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Structural and chemical modifications in linen fabrics due to scouring and bleaching

Anju Kushwaha, Priyanka Kesarwani[a] & Rashi Kushwaha
Department of Family & Community Sciences, University of Allahabad, Prayagraj 211 002, India

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This study examines the effects of scouring and bleaching on the morphological properties of the linen fabric. Scanning electron microscopy (SEM) analysis reveals slight changes in the surface characteristics of scoured and scoured & bleached linen fabrics compared to greige linen fabric, with increased surface roughness. Fourier transform infrared spectroscopy (FTIR) analysis confirms that the fundamental cellulosic bonds (–OH, C-O-C and C-H stretching) remain intact, as indicated by similar absorption peaks across greige, scoured and scoured & bleached linen fabrics. X-ray diffraction (XRD) analysis further supports these findings, showing an increase in the amorphous region in the scoured, and scoured & bleached linen fabrics.

Keywords: Bleaching, Crystallinity, Linen fabric, Scouring, Surface morphology

1 Introduction

Flax (*Linum usitatissimum*), also known as linseed, belongs to the genus “Linum” within the family “Linaceae”[1]. Textiles made from flax are known as linen and are among the oldest textiles in the world. As a natural cellulose fabric, linen attracts tremendous attention in the textile industry owing to its excellent moisture absorption, temperature regulation, high breaking strength, antibacterial characteristics[2-4], high hygroscopicity, cool and pleasant touch, excellent air permeability, and low accumulation of electrostatic charges on the surface. Linen products create an optimal microclimate for the skin, offering beneficial effects on humans[5].

Linen’s desirable properties make it increasingly popular for apparel production, as it ensures superior wearing comfort[6]. However, greige linen fabric contains various impurities, such as pectin, lignin, waxy materials, and natural oils, which hinder its hydrophilicity. Thus, prior to dyeing and finishing, scouring is necessary to remove these non-cellulosic impurities, such as pectin, contributing to its hydrophobic nature[7]. The success of dyeing, printing, and finishing depends on the effectiveness of pre-treatment of the fabrics.

This study examines the effects of conventional alkaline scouring and bleaching on the greige linen fabric’s surface morphology, chemical composition, and crystallinity.

[a]Corresponding author.
E-mail: kpriyanka1280@gmail.com

2 Materials and Methods

2.1 Materials

The greige 100% Linen fabric (plain weave, 40×40 yarns per cm) was purchased from Jain Bandhu Traders, Gandhi Nagar, New Delhi, India. A non-ionic surface-active agent (Felosan HLDN) was purchased from CHT India Pvt, Ltd. Maharashtra, India. Sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2) and sodium silicate (Na_2SiO_3) were obtained from the local market in Prayagraj, India.

2.2 Methods

2.2.1 Scouring

Scouring is a crucial pre-treatment process that removes impurities such as pectin, lignin, waxy substances, and natural oils from raw fibres or fabrics, ensuring a clean and smooth surface[8]. For scouring, greige linen fabric was treated with NaOH (2% owf) and a non-ionic surface-active agent (2 g/L) at 90ºC for 1 h, maintaining a material-to-liquor ratio of 1:20. After scouring, the fabric was thoroughly washed with cold water and subsequently treated with acetic acid (2 mL/L) for 20 min at room temperature (20-25°C) to neutralise residual alkali. Finally, the fabric was washed with cold water and dried[9].

2.2.2 Bleaching

The scoured linen fabric was bleached using H_2O_2 (5 g/L), NaOH (1 g/L) and Na_2SiO_3 (1 mL/L) at 80ºC for 1 h. The treated fabric was then rinsed with cold water, followed by washing with hot water for 5 min, and subsequently dried at room temperature.

2.2.3 Scanning Electron Microscopy (SEM)

The surface morphology of the greige, scoured and scoured & bleached linen fabrics was examined using a JSM-6490LV (JEOL, Japan) scanning electron microscope. Fabric samples were securely mounted on a specimen holder using carbon tape so that the sample did not shift or fall off while handling. The samples were coated with platinum to enhance conductivity and improve interaction with the electron beam. Micrographs were captured at magnifications ranging from X30 to X1000 to analyse surface characteristics.

2.2.4 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was done to recognise functional groups in the greige linen fabric and to analyse any structural modifications in the scoured, scoured & bleached linen fabric samples. FTIR spectra were recorded using a Nicolet 6700 spectrophotometer (Thermo Scientific, USA), where fabric samples were palletized with KBr powder. The number of scans was 32, and the resolution was 4 cm^{-1}.

2.2.5 X-ray Diffraction (XRD)

X-ray diffraction analysis was conducted to evaluate the crystallinity of the greige, scoured and scoured & bleached linen fabrics using a D8 Advance Eco diffractometer (Bruker, Germany). Changes in the porous structure of linen fabrics due to scouring were assessed, as these modifications may influence dye absorption and other post-treatment processes[10].

3 Results and Discussion

3.1 SEM Analysis

The surface morphology of greige, scoured, and scoured & bleached linen fabrics was examined using SEM to assess the impact of scouring and bleaching treatments. SEM images at ×550 magnification reveal distinct differences in fibre surface characteristics among the linen fabric samples (Fig. 1).

Fig. 1 (a) indicates the presence of non-cellulosic materials surrounding the fibres in the greige linen fabrics, giving them a relatively smooth appearance. In contrast, the scoured linen fabric [Fig. 1 (b)] exhibits a rougher surface attributed to the removal of the non-cellulosic layer composed of pectin, lignin, waxy substances, and natural oils. This observation is consistent with findings by Fracz *et al*.[11] and Verma & Goh[12], who reported that scouring removes fatty deposits, thereby increasing surface roughness. The increase in the roughness after scouring may also be due to the cell wall of linen fibres being constructed of spiraling fibrils composed of cellulose polymer[13]. Further analysis of scoured & bleached linen fabric reveals an even more pronounced roughness [Fig. 1 (c)]. This increase in surface irregularity may be due to the partial opening of spiralling fibrils on the fibre surface upon exposure to NaOH and H_2O_2 during bleaching, resulting in a more fibrillated and textured structure.

The fibre thickness measurements indicate that greige linen fabric exhibits an average fibre thickness of 16.27 μm. Upon scouring, the fibre thickness decreases to 14.75 μm, and a further reduction to 14.49 μm is observed after bleaching. The reduction in fibre thickness is likely due to the removal of residual non-cellulosic components, further confirming the effectiveness of scouring and bleaching processes.

Although the scouring process effectively removes impurities, all the impurities cannot be removed just by scouring. Therefore, the bleaching process is done to remove the leftover impurities and make the fabric whiter. Since the thickness of the fibres of bleached linen is lower than that of the other two samples, it

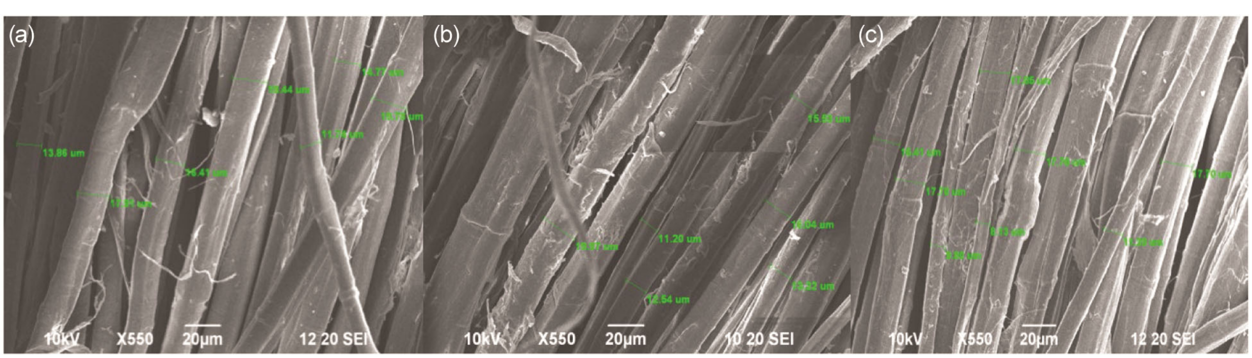

Fig. 1 — SEM images of (a) greige linen, (b) scoured linen and (c) scoured & bleached linen

confirms that the bleaching process removes the leftover impurities from the scoured linen sample. These findings align with the study by Karaca *et al.*[7], which reported a similar trend in fibre thinning following bleaching due to the elimination of remaining impurities.

SEM images also reveal that the fibres in scoured and scoured & bleached linen fabrics are more compact than greige linen fabric [Fig. 1 (b) and (c)]. This increased compactness is likely due to the removal of non-cellulosic substances, which allows the fibres to come closer together, resulting in a denser fabric structure.

3.2 FTIR Analysis

FTIR spectroscopy was conducted to examine changes in functional groups, particularly hydroxyl (-OH) groups, in the linen fabric samples. Published literature[14,15] shows that FTIR can be used to determine the -OH content of bast fibres. In the linen, cellulose exhibits strong inter and intra-molecular bonding interactions between adjacent cellulose chains involving the hydroxyl groups[16]. FTIR spectra of greige, scoured, and scoured & bleached linen fabrics are presented in Figure 2.

The spectra showed prominent peaks in the 400 to 4000 cm^{-1} fingerprint regions. The peak at 3311 cm^{-1} in the greige linen spectrum corresponds to the cellulose's -OH stretching vibration. After scouring, this peak shifts to 3352 cm^{-1}, and upon bleaching, it further shifts to 3426 cm^{-1}. The observed shift indicates increased exposure of hydroxyl groups, likely due to the removal of non-cellulosic impurities, enhancing fibre accessibility.

The C-H stretching vibration observed at 1434 cm^{-1} in both greige and scoured fabrics shifts slightly to 1436 cm^{-1} after bleaching. Similarly, the C-O-C stretching peak initially at 1150 cm^{-1} in greige linen shifts to 1158 cm^{-1} following scouring but reverts to 1150 cm^{-1} after bleaching, indicating the stability of ether linkages, which are unreactive, durable and least affected by degrading agents. These findings are in accordance with the results published by Gohl & Vilensky (1987)[13]. Arik[17] and El-Gaoudy *et al.*[16] also reported similar shifts in FTIR spectra of bast fibres following chemical treatments. Wang *et al.*[18] affirmed that –OH, C-O-C and C-H stretching are the backbone of the typical cellulose.

3.3 XRD Analysis

XRD analysis was performed to assess changes in crystallinity and amorphous content in the linen fabric samples. The diffraction patterns of greige, scoured, and scoured & bleached fabrics are shown in Fig. 3.

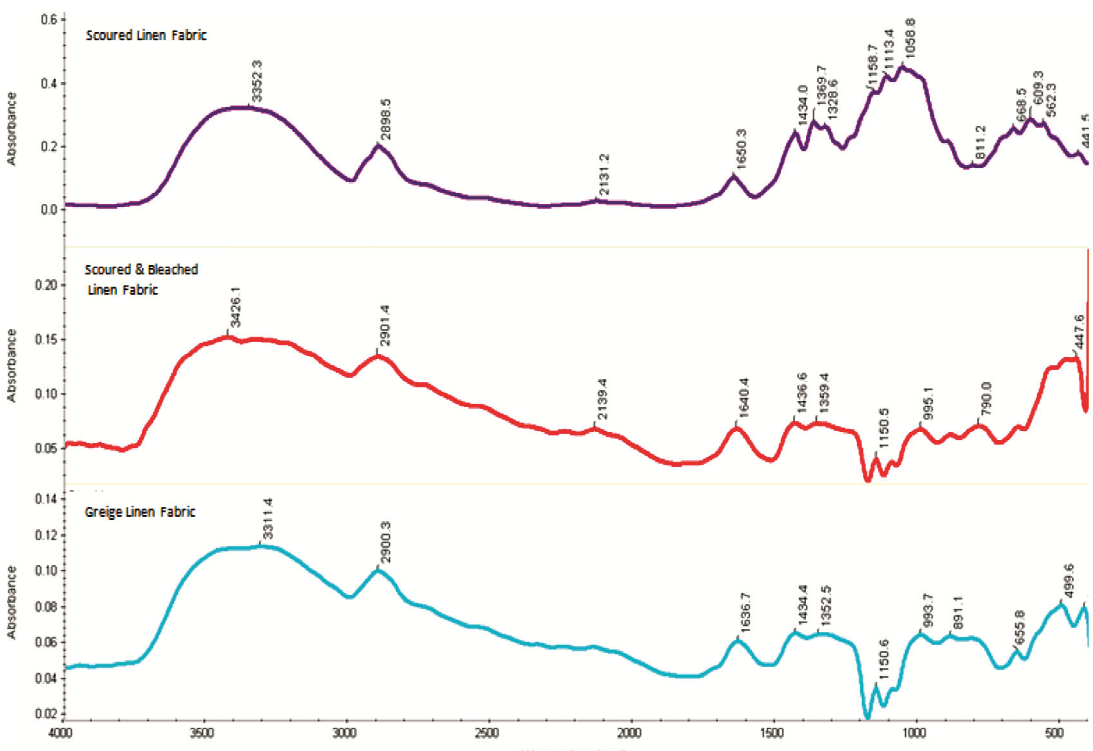

Fig. 2 — FTIR spectra of greige, scoured and scoured & bleached linen fabrics

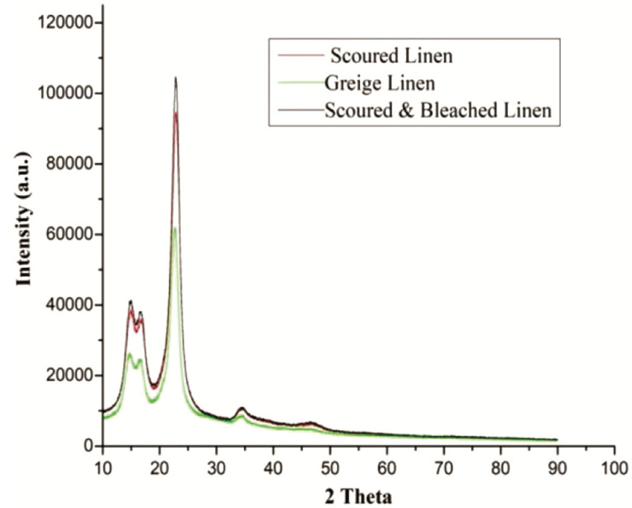

Fig. 3 — XRD patterns of greige, scoured and scoured & bleached linen fabrics

A sharp and thinner peak at 22.6° (2θ) is observed in all three fabric samples, indicating the presence of a crystalline region characteristic of cellulose. Additionally, a broad peak appears at approximately 17.2° on 2θ, representing the amorphous region.

Notably, the peak at 17.2° is broader in greige linen, indicating a higher proportion of amorphous content due to the presence of non-cellulosic materials. After scouring, the peak broadens further, suggesting an increase in amorphous regions as impurities are removed. This trend continues after bleaching, where a more pronounced broadening of the 17.2° peak is observed, confirming that the scouring and bleaching processes enhance fibre absorbency by increasing the amorphous content. These results align with previous studies[7, 12], which reported similar increases in amorphous content following scouring and bleaching, leading to improved dyeability and absorbency of bast fibres.

4 Conclusion

The study demonstrates that scouring and bleaching significantly alter the structural and chemical properties of linen fabrics. SEM analysis confirms increased fibre roughness, compactness, and reduced thickness due to the removal of non-cellulosic components. Furthermore, FTIR analysis confirms that -OH, C-O-C and C-H stretching is not diminished, confirmed by the similar absorption peaks of all three fabrics. XRD analysis shows increased amorphous content, improving fibre absorbency. These modifications enhance linen's process ability, making it more suitable for textile applications requiring improved dye ability, absorbency, and functional performance.

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