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Sequential Enzymatic and Oxidative Pre-Treatment Effect on Natural Lignocellulosic Fibres

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In order to get the aesthetic look or making fibre to fabric for apparel purpose or for good quality hessians, sackings, bags, etc. one must do the pre-treatment processes like scouring, degumming, bio-scouring, bleaching, etc. This will enhance the efficiency, easiness and effectiveness during yarn making, fabric making, spinnability, dyeing, etc. In this work, study has been targeted to achieve finer, brighter and whiter fibre while maintaining the strength of the fibre. Four natural fibres namely *Corchorus olitorius* (Jute), *Musa domestica* (Banana), *Linum usitatissimum* (Flax) and *Boehmeria nivea* (Ramie) were undergone pre-treatment processes like scouring, degumming, enzyme treatment and bleaching in sequential manner. Lignin content, gummy matter and yellowness of the fibre goes on decreasing while fineness, whiteness and brightness kept on increasing with each subsequent chemical treatment and washing. The outcome of the work was the scoured, enzyme treated, and bleached jute, banana, flax and ramie fibre with enhanced fineness, smoothness and whiteness which can be further used for yarn and fabric making, dyeing and apparel preparation.

Keywords: Bleaching, Brightness, Bio-scouring, Fabric, FTIR, Lignin

1 Introduction

Natural fibre plays an irreplaceable role in eco-friendly packaging, upholstery, mulching, handicrafts and artefacts, etc. Fibres play an aesthetic, beautifying and fashionable life style to the end users. It is the basic livelihood for farmers in some parts of India. It acts as the major cash crop for them and thus increases their socio-economic status. Major plant-based fibres are jute, flax, ramie, banana, sisal, cotton, etc. Today, jute and other natural fibre-based products have become versatile with a wide range of consumer products. Utilization of jute and other natural fibre for manufacture of market worthy products has opened up large opportunity for employment generation. A wide range of diversified jute and other natural fibres products are developed every day. These diversified products include fancy jute bags, soft luggage, footwear, door panels, check sarees, wide range of furnishing, gift items, table lamps, floor decor, wall decor and many more items. To make the yarn or fabric suitable for spinning, weaving or for beautification of the fabric one has to go for pre-treatment of the fibre. The pre-treatment may be in the form of scouring, enzyme treatment and bleaching, etc. This will help in removing dirt, stain and other impurities from the raw fibre and making it suitable for the desired products.

Researchers are doing their best to achieve the enhanced strength, whiteness, fineness and suitability of the fibre to be converted to yarn, spinnability in the mill to be converted into the final beautiful fascinating products like handicrafts, bags, upholstery, clothes, hessian bags, etc. In order to make finer, soft and smooth fibre suitable for blending with other fibre in different proportions and thus yarn making, researchers have done pre-treatment and surface modification of fibre. Chattopadhyay *et al.*[1] have blended hydrogen peroxide bleached banana fibre with jute to obtain soft and finer fibre suitable for yarn making and have further utilized for making cotton blended fabric. Sometimes pre-treatment methods and properties of modified yarn or fabric vary with type of fibre and fabric. Cotton fabrics were bleached via ozonation in combination with sodium persulfate and sodium perborate to enhance the dyeability and pre-treatment efficiency.[2] To treat cellulosic fibre like cotton, one can attain higher smoothness and finesses as compared to treatment of lignocellulosic fibres like jute, banana, ramie, and flax, etc. Even the treated fibre properties vary with the types of chemicals used (enzyme treated or whether no chemicals used at all).

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Hannan *et al.*[3] have treated the cotton knit fabric in water at different temperatures (105 °C, 120 °C and 130 °C) for different time (20, 40 and 60 minutes) to obtain the enhancement in colour and dyeability. They have witnessed the removal of wax and pectin from the fabric after treatment by interpretation with the FTIR spectra of the different treated fabrics. Ding *et al.*[4] have found that properties of flax fibre can be enhanced by pre-treatment with alkaline and enzymatic scouring, acidic bleaching with different oxygenated bleaching agents. They have attained better tenacity, whiteness, fineness and fibre elongation by removal of hemicellulose and lignin from flax fibre by their pre-treatment methods. Singh *et al.*[5] have demonstrated the effect of enzyme on the degumming and scouring of ramie fibres. They were able to utilize xylanase and pectinase enzyme as bioscouring agent for removal of xylan and pectin from the ramie fibre at different experimental conditions to enhance the whiteness. Yang *et al.*[6] have used ionic liquids (1-butyl-3-methylimidazolium acetate-water mixtures) to degum the bast fibre of *Apocynum venetum* and obtained very lustrous fibre at experimental conditions like 80% ionic liquid concentration, 1:20 (liquor ratio) and 90 °C temperature for a duration of 4 hour. Zhang *et al.*[7] have treated flax roves with enzymes like xylanase, cellulase and their mixtures to attain whiteness and residual gum content at an optimized condition of 3% enzyme dosage, 1:2 (cellulase: xylanase) enzyme ratio, under supercritical carbon dioxide conditions of 20 MPa pressure, 50 °C temperature and at 30 g/min flow of CO_2 for 90 minutes. Eco-friendly degumming of banana fibres was also obtained by Kaur *et al.*[8] by utilizing xylanase and pectinase enzyme for bio-scouring process. They were able to get finer and whiter banana fibre. By looking at the background information on the above-mentioned topic, a lot of work has been done by several researchers on different natural fibres across the world.

In the present work, a comparative, eco-friendly and chemical pre-treatments analysis of different fibres have been studied to reveal its impact on fibre strength, fineness and optical properties. For this work, we have selected the locally available natural lingocellulosic fibres (jute, banana, flax and ramie) which are available in abundance. The outcome of the study will be helpful in assessing the effect of different enzymatic as well as chemical pre-treatment methods on the fibre properties.

2 Materials and Methods

Fibres used in this study are locally available lignocellulosic fibre namely jute, banana, flax and ramie. Sodium hydroxide pellets was purchased from Merck Specialities Private Limited, Mumbai. Glacial acetic acid and sodium silicate solutions were procured from LobaChemie Private Limited, Mumbai. Ultravon JU oil was obtained from local market (Bara Bazar, Kolkata). Sodium trisulphite, Trisodium phosphate and hydrogen peroxide was procured from Merck Life Science Private Limited, Mumbai. Enzyme (Texzyme M and Texzyme J) was obtained from Tex Biosciences (P) Limited, Tirruvallur, TN (India). All the chemicals were used as it is obtained.

Fibres (Jute, banana, flax and ramie) were subsequently and sequentially undergone the pre-treatments like scouring (degumming in case of ramie fibre), enzyme treatments and bleaching. Chemical processing and characterization of the treated and raw fibres has been done in Chemical and Bio-chemical Processing Division and testing parameters like strength and fineness has been tested in Quality Evaluation and Improvement Division of ICAR-NINFET, Kolkata. Methodologies followed for scouring (degumming in case of ramie fibre), enzyme treatments and bleaching are mentioned below.

2.1 Scouring

Scouring is an alkaline treatment of fibre to remove the natural and added impurities during processing like wax, coloured stain, and clay or dust material adhered to the fibre at high temperature. It further aids in improving the processing of fibres to achieve better performance. Schematic representation of scouring process was presented in Fig. 1.

2.2 Degumming of ramie fibres

Degumming is the process of removal of gummy matter like pectins, polysachharides, etc. from raw ramie fibre by alkaline treatment. It has been done as per protocol presented in Fig. 2.

2.3 Enzyme treatment

Bio-scouring was done by treating the fibres with enzyme to achieve smooth and impurity free fibre. It has been done as follows as per Fig. 3.

2.4 Bleaching

Bleaching is a process of removal of unwanted colours and achieving the whiteness and brightness. It is done to obtain white fibre on which dyes can be

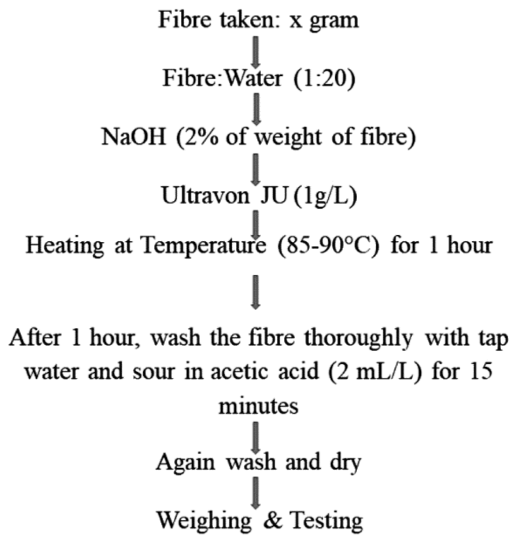

Fig. 1 — Scouring procedure followed for different fibres (jute, banana, flax and ramie).

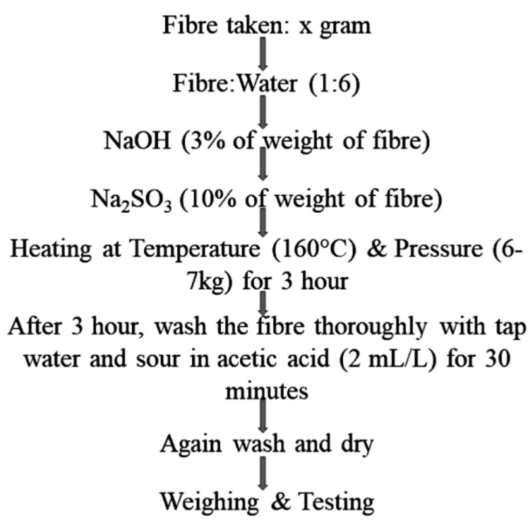

Fig. 2 — Degumming process of ramie fibres for removal of gummy material.

applied successfully. It has been done using hydrogen peroxide (H_2O_2) as bleaching agents (Fig. 4).

2.5 Experimental plan and design

The sequence of treatments followed for jute, banana and flax fibres are shown diagrammatically in Fig. 5. In case of ramie fibre, degumming is done for extraction of gummy material from the fibre. All the samples were washed thoroughly between each treatment and fibre obtained from each step were evaluated for optical as well as physical properties.

2.6 Testing parameters

After obtaining the scoured, degummed, enzyme treated and bleached fibres, following test parameters

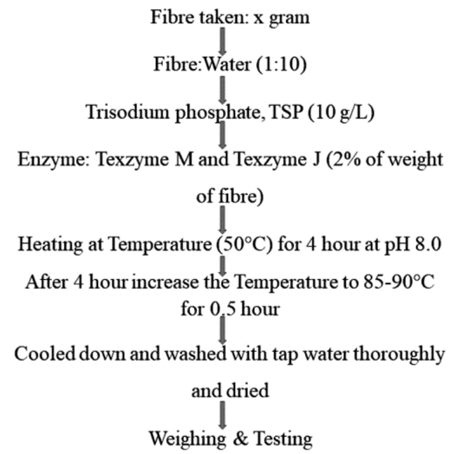

Fig. 3 — Enzymatic scouring of different lignocellulosic fibres (jute, banana, flax and ramie).

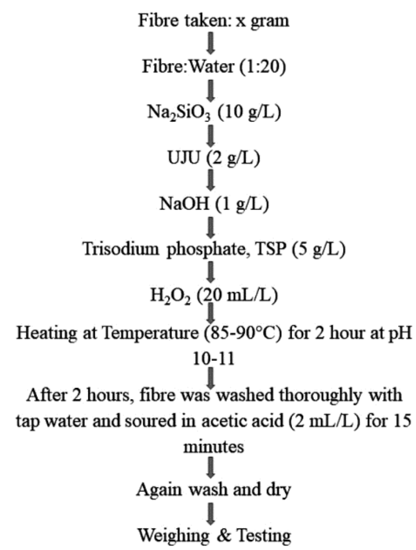

Fig. 4 — Bleaching of scoured and enzyme treated fibres (jute, banana, flax and ramie) with hydrogen peroxide.

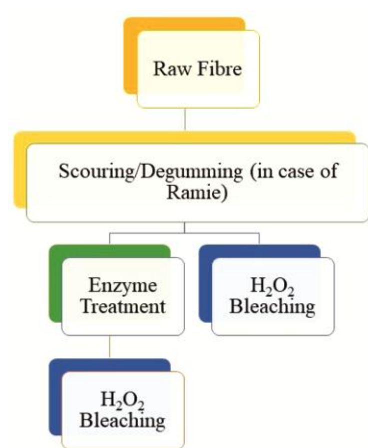

Fig. 5 — Pre-treatments processes and layout followed for jute, banana, flax and ramie fibres

were done to know the strength, fineness, whiteness, brightness, yellowness and lignin percentage of the treated fibres along with raw fibre:

(a) Fibre bundle strength was tested by NINFET Bundle Strength Tester
(b) Fineness of fibre was tested by NINFET Air-flow Fineness Tester (in case of jute) and by Gravimetric method (in case of banana, ramie and flax)
(c) Whiteness on HUNTER Scale, Yellowness on ASTM D1925 Scale, Brightness on TAPPI 452 Scale by Spectrascan-5100 computerized color matching system
(d) FTIR spectra of fibre was recorded by Model-ALPHA-Bruker, Germany, using Attenuate Total Reflection (ATR) method.

2.7 Lignin content estimation of fibres

Lignin present in all the treated fibres had been extracted during each chemical treatments and washing. Lignin act as natural adhesive and is responsible for binding the individual fibre and thus provides the strength. That is why lignin percentage present in each fibre after every treatment was estimated as per TAPPI 222 method[13] to know the effect of chemical treatment on the strength of the fibre. Percentage of lignin present in each fibre was correlated with FTIR spectra of each fibre.

3 Results and Discussion

Physico-chemical properties of jute, banana, flax and ramie fibres were presented in Table 1. Being different in chemical composition, the pre-treatment process has varying effect on these fibres.

After pre-treatment processes, scoured, degummed, enzyme treated and bleached fibres were obtained which can be further used for dying and other purpose. Photographs of different treated fibres were presented in Fig. 6 (jute), Fig. 7 (banana), Fig. 8 (flax) and Fig. 9 (ramie), respectively. Images reveals brighter and shiner fibre after each successive pre-treatment.

Table 1 — Chemical compositions of jute, banana, flax and ramie fibres

Fibre	Chemical compositions				References
	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Pectin (%)	
Jute	59-71	12-13	11.8-12.9	0.2-4.4	9
Banana	58.50	13.1	7.4	4.3	10
Flax	62–72	18.6–20.6	2–5	2.3	11
Ramie	68.6-76.2	13.1-16.7	0.6-0.7	1.9	12

3.1 Strength and Fineness of pre-treated fibres

Strength of fibre goes on decreasing with each sequential enzymatic and chemical treatment. Raw fibre possesses more strength as compared to other treated fibres. Reason behind decreasing fibre strength after each enzymatic and chemical treatment was the removal of lignin and other adhering components from fibre. Thus, making the fibre weaker, finer and brighter after each pre-treatment step. Strength is least in scoured-enzyme treated-hydrogen peroxide bleached fibre. Fineness of the different treated fibres goes on increasing (decrease in the value of tex) with

Fig. 6 — Raw, scoured, scoured-hydrogen peroxide bleached (S-H_2O_2), scoured-enzyme treated (S-ET), and scoured-enzyme treated- hydrogen peroxide bleached (S-ET-H_2O_2) jute fibres

Fig. 7 — Raw, scoured, scoured-hydrogen peroxide bleached (S-H_2O_2), scoured-enzyme treated (S-ET), and scoured-enzyme treated- hydrogen peroxide bleached (S-ET-H_2O_2) banana fibres

Fig. 8 — Raw, scoured, scoured-hydrogen peroxide bleached (S-H_2O_2), scoured-enzyme treated (S-ET), and scoured-enzyme treated- hydrogen peroxide bleached (S-ET-H_2O_2) flax fibres

Fig. 9 — Raw, degummed, degummed-hydrogen peroxide bleached (Deg-H_2O_2), degummed-enzyme treated (Deg-ET), and degummed-enzyme treated- hydrogen peroxide bleached (Deg-ET-H_2O_2) ramie fibres

subsequent enzymatic and chemical treatment followed by washing. It is more in case of scoured-enzyme treated-hydrogen peroxide bleached (S-ET-HP) fibre followed by scoured-hydrogen peroxide bleached (S-HP), scoured-enzyme treated (S-ET), scoured and raw fibre (Fig. 10). Ramie was found to be the finest fibre among all the other fibres (Jute, Banana and Flax) under study.

3.2 Optical properties (Whiteness index-WI, Yellowness index-YI and Brightness index-BI)of different pre-treated fibres

Whiteness, yellowness and brightness of different pre-treated fibres were tested by Spectrascan-5100 computerized color matching system. Whiteness was measured on HUNTER Scale, Yellowness on ASTM D1925 Scale and Brightness on TAPPI 452 Scale. With each enzymatic and chemical treatment, whiteness and brightness of fibre goes on increasing and yellowness follows the opposite trend. Whiteness is more in scoured-enzyme treated-hydrogen peroxide bleached (S-ET-HP) fibre followed by scoured-hydrogen peroxide bleached (S-HP), scoured-enzyme treated (S-ET), scoured and raw fibre. Whiteness, yellowness and brightness index of different pre-treated jute fibres are presented in Fig. 11. Scouring and enzyme treatment purify and cleans the fibres and results in some improvement in brightness and reduction of yellowness index. These processes aid in removing dirt, stains, dust and other impurities from the fibres. Bleaching of pretreated fibres produces significant improvement in whiteness and brightness and the fibres are ready for subsequent colouration process. All the pre-treated fibres appear to be glossy and shining by look.

3.3 FTIR Spectra and correlation with lignin content of different pre-treated fibres

Lignin estimation of different pre-treated fibres was estimated as per TAPPI 222 methods and FTIR spectra were recorded by Model: ALPHA-Bruker, Germany, using Attenuate Total Reflection (ATR) method. As we go on treating fibres enzymatically and chemically one after other in a subsequent manner, lignin extraction/removal from the fibre occurs at each step. Percentage of lignin was found to be more in raw fibre as compared to scoured/degummed, scoured/degummed-enzyme treated (S/DEG-ET), scoured/degummed-hydrogen peroxide bleached (S/DEG-HP), and scoured/ degummed-enzyme treated-hydrogen peroxide bleached (S/DEG-ET-HP) fibres. In FTIR-spectra,

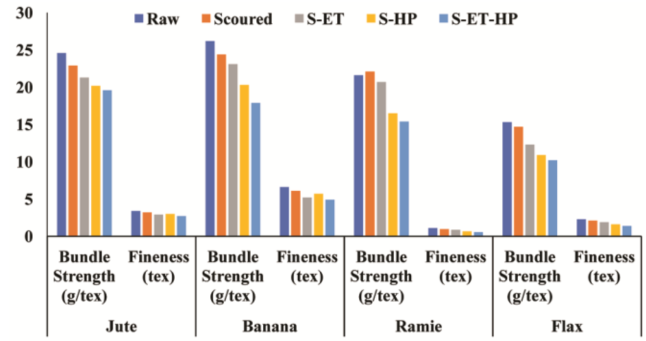

Fig. 10 — Fibre strength and fineness of different pre-treated fibres (Jute, Banana, Flax and Ramie)

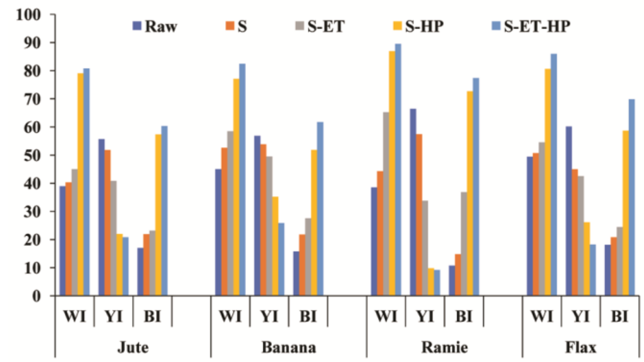

Fig. 11 — Optical properties like whiteness, yellowness and brightness of different pre-treated fibres (Jute, Banana, Flax and Ramie)

absorption band in the region of 3435 cm^{-1}, 1733 cm^{-1} and 2901 cm^{-1} are due to O-H stretching vibration (hydroxyl group in cellulose), C=O stretching vibration (carbonyl group of acetyl ester in hemicellulose) and C-H stretching vibration (carbonyl aldehyde in lignin), respectively[14]. In all the raw fibre, the peculiar band at 1510 cm^{-1} belongs to vibration of phenyl propane groups of lignin which gradually vanishes after each chemical treatment[15]. These peaks intensity goes on decreasing as we go on subsequent treatment from raw to scoured/degummed, scoured/degummed-enzyme treated (S/DEG-ET), scoured/degummed-hydrogen peroxide bleached (S/DEG-HP), and scoured/degummed-enzyme treated-hydrogen peroxide bleached (S/DEG-ET-HP) fibres (Fig. 12(a-d)). This happens because at each level of chemical treatment certain percentage of lignin and other materials like cellulose and hemicellulose goes on extraction which can be directly correlated with the FTIR spectra of different pre-treated fibres. Lignin percentages of different pre-treated fibres were presented in Table 2.

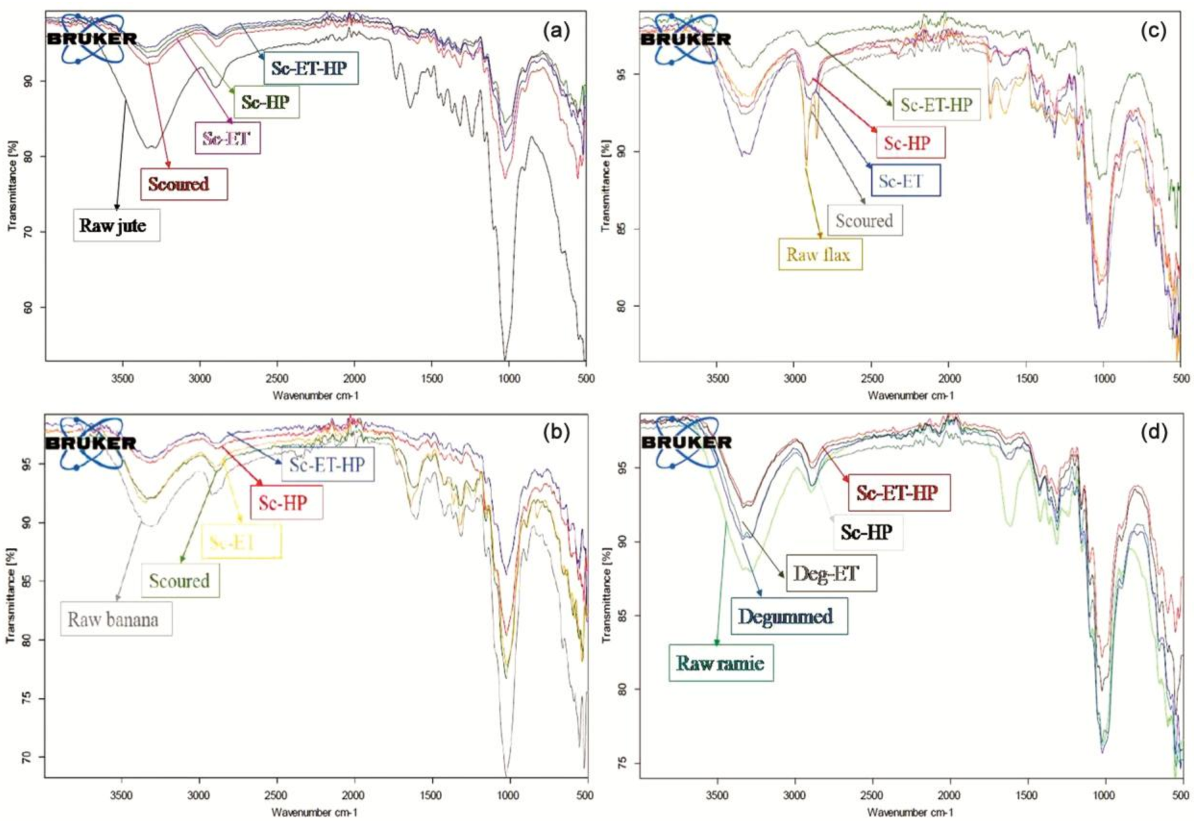

Fig. 12 — FTIR spectra of different pre-treated fibres (a) Jute, (b) Banana, (c) Flax, and (d) Ramie.

Table 2 — Percentage of lignin in different pre-treated fibres

	Lignin %			
Sample	Jute	Banana	Flax	Ramie
Raw	13.37	7.37	4.94	0.58
Scoured/Degummed	8.36	6.33	3.43	0.40
S/DEG-ET	7.58	5.89	2.76	0.33
S/DEG-HP	5.73	5.01	2.39	0.18
S/DEG-ET-HP	5.06	4.03	1.80	0.11

4 Conclusions

Jute, banana, flax and ramie fibres are natural, biodegradable and renewable lignocellulosic fibres. These fibres have immense potential in textile and non-textile industries. Fibres as such in raw form can’t be used for making diversified and value added products hence, these fibres have to be processed with certain unit operations like scouring (degumming in case of ramie), enzyme treatment to achieve cleanliness and softening which can further be bleached to gain whiteness. Degumming is the first step for extraction of ramie after decortication while all other fibres need scouring for its cleaning to remove dirt, dust and added impurities. Enzyme treatment is done for further cleaning and softening of the fibre. The scoured and enzyme treated fibres were bleached using hydrogen peroxide to produce white and bright fibres. The enzyme treated fibres are found to be glossy as compared to that produced by bleaching only scoured fibers. All the fibres show improvement of fineness and some loss in tensile strength after successive treatments due to removal of major part of lignin, hemicellulose and minor part of cellulose which is evident from FRIR spectra. These treated fibres are ready for further processing like dyeing and finishing to achieve the improved effect on diversified products.

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