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Knit Fabric Mercerisation through the Use of High-Concentration NaOH in a Scouring and Bleaching Bath using an Exhaustion Method

Mercerizacija pletiva z uporabo visoke koncentracije NaOH v kopelih izkuhavanja in beljenja z metodo izčrpavanja

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Abstract
The combined scouring, bleaching and mercerising of single jersey cotton knitted fabrics through the use of high-concentration NaOH was evaluated in this study. The effect of that new combined process was compared, contrasted and analysed with pristine scoured, bleached fabric and conventional bleached fabric. Surface morphology, structural analysis and the colour strength of dyed fabrics were examined using a scanning electron microscope (SEM), X-ray diffraction (XRD) and spectrophotometric analysis. The barium activity number and bursting strength of treated fabric was evaluated and analysed in accordance with the relevant AATCC test standards. It was found that a sample treated through combined scouring, bleaching and mercerising demonstrated a higher level of smoothness and lustre than typical scoured and bleached fabric. The treated sample exhibited decreased crystallinity, as well as the transformation of the crystalline phase in a similar manner. The barium activity number, bursting strength and improved K/S value were also in line with the separately mercerised fabric. The above described properties of the combined scoured, bleached and mercerised fabric are sound evidence of the effectiveness of the process.
Keywords: surface morphology, X-ray diffraction, barium activity number, crystallinity, bursting strength

Izvleček
V študiji je bil ovrednoten vpliv kombiniranja procesov izkuhavanja, beljenja in merceriziranja bombažnih desnolevih pletiv z uporabo visoke koncentriracije NaOH. Učinek novega kombiniranega postopka je analiziran in primerjan na surovem izkuhanem, beljenem in konvencionalno beljenem pletivu. Analiza morfologije površine vlaken, strukture in obarvanja pletiv je bila opravljena z elektronsko vrstično mikroskopijo (SEM), sipanjem rentgenskih žarkov (XRD) in spektrofotometrično analizo. Barijsko število aktivnosti in razpočna trdnost obdelanih pletiv sta bila analizirana in ovrednotena s pomočjo ustreznih standardov AATCC. Ugotovljeno je bilo, da je bil vzorec, obdelan po kombiniranem postopku izkuhavanja, beljenja in merceriziranja, bolj gladek in je imel višji lesk kot izkuhano in beljeno pletivo po klasičnem postopku. Končni vzorec je imel nižjo kristaliničnost in tudi nižjo pretvorbo kristaline faze. Barijsko število aktivnosti, razpočna trdnost in izboljšanje vrednosti K/S so se ujemali z vrednostmi za mercerizirano pletivo pri klasičnem postopku. Lastnosti, dosežene pri v kombiniranem postopku oplemenitenega pletiva, potrjujejo učinkovitost novega postopka.
Ključne besede: morfologija površine, sipanje x-žarkov, barijsko število aktivnosti, kristaliničnost, razpočna trdnost

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1 Introduction

Mercerisation is the best-known method for enhancing the physical and dyeing properties of cotton fibre. Mercerisation changes the surface morphology, fine structure (i.e. crystallinity), crystal size, etc. of cotton fibre [1, 2]. It converts the cellulose chain from cellulose I to cellulose II [3–5]. A significant improvement in dye affinity, colour strength, lustre, tensile strength and the smoothness of cotton fabric also occurred due to mercerisation [1, 6, 7]. The degree of change depends on different parameters, such as processing time, processing temperature, tension, the concentration of caustic soda, etc. [8]. The mercerisation of knitted fabric was considered impractical due to stretching in the process itself, but is essential for enhancing the properties of knitted fabric [9]. Today, different manufacturers design machines for knit fabric mercerisation that does not harm the dimensional stability of fabric. Both open width and tubular fabric can be mercerised with newly invented machinery without damaging the quality of fabric. Highly concentrated NaOH solution (14% or higher) has been used extensively to modify cellulosic fibre for a long period of time. Such a modification of cellulosic fabric improves the dye receptivity of cotton and reduces the shrinkage of cotton fabric [1]. The development of slack mercerisation resolves many problems associated with knit fabric mercerisation [10]. Fabric used to be treated with high-concentration caustic soda without tension to achieve excellent physical and dyeing properties [11, 12]. Mercerisation at high temperature is now a well-established method [13]. The penetration of caustic soda into the fibre structure is more rapid [14], which ensures more uniform treatment but less swelling [8]. This in turn enhances lustre without affecting the strength of the fibre. On the other hand, high temperatures and high-concentration caustic soda give fabric a plastic-like nature, which results in significant improvements in properties [15]. A compatibility chart of dyes and chemicals shows the compatibility of caustic soda with hydrogen peroxide. Several studies have reported combined scouring and mercerising at high temperatures for different methods. Processing, however, is not deemed sustainable due to its limited application.

Taking into account all of the above facts, this study hypothesised that modified slack mercerisation without tension through the use of high temperatures, caustic soda and hydrogen peroxide would prove to be a simple and novel approach. As a part of our study, the properties of fabric pre-treated with combined scouring, bleaching and mercerising were contracted and compared with mercerised fabric and with individual scoured, bleached fabric. The changes in surface morphology, fine structure and physical properties were assessed to validate the effectiveness of combined scouring, bleaching and mercerising.

2 Materials and methodology

2.1 Materials

Single jersey cotton knitted fabric of 160 g/m^2 was purchased from Fakhruddin Textile Mills Ltd., Bangladesh. All the chemicals (NaOH, H_2O_2, sequestering agent, anti-creasing agent, stabiliser and detergent) were purchased from the Aziz Group, Bangladesh.

2.2 Methodology

Three samples were prepared for the study. Sample 1 (S1) was a conventional scoured and bleached single jersey knitted fabric, while sample 2 (S2) was fabric treated by conventional scouring and bleaching followed by caustic mercerisation at room temperature and sample 3 (S3) was single jersey cotton knitted fabric treated with combined scouring, bleaching and mercerising. All the parameters of common processes were kept constant for all three samples. Figure 1 illustrates the process curve of the preparation of all samples. Conventional scouring and bleaching of S1 and S2 was performed using 0.6 g/l detergent, 0.5 g/l sequestering agent, 0.5 g/l anti-creasing agent, 2 g/l NaOH, 3 g/l H_2O_2 and 0.4 g/l stabiliser. S2 was later treated with 20% NaOH solution for mercerisation purposes. S3 was prepared to bring out the effect of mercerisation at a high concentration (16 g/l) of NaOH during scouring and bleaching, which is referred to as combined scouring, bleaching and mercerising. For further study, S1, S2, and S3 were bound together and dyed with a vinyl-sulfone reactive dye applying an exhaustion method in the same bath. To achieve precise results, dyeing was performed using three different shade percentages, i.e. 1%, 3% and 5%.

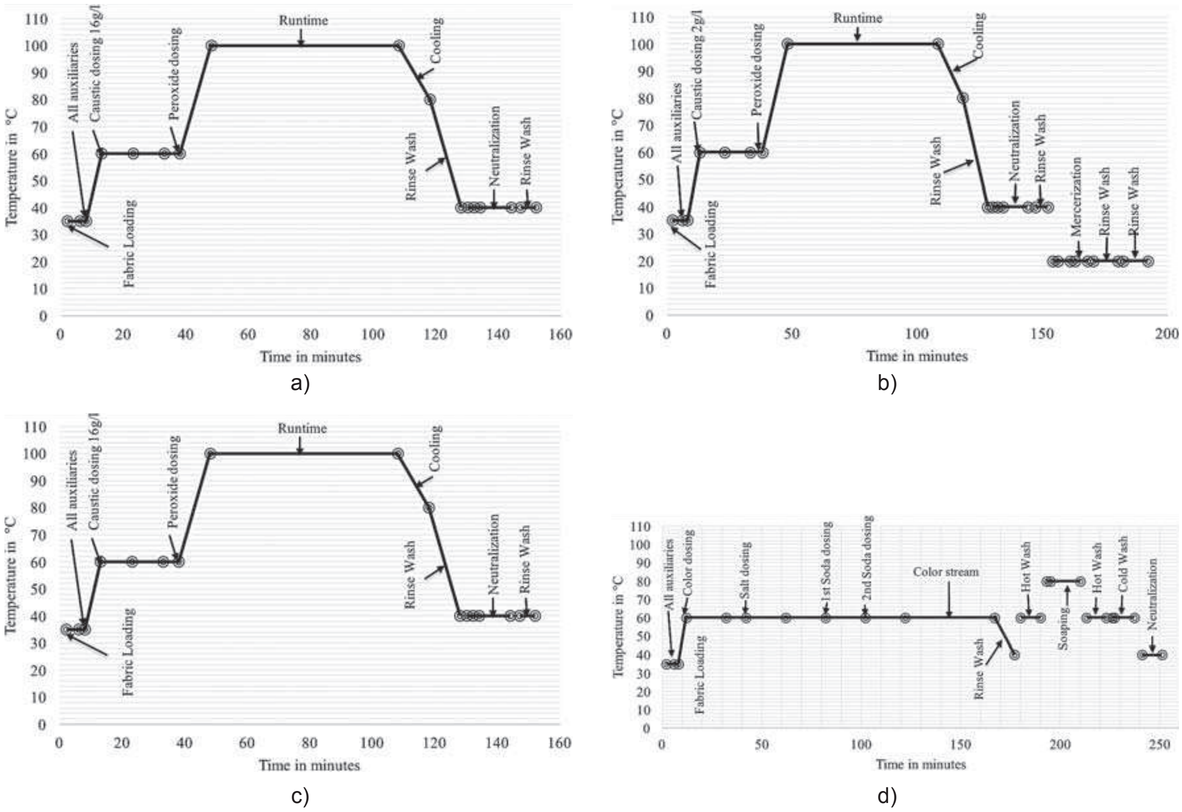


Figure 1: Process curves for: a) scouring and bleaching, b) scouring, bleaching and separate mercerising, c) combined scouring, bleaching and mercerising, and d) dyeing of all types

2.3 Characterisations

Changes of surface morphology were assessed using a JSM-6510 scanning electron microscope (JEOL Ltd.). Accelerating voltage of 20 KV and magnifications of 2000x and 3000x were used during scanning microscopy. Fine structural changes were analysed using an MDI JADE-6 X-ray diffractometer. Crystallinity was determined using the ratio of integrated crystallite scattering intensity to total scattering intensity ranging from 10 to 36°. The amount of crystallinity and amorphous fraction were determined using synthesised curves to diffractograms obtained from the experiment through successive approximation. Lorentzian distributions, which represent crystalline component peaks, the amorphous component and contribution due to the air scattering of the X-ray beam, were calculated using a programmable calculator. The degree of mercerisation and barium activity number were measured using the AATCC 89-1998 test method. Samples were taken from all three fabrics and cut into small pieces. This was followed by the treatment of 1 g of each fabric type with 30 ml of 0.25 barium hydroxide solution in a 100 ml conical flask for two hours. Ten ml from each sample solution was then titrated against 0.1 N HCI. To calculate the result, a blank solution of barium hydroxide was also titrated. The result was obtained using the following equation:

$$BAN = \frac{T_1 - T_2}{T_1 - \mathrm{T}_2} \times 100\ [\%] \qquad (1),$$

where: *BAN* represents the barium activity number; T_1 represents the titration reading for untreated fabric; T_2 represents the titration reading for mercerised fabric; and T_3 represents the titration reading for un-mercerised fabric.

Before testing bursting strength, all fabrics were conditioned and left to achieve a relaxed state. After full relaxation, bursting strength was measured using a bursting strength tester. The test was carried out 10 times for each sample, and the average value recorded and expressed in kg/cm^2. The K/S value of dyed fabric was determined using a spectrophotometer (Datacolor-650). Initially, the reflectance percentage of dyed fabric was measured. The *K/S* value was then calculated applying the Kubelka-Munk theory. The K/S value can be obtained using the following equation:

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

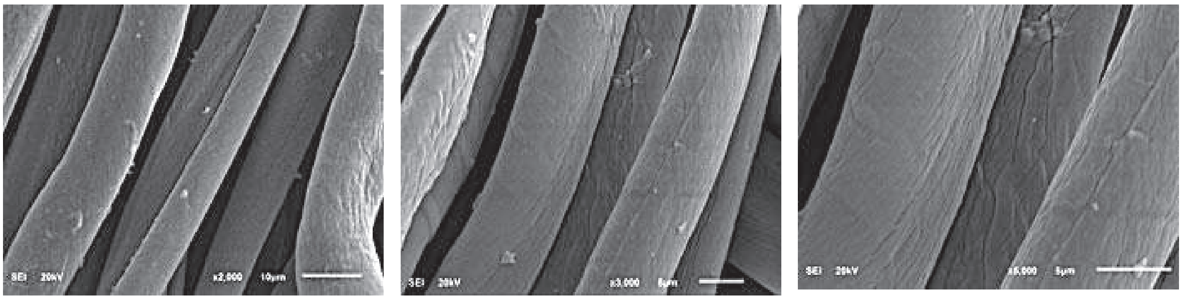
where: K represents absorbance; S represents scattering; and R represents reflectance [%].

3 Results and discussion

3.1 Changes in surface morphology

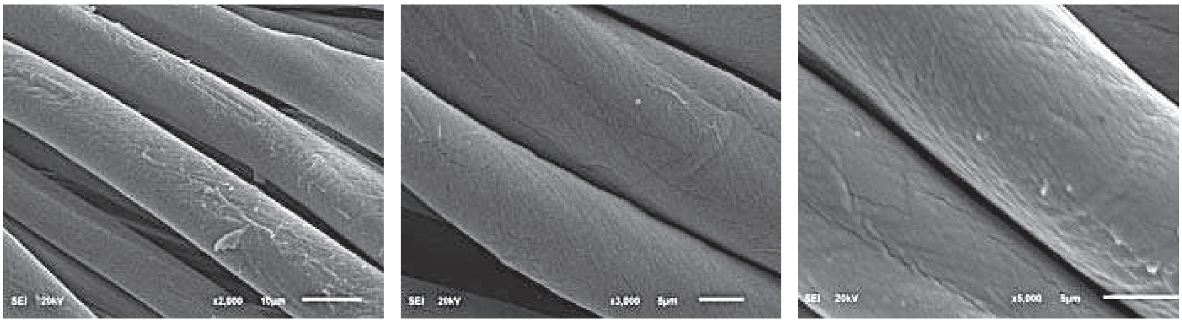
It is a well-known fact that the natural deep wrinkles from the surface of cotton fibre tend to vanish due to caustic mercerisation. The cell wall of the fibre becomes thicker, depending on maturity, while the natural twisted ribbon-like structure tends to change and form a round-like structure. Porous or granular phenomena appeared on the surface of the fibre.

The figures clearly show that the fibres demonstrate a round structure instead of a twisted ribbon-like structure. A significant number of the natural deep wrinkles were removed from the surface. In all cases, the fibre structure appears to have a smoother surface than the previous one. A smoother surface allowed for more regular reflectance, which resulted in the more lustrous surface of the fabric. The lustre of the combined scoured, bleached and mercerised



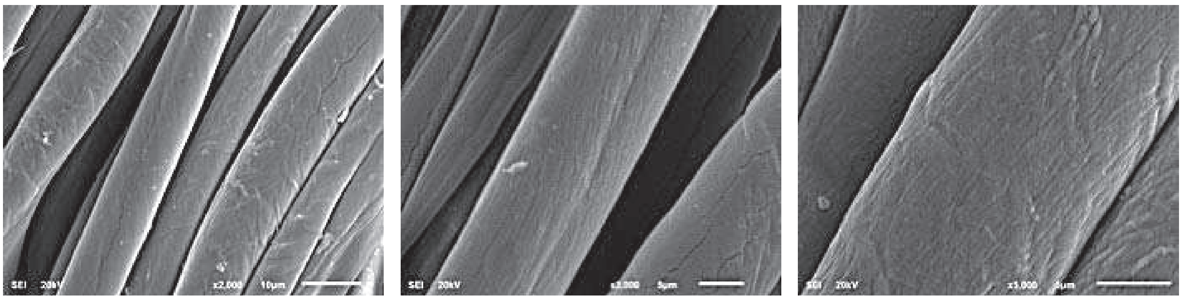
a) b) c)

Figure 2: SEM image of sample S1 (magnifications: a) 2000x, b) 3000x and c) 5000x)



a) b) c)

Figure 3: SEM image of sample S2 (magnifications: a) 2000x, b) 3000x and c) 5000x)



a) b) c)

Figure 4: SEM images of sample S3 (magnifications: a) 2000x, b) 3000x and c) 5000x)

fabric (S3) is far better than the conventional scoured and bleached fabric (S1). There were no significant differences in terms of lustre between the combined scoured, bleached and mercerised fabric (S3) and separately mercerised fabric (S2).

3.2 Changes in fine structure

After the cotton fibre was treated with high-concentration NaOH, it formed a cellulose-soda-water compound. During the destruction of the aforementioned, cellulose converted from cellulose I to cellulose II. This conversion of cellulose depended entirely on the treatment condition. Most of the converted cellulose failed to return to its original position, resulting in an increase in the amorphous region of the cotton fibre and a decrease in crystallinity. Figure 5 presents an XRD diagram of conventional scoured and bleached fabric (S1), separately mercerised fabric (S2) and combined scoured, bleached and mercerised fabric (S3). It is evident that S1 demonstrates normal crystallinity, while S2 demonstrates a significantly reduced crystallinity, which may have occurred due to the penetration of high-concentration caustic soda into the highly oriented crystalline region. The same phenomenon occurred in the case of S3. The results obtained from the XRD are summarised in Table 1. Among the three tested samples, the crystallinity of S2 and S3 decreased significantly, with S3 demonstrating a more significant decrease in crystallinity than S2. This can be explained by differences in temperature. A high mercerisation temperature decreased the viscosity of NaOH solution and enhanced the diffusion property by swelling the highly ordered area of fibre and decreasing the hydrophobic nature of natural impurities. However, the difference in crystallinity between the two mercerised fabrics is immaterial.

This is due to the lower concentration of caustic soda in S3 than in S2.

Table shows that both mercerised fabrics (S2 and S3) exhibit the crystalline phase of cellulose II, while the amount of cellulose II is higher in S2. The extent of the decrease in crystallinity and the conversion of cellulose I to cellulose II are strong evidence of the effectiveness of combined scouring, bleaching and mercerising.

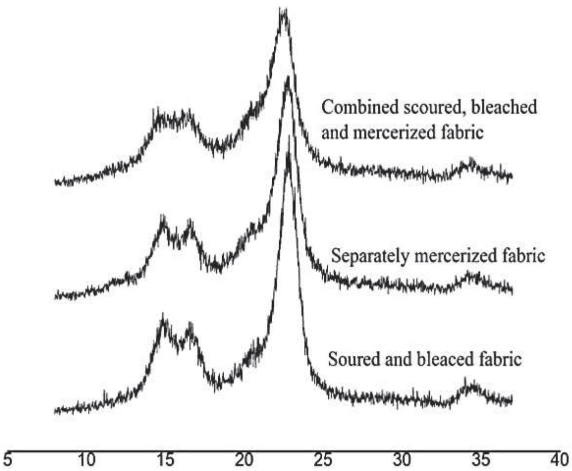


Figure 5: XRD diagram of samples S1, S2 and S3

3.3 Barium activity number

The most widely used method to determine the extent of mercerisation is the barium activity number. Table 2 shows the barium activity number of both S2 and S3.

Table 2: Barium activity number of S2 and S3

Samples	Barium activity number
S2	155
S3	141.5

A comparison of S2 and S3 indicated that combined scouring, bleaching and mercerising results in a lower barium activity number. Although the value of the barium activity number is lower, it is still within the standard range (130–150). This lower value can be explained by the higher temperature and lower concentration of caustic soda.

Table 1: Summary of information from XRD diagram

Samples	Crystallinity [%]	Crystalline phase	Total area [arbitrary units]
S1	69.58	I	78282
S2	61.24	II	77297
S3	57.53	II	72134

3.4 Bursting strength

Table 3 shows the bursting strength of the three tested samples. The bursting strength of both S2 and S3 is higher than S1. The increase in strength can be attributed to the alleviation of internal stresses and the de-twisting of the normal ribbon-like structure of fibre due to the swelling process. The bursting strength of S2 is higher than that of S3. The reason lies in the lower concentration of caustic soda and tension during mercerisation. A higher degree of orientation can be achieved if the fabric is kept under tension during mercerisation. High temperature is also somewhat correlated to strength, as high-concentration alkali can cause harm to the fibre at high temperatures. It is a well-known fact that mercerisation increases the strength of cotton fabric. In both processes (S2 and S3), the strength of fabric increased significantly, clear evidence of the effectiveness of combined scouring, bleaching and mercerisation.

Table 3: Bursting strength of different fabrics

Samples	Bursting strength [kg/cm^2]
S1	10.33
S2	11.98
S3	10.68

3.5 Change in colour strength

Figure 6 shows the *K*/*S* value of all three samples dyed with vinyl-sulfone reactive dye at different concentrations. S2 and S3 showed a higher *K*/*S* value than S1 for all shade percentages. An X-ray diffraction diagram and scanning electron microscopy show that significant changes occurred in the structure of the fabrics due to the treatment. These structural changes had a remarkable effect on the colour strength of the dyed cotton fabric. As the crystallinity of both mercerised fabrics decreased, dye molecules easily penetrated the fibre. A comparison of the two mercerised fabrics (S2 and S3) indicate that the separately mercerised fabric (S2) demonstrated a higher *K*/*S* value because of its higher crystallinity relative to the other fabric. This can be attributed to the crystallite size and crystalline phase, i.e. the orientation of fibre structure. S2 has a higher crystallite size and higher amount of cellulose II in the structure of fibre than S3. Although there is little difference between S2 and S3, both demonstrated a higher *K*/*S* value than the un-mercerised fabric (S1).

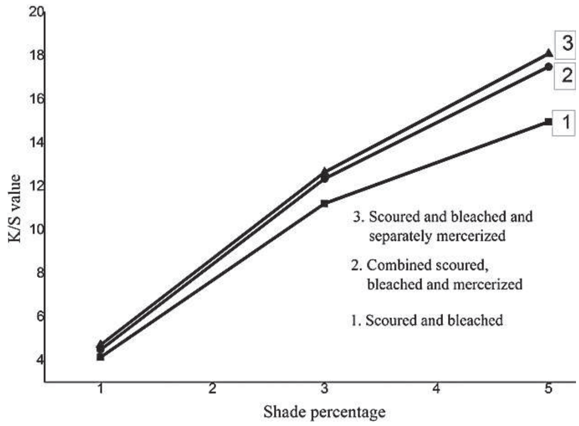


Figure 6: K-S value of samples

There was strong evidence that mercerisation can also be achieved by using high-concentration caustic soda during scouring and bleaching.

4 Conclusion

Cotton single jersey knitted fabric was mercerised using a new process, and the effect of that process on several physical properties of treated fabric successfully validated. The effectiveness of combined scouring, bleaching and mercerisation was proven through surface morphology, fine structure (crystallinity, crystallite size and crystalline phase) and barium activity number. The *K*/*S* value of all three fabrics dyed in different concentrations was also evidence of the effect of the combined process. An SEM surface imagery analysis of all the samples indicated the effect of the new method on smoothness and lustre due to the round-like structure. The decrease in crystallinity, the conversion of cellulose I to cellulose II and crystallite size confirm the effectiveness of the process. The barium activity number of the two mercerised fabrics falls within the range of standard values. The improvement in the bursting strength and *K*/*S* value of the dyed fabrics prove that combined scouring, bleaching and mercerising enhance physical properties to the same extent as separately mercerised fabric. Further research is required regarding this sustainable approach to textile processing. Additional analysis and validation of changes in the dimensions and density of the treated fabric can be enhanced during advanced studies.

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