)	Xylanase	Esperase	Alcalase/cellulase
<u>After bio-scouring</u>				
0	4.36±0.25	-	-	-
1		0.28±0.08	1.21±0.17	3.82±0.10
2		1.05±0.22	1.53±0.15	4.03±0.25
3		1.13±0.08	1.78±0.12	4.39±0.18
4		1.69±0.11	1.83±0.15	4.85±0.23
<u>After bleaching</u>				
0	7.66±0.18	-	-	-
1		3.39±0.34	3.81±0.16	4.24±0.12
2		4.78±0.27	5.10±0.22	5.25±0.18
3		5.42±0.39	5.44±0.25	5.81±0.15
4		5.47±0.27	5.80±0.20	6.56±0.10



**Table 3.** CIE  $L^*, a^*, b^*$  values and whiteness index of jute fabrics after bio-scouring and also after bleaching with hydrogen peroxide.

Applied dosage (% owf)	After bio-scouring				After bleaching			
	$L^*$	$a^*$	$b^*$	WI	$L$	$a^*$	$b^*$	WI
Alkali-scoured	50.09	5.18	14.64	47.72	73.72	2.41	19.6	67.12
Xylanase								
1	51.70	6.33	17.85	48.11	74.22	3.75	22.87	65.30
2	52.5	6.78	17.71	48.85	76.24	3.70	22.79	66.90
3	52.44	6.56	17.68	48.83	77.60	3.35	22.31	68.20
4	52.84	6.42	17.42	49.31	77.44	3.96	22.82	67.70
Esperase								
1	51.06	6.87	17.15	47.70	72.16	3.8	21.29	64.74
2	52.84	6.45	16.90	49.48	74.41	3.25	21.64	66.32
3	52.98	6.71	17.06	49.53	77.29	3.08	21.23	68.75
4	53.29	6.42	16.70	50.00	77.45	3.36	21.72	68.51
Alcalase/cellulase (50%/50%)								
1	53.01	6.39	16.88	49.70	79.18	3.63	22.83	68.90
2	53.62	6.54	17.21	50.10	79.36	3.37	22.73	69.10
3	54.02	5.87	17.09	50.60	80.56	3.03	21.63	70.80
4	55.07	6.41	16.93	51.60	81.31	2.74	21.09	71.70

**Table 4.** Colour strength of various enzyme-treated jute fabrics dyed with two reactive dyes.

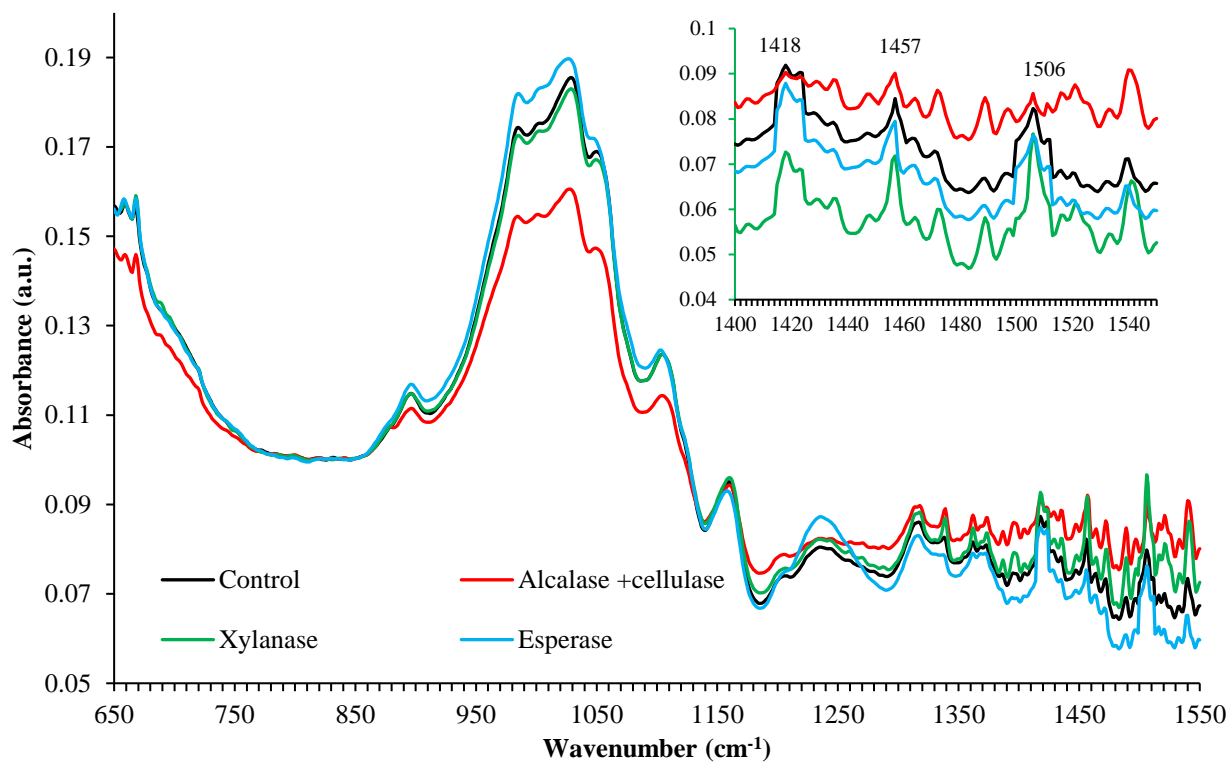
Applied dosage of enzymes (% owf)	Color strength (K/S)			
	Control (alkali scoured)	Xylanase	Esperase	Alcalase/cellulase
<u>1% Rifazol Brilliant Orange 3R</u>				
0	10.87±0.11	-	-	-
1	-	10.50±0.08	10.34±0.05	10.71±0.10
2	-	11.83±0.12	11.52±0.11	12.15±0.12
3	-	13.33±0.06	12.68±0.07	14.68±0.09
4	-	13.68±0.10	13.32±0.12	15.59±0.15
<u>2% Rifacion Red H-E3B</u>				
0	21.23±0.12	-	-	-
1	-	20.74±0.15	18.02±0.12	20.55±0.14
2	-	21.28±0.10	21.75±0.17	22.20±0.22
3	-	23.64±0.20	21.14±0.21	25.64±0.25
4	-	24.52±0.17	23.16±0.18	26.33±0.12
<u>2% Solophenyl Violet 4BLE</u>				
0	32.10±0.09	-	-	-
1	-	30.74±0.14	30.10±0.15	31.35±0.12
2	-	32.28±0.10	32.65±0.12	32.89±0.15
3	-	32.64±0.21	33.80±0.20	35.30±0.10
4	-	33.52±0.15	34.50±0.22	35.66±0.15

### **Figure captions**

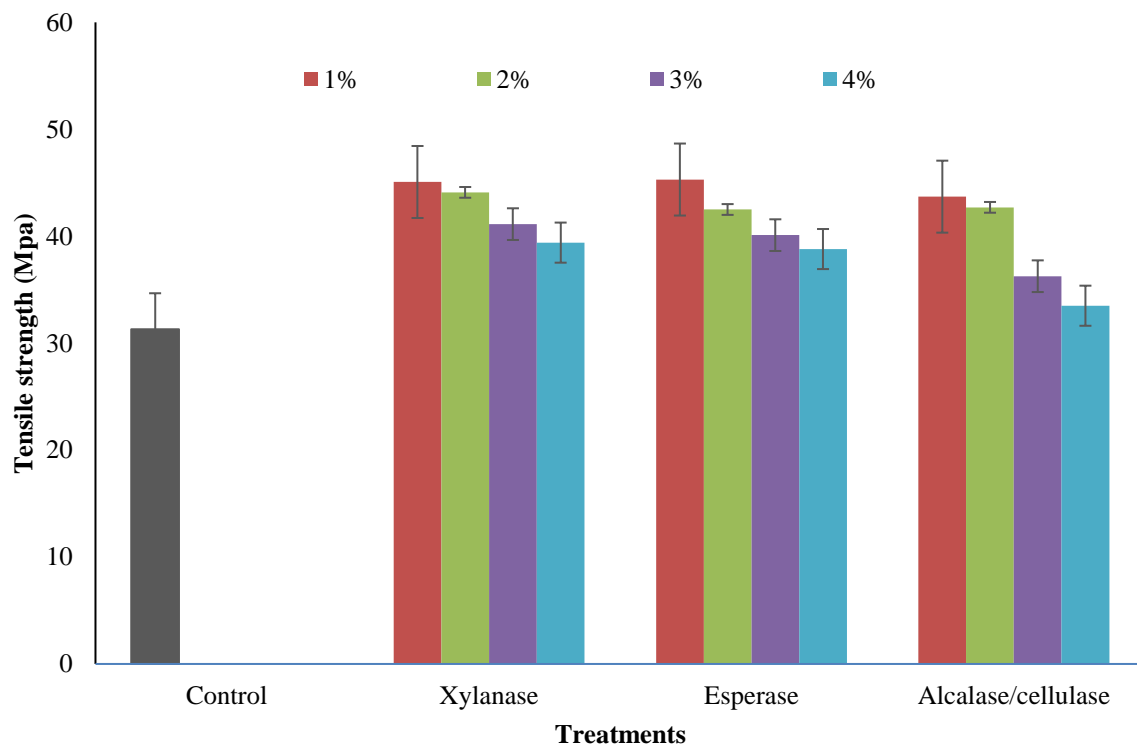
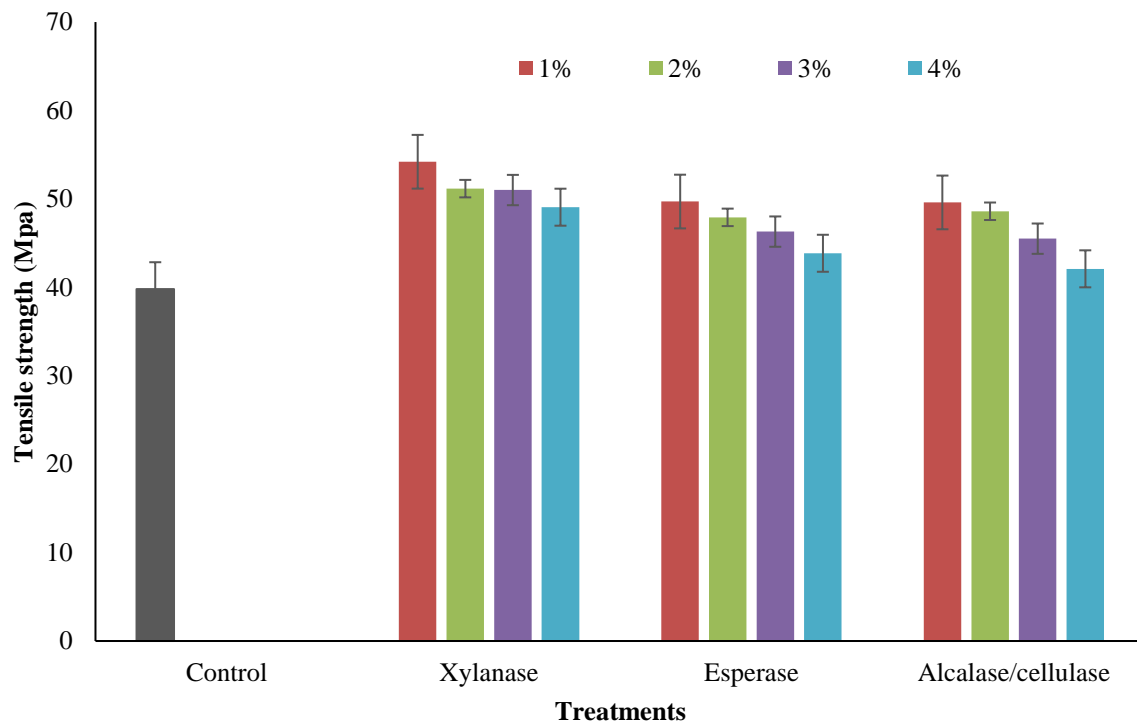
**Figure 1** ATR-FTIR spectra of alkali-scoured jute fabric and also jute fabric bio-scoured with various enzymes at 4% owf.

**Figure 2** Effect of various enzymatic treatments on the tensile strength of jute fabrics in the direction of warp (top) and weft (bottom).

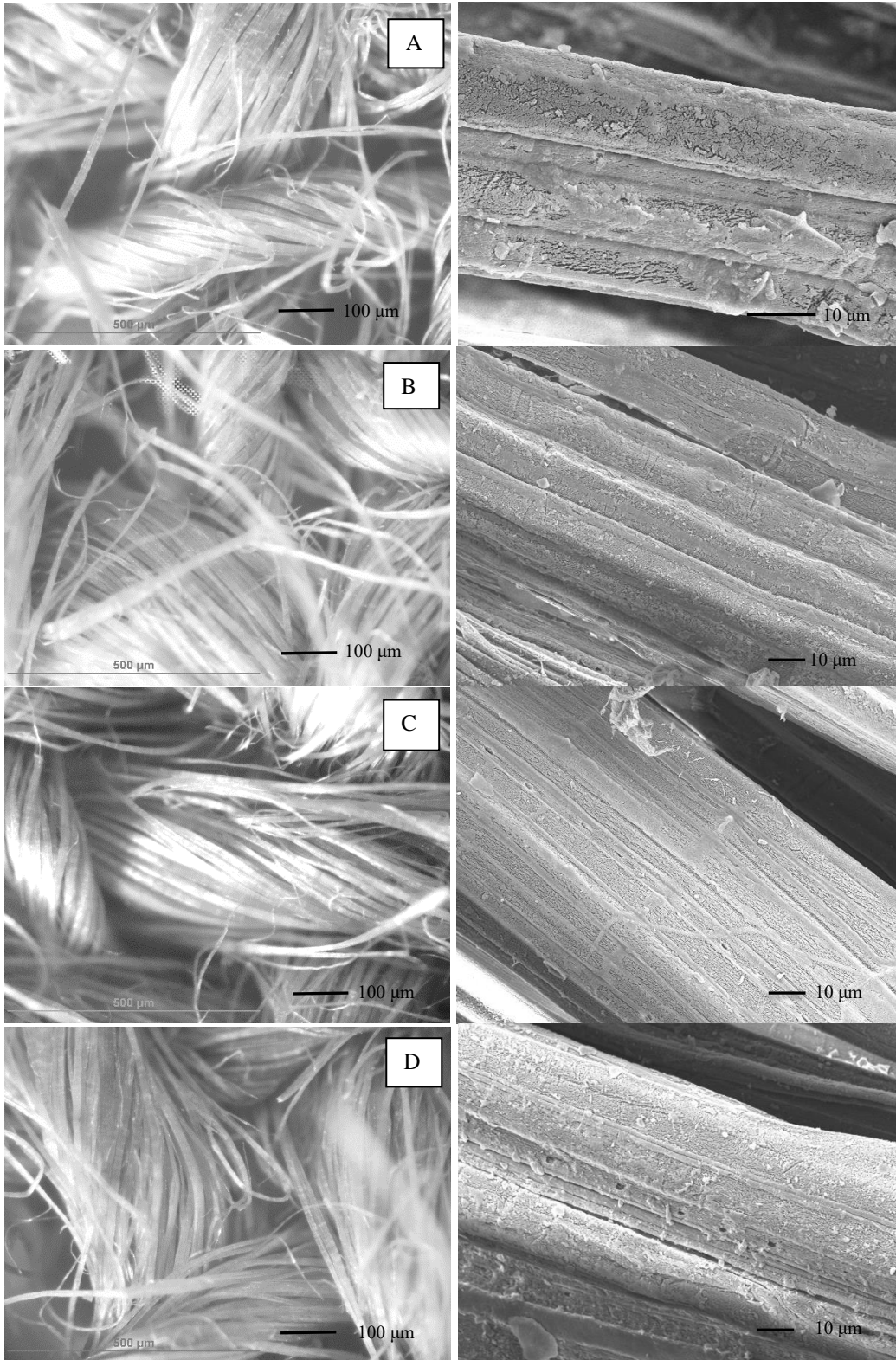
**Figure 3** Optical microscopic (left) and SEM (right) images of jute fabrics scoured with various enzymes at 4% owf. A = alkali-scoured; B = xylanase; C = Esperase; D = Alcalase/cellulase.



**Figure 1.** ATR-FTIR spectra of control scoured (alkali) jute fabric and also jute fabric bio-scoured with various enzymes at 4% owf.



**Figure 2.** Effect of various enzymatic treatments on the tensile strength of jute fabrics in the direction of warp (top) and weft (bottom).



**Figure 3.** Optical microscopic (left) and SEM (right) images of jute fabrics scoured with various enzymes at 4% owf. A = alkali-scoured; B = xylanase; C = Esperase; D = Alcalase/cellulase.