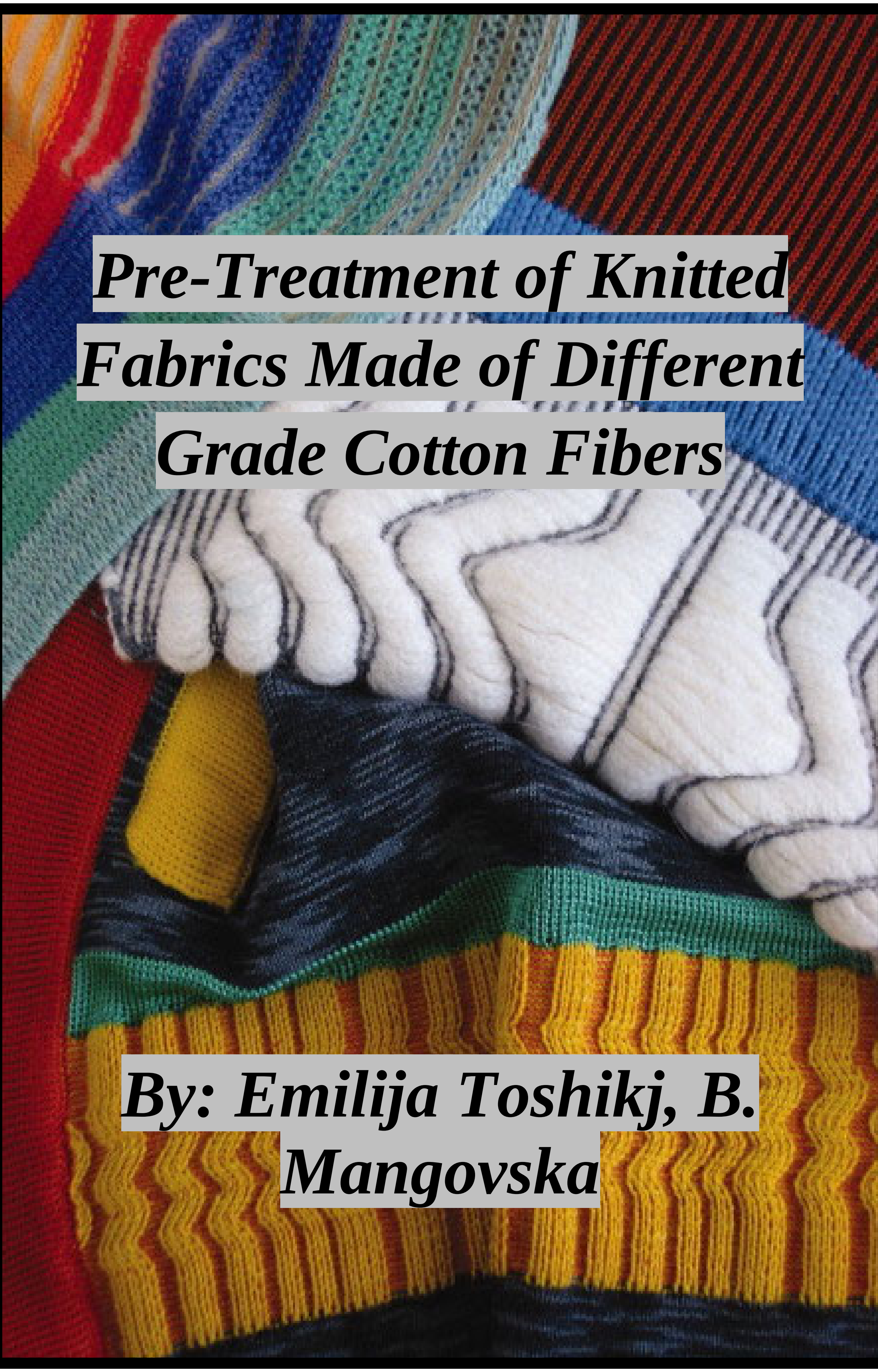

# Pre-Treatment of Knitted Fabrics Made of Different Grade Cotton Fibers

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## Abstract

*Pre-treatment process of cotton knitted fabrics made from different grade cotton fibers with variable physicochemical properties using a single recipe was investigated to obtain a more comprehensive picture about the reproducibility and repeatability of the process. Single jersey knitted fabrics with different characteristic properties, were subjected to same pre-treatment processes. This, pre-treatment processes included acid demineralization and one bath scouring, peroxide bleaching and optical brightening. The fabrics made of different grade cotton fibers were monitored for metallic impurities, as well the whiteness index, and colour coordinates during different steps of finishing and washing. The stability of whiteness after first and fifth washing cycles with detergent for white programme carried out in industrial washing machine was analyzed by Berger scale and colour coordinates and compared with bleached-thermo stabilized knitted fabric. The results obtained showed that the pre-treatment process of demineralization followed by one bath scouring, peroxide bleaching and optical brightening is stable giving knitted fabrics with whiteness index with variance coefficient less than three in all three different grade of cotton. Knitted fabrics after first washing cycle have the same whiteness index as before.*


**Key words:** *cotton, pre-treatment, demineralization, scouring-bleaching, whiteness.*

## 1. Introduction

The importance of knitted fabrics from cotton fibers has increased with the growing demand for casual-, sports- and leisure-wear fabrics which require a high comfort factor [1]. But cotton and textile mills, in particular the pre-treatment, bleaching, dyeing and finishing sectors find themselves dealing with cotton of different origins and with different qualities, as well as physical and chemical properties. Exposure to various elements in the environment and contamination from geology of the cultivation area, weather conditions during maturing period, cultivation techniques and fertilizers, the use of chemical pesticides, de-foliants, harvesting techniques all cause variations in the colour and impurities content of raw cotton. The structure of the primary cell wall and cuticle of cotton fiber, particularly the outermost surface layer, has a determinant influence on textile processes [2]. Cotton impurities include protein (1.0-1.9 %), wax (0.4-1.2 %), ash (0.7-1.6 %), pectin (0.4-1.2 %) and others (resins, pigments, hemi-cellulose) (0.5-0.8 %) [3]. Traditionally, the cotton industry placed great emphasis on the colour of raw cotton and regards it as an indication of quality. It is even generally believed that the presence of colour different from normal white indicates there will be some problems in subsequent processing, and the colour of finished goods will be adversely affected. The differences in fabric whiteness and the colour strength are arising from these different grade cotton fibers. These problems are connected with the total level of impurities in cotton. Unless cotton fabrics are uniformly high in wettability, whiteness, absorbency and cellulose content, dyes, chemicals and finishing agents will not be absorbed readily and evenly. Problems of uneven whiteness or colour shade arise after pre-treatment, bleaching and/or dyeing. In order to reduce these differences cotton fabrics must be prepared before dyeing or bleaching by removing impurities, together with the spinning lubricant applied to the yarn before knitting. Pre-treatment, bleaching and/or dyeing does not mean only to impart attractive hue or whiteness but also to attain the quality of the fabric with desired hue, lightness, chroma, levelness as well as reproducibility and stability of the process. The stability of colour or whiteness index is one of the most important requirements of valuable customers. The current study is taken to evaluate the reproducibility and repeatability of the same pre-treatment processes of knitted fabrics made of different grade cotton fibers. To achieve this goal, knitted fabrics made of different grade cotton fibers with different characteristic properties were subjected to the same pre-treatment processes that included acid demineralization and one bath scouring, peroxide bleaching and optical brightening. The whiteness index, and colour coordinates during different steps of finishing and washing were investigated, as well as the metallic impurities in cotton from different sources.

## 2. Experimental

### 2.1. Materials

#### *Cotton fabric and chemicals*

Single jersey weft-knitted fabrics made of combed ring-spun 100 % yarns of three different grades cotton were carried out from circular-bed knitting machines (Orizio JOHN/C, E 28, 30coll and Albi RCU4, E 26, 30coll). Tubular-knitted fabrics were kept on a flat surface for two weeks under standard atmospheric condition and after dry relaxing, structural characteristics (weight per unit area in g/m², thickness in mm, vertical and horizontal density in $cm^{-1}$) were measured. Greige knitted fabrics had following structural characteristics: weigh per unit area from 113 to 156 g/m², thickness from 0.50 to 0.62 mm, vertical density from 18.83 to 24.17 $cm^{-1}$, horizontal density from 11.17 to 13.83 $cm^{-1}$. Pre-treatment process was conducted in industrial conditions by the method of exhaustion. The water used for this process was technical water.

The chemicals used, were hydrogen peroxide (30 % w/w) and $HNO_3$ conc. of laboratory grade. A sequestering agent based on polyphosphonates with individual buffer for regulation of pH media (HEPTOL EMG), a wetting agent (FELOSAN NFG) and a crease preventing agent (BIAVIN BPA) from BEZEMA (Switzerland), stabilizer THORSTABILIZATOR POS and optical brightening agent UVITEX-BHB from Ciba. Sodium hydroxide, hydrogen peroxide (50 % w/w) and acetic acid were chemicals of technical grade. Standard detergent Ariel for white programme was also used (Procter & Gamble).

## 2.2. Methods

### *Acid demineralization*

Acid demineralization treatment of single jersey knitted fabric was conducted as a separate step before the $H_2O_2$ bleaching process. Greige cotton fabrics were treated with an aqueous solution containing 1 % FELOSAN NFG, 1 % BIAVIN BPA, and 2 % HEPTOL EMG using a material to liquor ratio (LR) of 1:10 at 50 °C for 20 min., at pH 3.5. After demineralization treatment, the substrate was rinsed with cold water and submitted to $H_2O_2$ bleaching process.

### *One bath peroxide bleaching and optical brightening*

One bath scouring, peroxide bleaching and optical brightening started at 40 °C in the bath with liquor ratio 1:10 in the presence of 1 % BIAVIN BPA. The bath circulated for 5 min., than 2 % sodium hydroxide and 1 % THORSTABILIZATOR POS were added, the bath circulated for 10 min. and the temperature was raised to 50 °C, 10 % of $H_2O_2$ 50 % was added, and within 10 min. the temperature was raised to 70 °C and 0.8 % UVITEX-BHB was added. The temperature was further raised to 98 °C at a gradient of 3 °C/min. and bleaching and optical brightening proceeded for 90 min. The fabric was then rinsed twice with hot water at 80 °C and 60 °C, then with cold water, where the magnitude of pH was 10.2, 9.9 and 8.7, respectively. After these processes, knitted fabrics were subjected to softening with 3 % Dimicol Soft 6073 and 0.5 % $CH_3COOH$, drying and thermo stabilization by compacting.

## 2.3. Testing and analysis

***Metals determination:*** Preparation and analysis of samples by ashing followed the procedure previously reported [4]. Analysis were conducted using an atomic absorption (Perkin Elmer 700) spectrophotometer with an air/acetylene flame in the absorbance mode. Individual metal concentrations were determined from the calibration curves constructed with minimum of three standard concentrations. Dilutions made to obtain effective absorbencies at each metal respective wave lengths in their linear operating ranges were 1250X for potassium and 625X for both calcium and magnesium and 6000X for sodium analysis.

***Fastness to washing:*** The thermo stabilized knitted fabrics were subjected to washing in the industrial washing machine with detergent for white programme. The 35x35 cm specimens of different grade cotton knitted fabrics were cut and marked. The change of the degree of whiteness of the specimens before and after 1 and 5 washing cycles were assessed by colour coordinates and whiteness index.

***CIE Lab coordinates and whiteness index:*** The degree of whiteness and CIELab coordinates such as $a^*$ and $b^*$ were measured on X-RITE CA 22 spectrophotometer

(D65 light source, observed $10^{o}$). The degree of whiteness was expressed as Berger whiteness.

***The water absorbency test*** was measured by AATCC Test Method of Evaluation of Wettability which determines fabric wettability by counting the elapsed seconds between the contact of water drop with the fabric and the disappearance of the drop into the fabric [5].

## 3. Results and discussion

The object of the pre-treatment process of demineralization followed by one bath scouring, peroxide bleaching and optical brightening is to remove the alkali, alkaline-earth and heavy metals ions by demineralization stage, and remove other cotton impurities during alkaline scouring-bleaching and optical brightening stage. Therefore, to obtain a more comprehensive picture about reproducibility and repeatability of the same pre-treatment process performed in industrial conditions, knitted fabrics from different grade cotton fibers were used. The reproducibility and repeatability of pre-treatment process between all the substrates under investigation (three different grades of cotton fibers) was determined by statistical calculation after each stage.

The values of colour coordinates and whiteness index before and after each treatment were statistically analyzed in order to validate each pre-treatment process in covering the differences in knitted fabrics from different grade cotton by statistical calculation of the mean deviation (M.D) and the variance coefficient (V.C). The colour coordinates and whiteness index values of greige knitted fabrics made of different cotton fibers, as shown in figures 1 and 2, have $a^*$ values lower or near to 1, $b^*$ values between 10.61 to 12.34 and whiteness index values ranged between 16.63 to 22.63. The yellowness that indicates the degree of colour pigmentation of the cotton fibers is expressed as $b^*$ determined by the CIELab system. A positive value of $b^*$ represents an increase in the degree of yellowness. Greige knitted fabrics under investigation are characterized by negligible red colour shade and the presence of a yellow colour shade. Knitted fabrics of grade cotton 2 have lower value of $b^*$ and higher value of witness than knitted fabrics of grade cotton 1. As witness index increased, the $b^*$ values on the fiber decreased. The correlation coefficient between the $b^*$ values and W values of all greige samples was found to be $r = -0.95$. Knitted fabrics of grade cotton 2 have lower value of $b^*$ with higher degree of whiteness then knitted fabrics of grade cotton 1 (Fig. 1). Metallic impurities in greige knitted fabrics require appropriate pre-treatment to reduce their presence on the fiber as much as possible before bleaching.

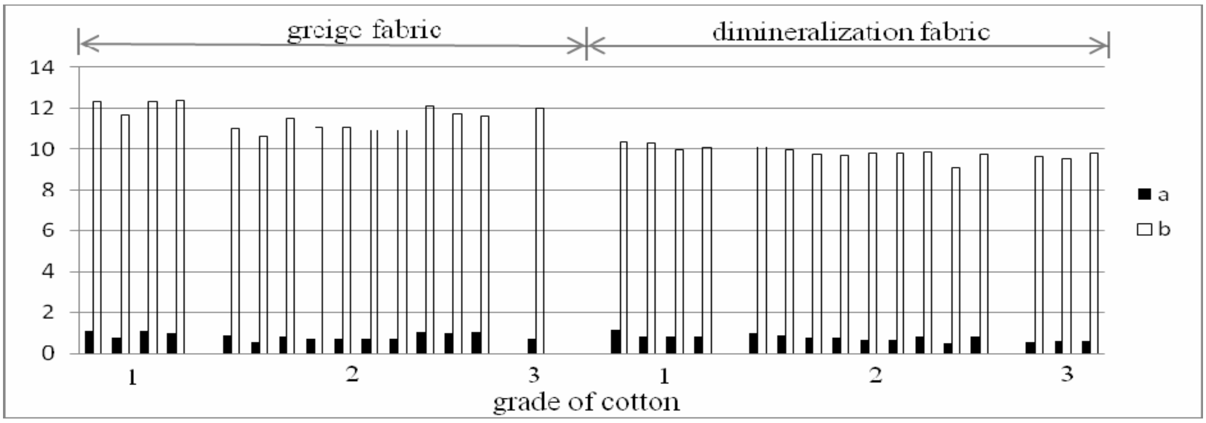


**Figure 1** Colour coordinates results of greige and demineralization cotton knitted fabrics from different grade cotton fibers

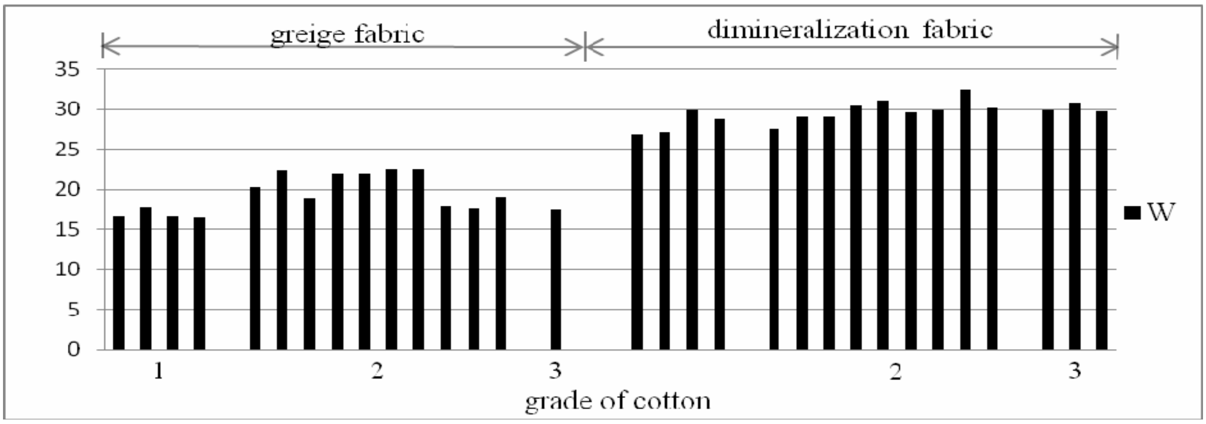


**Figure 2** Berger whiteness index results of greige and demineralization cotton knitted fabrics from different grade cotton fibers

If calcium and magnesium ions are not eliminated, there is a strong possibility of their combining with natural soaps which have been generated during the alkaline scouring process, to form waxy substances. These have been referred to as lime soap deposits. They can deposit not only on the substrate itself, but also on the surfaces of the machinery. In addition to the re-deposition of waxy substances, there is a further risk of white deposits of calcium and magnesium hydroxide onto the cotton goods which can detract from appearance, absorbency, handle and frictional properties. There are further dangers which relate specifically to the peroxide bleaching of cotton. There is a need to promote, and ensure, a controlled rate of release of active peroxy radical. This being the transient species which promotes the removal of seed coat fragments and colour. The presence of iron, copper or manganese, even in trace amounts, can cause catalytic and uncontrolled decomposition of hydrogen peroxide. This can result in inefficient use of peroxide, reduced degree of whiteness, loss of tensile strength of the fiber, pinhole damages. Potassium, which is the most abundant metal on cotton fiber, usually accounted for 60 to 75 % of the light metal content of the fiber [6] and it exists in or on fiber in form of highly water-soluble salts [7-8]. About 85 % of carboxyl groups present in the cotton pectin are methylated, the rest available in the form of insoluble calcium, magnesium and iron salts of polygalacturonic acid, which contribute to the hydrophobic characteristics of raw cotton [9]. The effective removing of mineral content comprised better levelness and more brilliance, lower peroxide consumption, increase in degree of whiteness, regular decomposition of peroxide and no catalytic damage of the fibers [10]. The concentration of Ca, Mg, Na and K ions in the all three different grade cotton fibers are shown in the Table 1. The amounts of metallic cations present on the cotton fiber vary for Ca from 540 to 2711 ppm, Mg from 334 to 1119 ppm, Fe from 12 to 300 ppm, Cu from 1 to 6 ppm, Mn from 1 to 36 ppm [11]. The results of the Table 1 indicate that concentrations of Ca and Mg ions in the examined cotton knitted fabrics were in low range.

**Table 1** Metal contents of three different grade raw cotton fibers and their statistical deviation; (a) M.D = Mean deviation, V.C = Variance coefficient

| **Metallic ion (ppm)** | **Substrate number** | | | **Statistical analysis** | |
|---|---|---|---|---|---|
| | **1** | **2** | **3** | **M.D** | **V.C** |
| **Ca** | 771.75 | 850.00 | 815.75 | 39.23 | 4.83 |
| **Mg** | 479.75 | 462.50 | 449.00 | 15.41 | 3.32 |
| **K** | 3417.50 | 3630.00 | 3317.50 | 159.59 | 4.62 |
| **Na** | 7442.50 | 8061.25 | 7642.50 | 315.75 | 4.09 |
| **Ca+Mg+K+Na** | 12111.5 | 13003.75 | 12224.75 | 485.76 | 3.90 |

Demineralization stage enhanced whiteness index values for about 10 units, and decreased the $b^*$ values among all the prepared samples of different grade cotton fibers compared with greige knitted fabrics. The results obtained are set in figures 1 and 2. The average $a^*$, $b^*$ and W values after demineralization stages were lower than 1, between 9.10 and 10.37 and between 26.90 and 32.4, respectively. The amount of alkaline earths and heavy metal ions after demineralization stage is not evaluated because in greige fabrics they were in lower range, as well as in the stages of demineralization they were probably fully removed. The increased degree of whiteness for about 10 units is probably due not only to the removal of metallic ions some probably incorporated with some pigments but also partial elimination of some cotton impurities and partial degradation of coloured substances in strongly acidic media. Although a demineralization stages are associated with removing of some cotton impurities the wettability was low. The time of the disappearance of a drop of distilled water was above 180 s, indicating presence of waxy substances and other hydrophobic impurities on the surface of cotton.

The fabrics were than subjected to one bath scouring, peroxide bleaching and optical brightening using exhaustion technique previously described. Many impurities are removed by alkaline scouring with the exception of natural colouring matters, which can be removed by bleaching. Under the influence of alkali, cotton fibre undergoes some physical changes, it swells, thus facilitating all colloid-chemical processes. Such treatment tends to remove the non-cellulose material by turning pectin into sodium pectat, proteins into soluble salts of different amino acids, solubilizes the ash and certain proportion of lignified substances and almost completely removes the seed coat fragments. Part of cotton proteins in turn of protoplasmatic residues, that are thought to cause cotton colouration, are hydrolyzed which results in higher degree of whiteness between 116.13 to 125.5 while $b^*$ values ranged from -7.75 to -8.6.

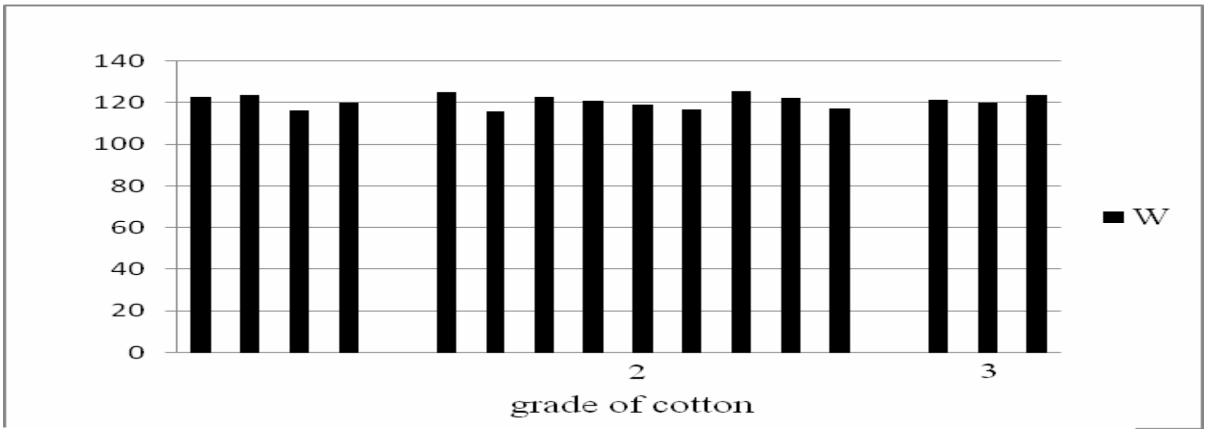


**Figure 3** Whiteness index results of bleached-thermostabilized cotton knitted fabrics from different grade cotton fibers.

The reproducibility and repeatability of pre-treatment process among all the grade cotton fibers under investigation (three cotton substrate in our case) was evaluated by calculating the mean deviation (M.D) and variance coefficient (V.C) of the witness (W) and colour coordinates after different steps of finishing and washing. The results obtained are set in tables 2 and 3.

**Table 2** Effect of same pretreatment process on average colour coordinates and whiteness index of knitted fabrics made of different grade cotton fabrics during different steps of finishing

| Treatments | Substrate Number | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | | | | 2 | | | | 3 | | | |
| | No | $a^*$ | $b^*$ | W | No | $a^*$ | $b^*$ | W | No | $a^*$ | $b^*$ | W |
| **Greige** | 4 | 0.97 | 12.16 | 16.95 | 10 | 0.81 | 11.24 | 20.50 | 1 | 0.71 | 12.00 | 17.56 |
| **Demineralisation** | 4 | 0.90 | 10.17 | 28.19 | 9 | 0.75 | 9.74 | 29.94 | 3 | 0.61 | 9.66 | 30.15 |
| **Bleaching** | 4 | 2.15 | -8.36 | 123.34 | 9 | 2.27 | -8.43 | 123.34 | 3 | 2.19 | -8.44 | 121.66 |
| **Softening** | 4 | 2.16 | -8.34 | 123.43 | 10 | 2.26 | -8.40 | 122.73 | 3 | 2.21 | -8.44 | 122.35 |
| **Thermostabilization** | 4 | 2.24 | -8.20 | 120.80 | 9 | 2.23 | -8.17 | 120.77 | 3 | 2.22 | -8.45 | 121.78 |
| **After 1st washing** | 4 | 1.59 | -7.66 | 119.52 | 10 | 1.67 | -7.73 | 120.37 | 3 | 1.63 | -7.56 | 118.56 |
| **After 5th washing** | 3 | 1.96 | -7.18 | 112.02 | 10 | 1.89 | -7.19 | 112.44 | 3 | 1.94 | -7.30 | 118.08 |

**Table 3** Effect of pretreatment process on statistical deviation in colour coordinates and whiteness index; M.D = Mean deviation, V.C = Variance coefficient

| Treatments | Statistical Analysis | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | | | | | | 2 | | | | | | 3 | | | | | |
| | $a^*$ | | $b^*$ | | W | | $a^*$ | | $b^*$ | | W | | $a^*$ | | $b^*$ | | W | |
| | M.D | V.C | M.D | V.C | M.D | V.C | M.D | V.C | M.D | V.C | M.D | V.C | M.D | V.C | M.D | V.C | M.D | V.C |
| Greige | 0.13 | 12.37 | 0.35 | 2.89 | 0.54 | 3.2 | 0.15 | 18.38 | 0.45 | 3.99 | 2.02 | 9.87 | 0.05 | 7.02 | 0.02 | 0.19 | 0.11 | 0.66 |
| Demineralisation | 0.14 | 15.84 | 0.2 | 2.02 | 1.41 | 5.03 | 0.13 | 17.64 | 0.27 | 2.78 | 1.38 | 4.61 | 0.04 | 6.21 | 0.12 | 1.28 | 0.56 | 1.86 |
| Bleaching | 0.1 | 4.97 | 0.48 | 5.70 | 1.72 | 1.4 | 0.16 | 6.95 | 0.23 | 2.73 | 1.72 | 1.40 | 0.08 | 3.62 | 0.26 | 3.08 | 1.55 | 1.28 |
| Softening | 0.05 | 2.31 | 0.51 | 6.10 | 2.86 | 2.31 | 0.17 | 7.62 | 0.21 | 2.49 | 2.00 | 1.63 | 0.09 | 4.28 | 0.21 | 2.54 | 1.96 | 1.60 |
| Thermostabilization | 0.21 | 9.47 | 0.32 | 3.91 | 3.21 | 2.66 | 0.21 | 9.43 | 0.26 | 3.25 | 3.45 | 2.86 | 0.01 | 0.69 | 0.10 | 1.23 | 1.84 | 1.51 |
| After 1st washing | 0.09 | 5.42 | 0.3 | 3.96 | 3.03 | 2.54 | 0.17 | 10.11 | 0.49 | 6.29 | 2.77 | 2.3 | 0.04 | 2.67 | 0.31 | 4.18 | 2.94 | 2.48 |
| After 5th washing | 0.26 | 13.26 | 0.11 | 1.53 | 3.22 | 2.87 | 0.13 | 6.88 | 0.32 | 4.45 | 2.93 | 2.60 | 0.06 | 3.09 | 0.06 | 0.82 | 3.64 | 3.28 |

The acid demineralization followed by one bath scouring, peroxide bleaching and optical brightening decreases the variance coefficient (V.C) among the sample. Knitted fabrics before pre-treatment show a wide range of whiteness index with higher magnitude of variance coefficient, indicating low uniformity in colour shade among all the samples. The analysis of the results indicated the highest variance coefficient values of greige knitted fabrics made of grade cotton 2 followed by grade cotton 1 and 3. The results in tables 2 and 3 showed that the magnitude of V.C values of whiteness index between all three different knitted fabrics (cotton grade 1, 2 and 3) decreases from 3,20, 9.87 and 0.66 to 2.66, 2.86 and 1.51, respectively. The results obtained showed that the pre-treatment process of demineralization followed by one bath scouring, peroxide bleaching and optical brightening is stable giving knitted fabrics with whiteness index with variance coefficient less than three in all three different grades of cotton. The effect of different pre-treatment process on different types of cotton fibers was demonstrated by the rather extensive studies of Hashem M. M. [12]. He has come to the conclusion that reductive scouring of cotton fabric using D-glucose along with sodium hydroxide and acid demineralization with D-gluconic acid, as well as acid pectinase scouring provided similar effectiveness with variance coefficient less than 2 % regardless of the grade of cotton.

The influence of the detergent for a white product on whiteness after first and fifth washing cycles at 90 °C was analyzed by colour coordinates and whiteness index and than compared with thermo-stabilized knitted fabric. The results obtained are set in tables 2 and 3. These results show that the knitted fabrics after first washing cycle have good stability because no significantly changes in whiteness were observed. Knitted fabrics after first washing cycle have the same whiteness index as before. The colour coordinates indicated the average $a^*$ values among grade cotton fibers 1, 2 and 3 decrease to 1.59, 1.67 and 1.63, respectively, while $b^*$ values enhance to -7.18, -7.19 and -7.30 regardless to the grade of the cotton. It can be observed from the Table 2 that there is a significant difference between the whiteness after first and fifth washing cycles and there is a gradual decrease in whiteness index values among the grade of cotton. The whiteness index of grade cotton 1 and 2 decrease to 112.02 and 112.44, respectively. This may be due to the partial removal of the optical brightening agency during washing cycle.

## 4. Conclusion

From the analysis of the results described above, it may be concluded that pre-treatment process, demineralization followed by one bath scouring, bleaching and optical brightening used as single recipe to yield single results is most effective and good appropriate formulation that can be applied to cotton knitted fabrics and reduced the differences in staining and whiteness index arising from different fiber grade, as well as stable and reproducible method giving knitted fabrics with variance coefficient less than three without fabrics pinhole damage.

## ■ Acknowledgment

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