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Achieving the mercerization effect in cotton knitted fabric by high concentration NaOH in scouring and bleaching in exhaustion method

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Abstract-Single jersey cotton knitted fabrics are scoured and bleached using high concentration NaOH in order to attaining the effects of mercerization in a solitary process which can be referred to as combined scouring, bleaching and mercerizing process.The aforementioned combined processed fabric's surface morphology, fine structure, barium activity number, bursting strength, K/S value of dyed fabric were scrutinized. These properties are compared with the properties of a regular scoured and bleached fabric and a separately bleached fabric to manifest the potency of the process. Fabric treated with new combined process can be characterized by smoother, lustrous surface than the typical scoured and bleached fabric and almost same as separately mercerized fabric. It exhibits decrease in crystallinityand conversion of crystalline phase from cellulose I to cellulose II in similar manner to the separately mercerized fabric. Barium activity number, bursting strength and improvement of K/S value are also in line with the separately mercerized fabric. Above mentioned properties of combined scoured, bleached and mercerized fabric well evident the effectiveness of the process.

Index Terms— Mercerization, surface morphology, XRD, barium activity number, crystallinity, bursting strength.

1 INTRODUCTION

Mercerization is the most well-known method to enhance physical and dyeing properties of cotton fiber. Mercerization changes the surface morphology, fine structure i.e. crystallinity, crystallite size etc. of cotton fiber(Saapan et al. 1984; Wakida et al. 2000a).It converts the cellulose chain from cellulose I to cellulose II(Bertoniere and King 1989; Haga and Takagishi 2001; Rousselle et al. 1976). A significant improvement in dye affinity, color strength, luster, tensile strength, smoothness of cotton fabric also occurred due to mercerization(Haga et al. 2000; Saapan et al. 1984; Sameii et al. 2008). The degree of changes depends upon the different parameters i.e. processing time, processing temperature, tension, concentration of caustic soda etc.(Wakida et al. 2002b).Mercerization of knitted fabric was considered to impracticable because of the stretching during mercerizing process but it is essential to enhance the properties of knitted fabric(Greenwood 1987). Nowadays different manufacturers design machine for knit fabric mercerization without harming the dimensional stability of fabric. Both open width and tubular fabric can be mercerized with the new invented machinery without harming the quality of fabric. Over a long period of time high concentrated NaOH solution (14% or higher) has been used very widely to modify cellulosic fiber.

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This modification of the cellulosic fabric improves dye receptivity of the cotton and reduces shrinkage of the cotton fabric(Saapan et al. 1984). The development of slack mercerization solves many problems of knit fabric mercerization(Ziifle et al. 1959). In slack mercerization fabric is treated with high concentration caustic soda without tension and excellent physical and dyeing properties can be achieved by this method as variation in tension has very little or no effect on the fine structure of cotton fiber(Haig Zeronian et al. 1990; Kokot and Stewart 1995). A Mercerization at high temperature is now well established method(Montazer and Sadighi 2006). In the method of high temperature mercerization, penetration of caustic soda in the fiber structure is more rapid(Wakida et al. 2000b).High temperature mercerization provides more uniform treatment but less swelling is occurred(Wakida et al. 2002b). This can enhance the luster without affecting the strength of the fiber. At high temperature and high concentration caustic the fabric become plastic in nature and due to low elastic in nature stretch readily resulting high degree of improvement in properties(Wakida et al. 2002a). Several studies reported combined scouring and mercerizing at high temperature but machine and processing is quite different from the as usual one.

In exhaustion method of knit fabric dyeing, the fabric is prepared by combined scouring and bleaching using caustic soda, hydrogen peroxide, stabilizer and some others auxiliaries. From the compatibility chart of dyes and chemicals, it has been found that caustic soda is very much compatible with hydrogen peroxide. This work is the combination of slack mercerization more specifically mercerization without tension, mercerization at high temperature and the compatibility of caustic soda with hydrogen peroxide. During scouring and bleaching of knit fabric dyeing in exhaustion method a high concentration of caustic soda is used at high temperature which is referred to as combined scouring, bleaching and mercerizing. As a part of the investigation, the properties of fabric pretreated with combined scouring, bleaching and mercerizing are compared to separately mercerized fabric and also only scoured and bleached fabricindividually. The changes in surface morphology, fine structure, physical property strongly evident the effectiveness of combined scouring, bleaching and mercerizing.

2 Methodology

2.1 Materials

Three single jersey knit fabrics of same construction are prepared for the investigation. The first one is prepared by conventional scouring and bleaching in a sample winch machine (Dilmenler, HTHP, Turkiye). Preparation of second sample involved conventional scouring and bleaching same as the first one followed by caustic mercerization at room temperature. The final one is also prepared in the same machine by keeping all the processing parameter same as the first one and first stage of second one.**Figure 1** represents the process curve of the preparation of all samples. For the preparation of first one the fabric was treated with 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 stabilizer. In the first stage of second sample preparation all the chemicals and the concentration of all chemicals were also same as the first one and in second stage the scoured and bleached fabric was treated with 20% NaOH solution for mercerization. For the preparation of third one all the chemicals were same as first sample and first stage of second sample and the concentration of chemicals were also same except the concentration of NaOH. To bring out the effect of mercerization high concentration (16 g/l) of NaOH was used during scouring and bleaching which is referred to as combined scouring, bleaching and mercerizing in this work. For further investigation all three types of fabric were connected together and dyed with vinylsulfone reactive dye in exhaustion method in the same bath to keep all the processing parameters same. For achieving precision in result, dyeing was done with three shade percentage i.e., 1%, 3% and 5%.

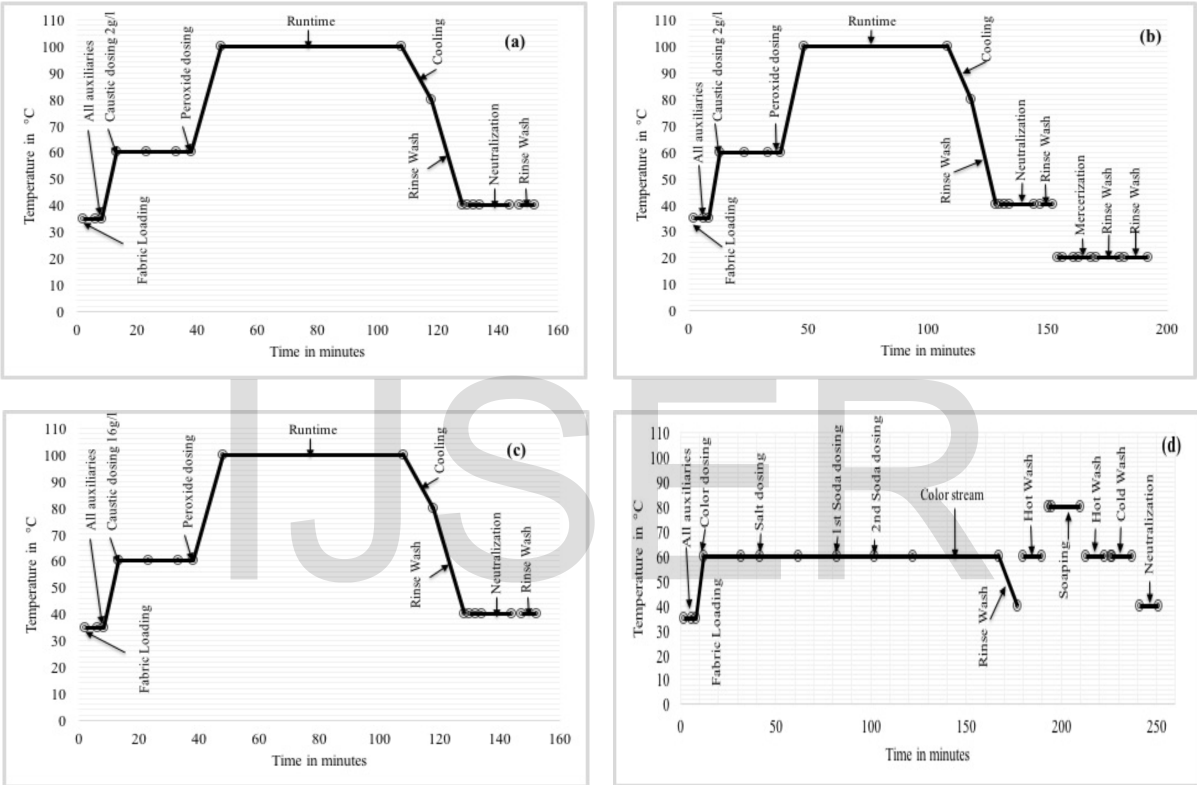

Figure 1 Process curve (a) Scouring and bleaching (b) Scouring bleaching and separately mercerizing (c) Combined scouring, bleaching and mercerizing (d) Dyeing of all types

2.2 Scanning Electron Microscopy

JEOL, JSM-6510 scanning electron microscope was used to see the changes of surface morphology. During scanning microscopy, accelerating voltage of 20 KV and magnification of 2000X and 3000X were used.

2.3 X-ray Diffraction

To find out fine structural changes X-ray diffractometer MDI, JADE-6 was used.The representation of crystallinity is done by the ratio of integrated crystallite scattering intensity to total scattering intensity ranging from 10 to 36°. The amount of crystallinity and amorphous fraction is determined by the synthesized curves to diffractograms obtained from the experiment through successive approximation. Lorentzian distributions which represent the crystalline component peaks, amorphous component and contribution due to air scatter of X-ray beam were calculated by using programmable calculator.

2.4 Barium activity number

To determine the degree of mercerization, barium activity number was measured by the test method AATCC 89-1998. Samples were taken from all three fabrics and were cut into small pieces. Then 1 g from each type is treated with 30 ml of 0.25 barium hydroxide solution in 100 ml of conical flask for two hours. Then 10 ml from each sample solution was titrated against 0.1 N HCl. To calculate the result, a blank solution of barium hydroxide was also titrated. The result was achieved by the following equation:

$$\text{Barium activity number} = \frac{\text{Titration reading for blank} - \text{Titration reading for mercerized fabric}}{\text{Titration reading for blank} - \text{Titration reading for unmercerized fabric}} X100$$

2.5 Bursting strength

Before testing the bursting strength all the fabrics are conditioned and allowed to become relaxation state. After full relaxation the bursting strength was measured by using bursting strength tester. For each sample the test was carried out for 10 times and average value was taken which is expressed in kg/cm^2.

2.6 Measuring of K/S value

For measuring the K/S value of dyed fabric spectrophotometer (DATACOLOR-650) was used. At first the reflectance percentage of dyed fabric was measured and then K/S value was calculated by using Kubelka-Munk theory. K/S value can be obtained by following equation

$$K/S = \frac{(1 - \text{R})^2}{2R}$$

Where, R is the reflectance percentage, K is the absorbance and S is the scattering.

K/s value of different fabrics dyed with reactive dye in different shade percentage is given below:

Table 1 K/S value of different fabrics dyed with reactive dye at different shade percentage

Shade percentage	Scoured and bleached	Scoured and bleached and separately mercerized	Combined scoured, bleached and mercerized
1	4.14	4.71	4.49
3	11.21	12.64	12.35
5	14.96	18.086	17.486

3 Result and Discussion

3.1 Change in surface morphology

It is well established that due to caustic mercerization the natural deep wrinkles from the surface of the cotton fiber tend to be vanished.The cell wall of the fiber becomes thicker depending upon the maturity and natural twisted-ribbon like structure tend to be changed and form a round like structure.

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Porous or granular appearances appeared on the surface of the fiber. Figure 1displays the surface image of conventional scoured and bleached fabric at the magnification of 2000X, 3000X and 5000X respectively. The figure clearly represents the twisted ribbon like structure of cotton fibers. From the figure, the fibers are also characterized by innumerable natural wrinkle on the surface.

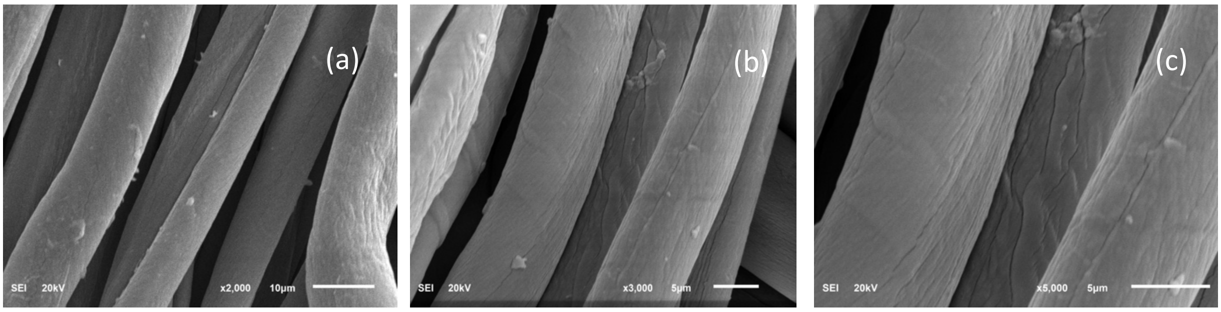

Figure 2 SEM image of conventional scoured and bleached fabric (a) 2000X (b) 3000X (c) 5000X

Figure 2 represent the surface image separately mercerized fabric and Figure 3 represent the surface image of combined scoured, bleached and mercerized fabric at 2000X, 3000X and 5000X magnification. These two Figures clearly represent that fiber become round like structure instead of twisted-ribbon like structure. The natural deep wrinkles from the surface removed significantly.

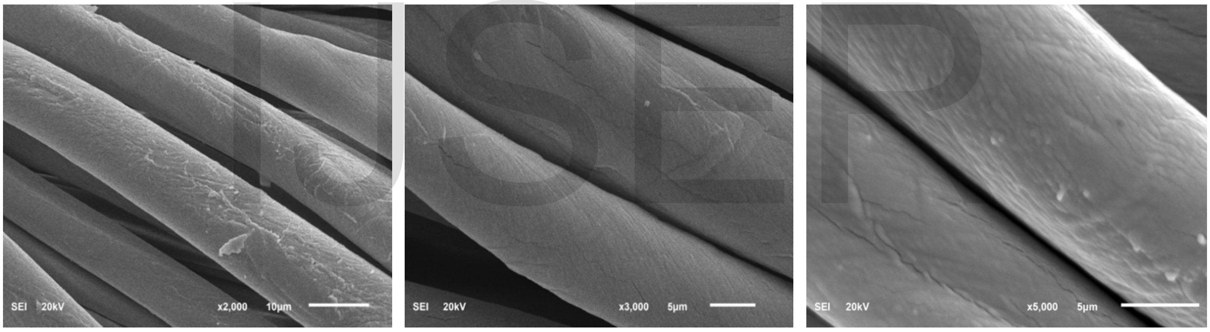

Figure 3 SEM image of conventional scoured and bleached and separately mercerized fabric (a) 2000X (b) 3000X (c) 5000X

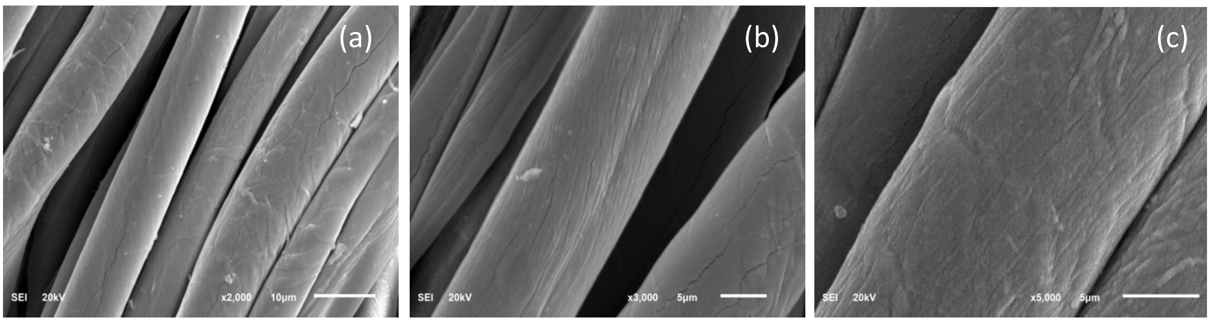

Figure 4 SEM image of combined scoured, bleached and mercerized fabric (a) 2000X (b) 3000X (c) 5000X

In both cases fiber structure can be characterized by smoother surface than the previous one. Smoother surface permitted more regular reflectance which resulting more lustrous surface of the fabric. The luster of the combined scoured, bleached and mercerized fabric is far better than the conventional scoured and bleached fabric. In compared to combined scoured, bleached and mercerized fabric and separately mercerized fabric, there is no significant difference found in terms of luster.

3.2 Changes in fine structure

When cotton fiber treated with high concentration NaOH then it forms a cellulose-soda-water compound. During the destruction of this compound, cellulose converts from cellulose I to cellulose II. This conversion of cellulose completely depends upon the condition of treatment. Most of the converted cellulose does not come back to its original position which resulted increase in amorphous region in cotton fiber and decrease in crystallinity.

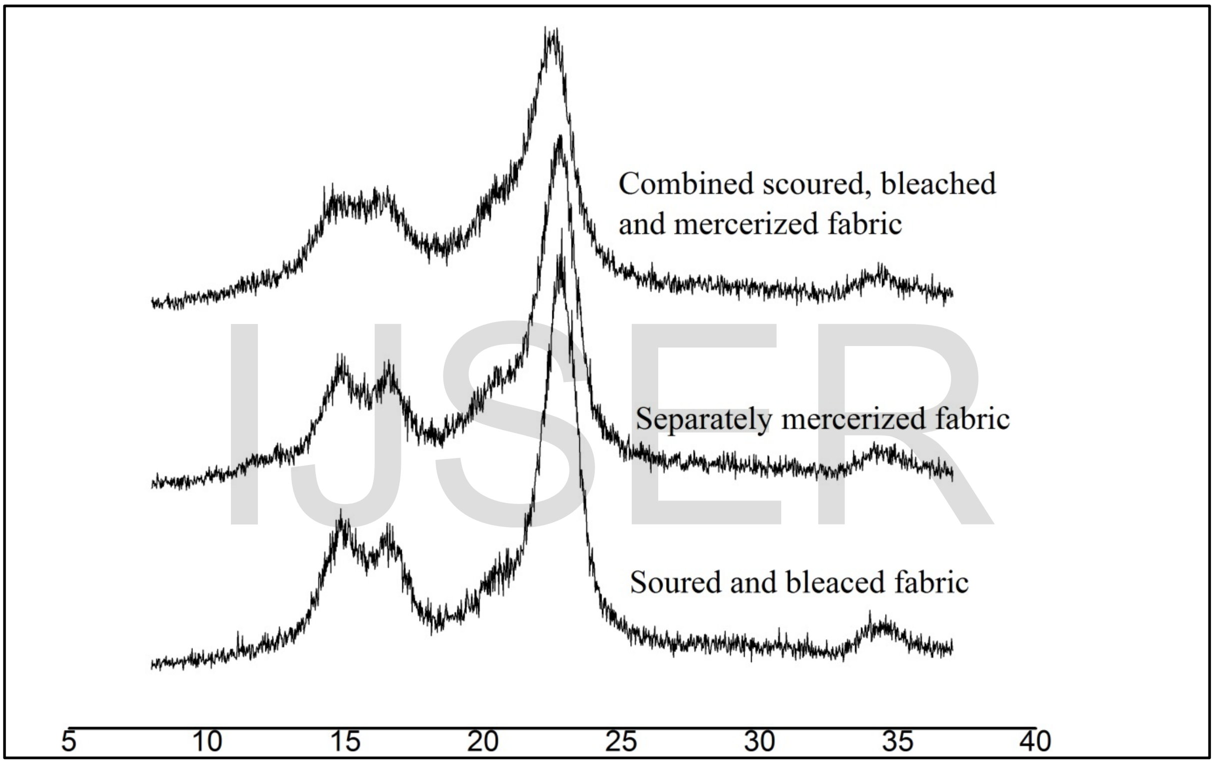

Figure 5 XRD diagram of three different fabrics

The XRD diagram of conventional scoured and bleached fabric, separately mercerized fabric and combined scoured, bleached and mercerized fabric is presented in the **Figure 4**. It is seen that conventional scoured and bleached fabric shows normal crystallinity. In case of separately mercerized fabric crystallinity reduces significantly due to penetration of high concentration caustic into the highly oriented crystalline region. The same phenomenon occurred in case of combined scouring, bleaching and mercerizing.

Table 2 Summary of information from XRD diagram

Fabric type	Crystallinity %	Crystalline phase	Total Area

Scoured, bleached	69.58	I	78282
Scoured and bleached and separately mercerized	61.24	II	77297
Combined scoured, bleached and mercerized	57.53	II	72134

The information obtained from the XRD is summarized in Table 2 In comparison among three samples crystallinity of two mercerized fabrics decrease significantly. Combined scoured, bleached and mercerized fabric shows higher decrease in crystallinity than the separately bleached fabric. This can be explained by the differences of temperature. High mercerization temperature decrease the viscosity of NaOH solution and enhance the diffusion property by swelling the highly ordered area of fiber and decreasing hydrophobic nature of natural impurities. But the difference of crystallinityamong these two mercerized fabrics is too little. This is due to the lower concentration of caustic in combined scouring, bleaching and mercerizing than the separate mercerizing. Table 2shows that both two mercerized fabric exhibit crystalline phase of cellulose II. In separately mercerized fabric amount of cellulose II is higher than the other one. The extent of decrease in crystallinity, conversion of cellulose I to cellulose II strongly evident that the effectiveness of combined scouring, bleaching and mercerizing.

3.3 Barium activity number

The most widely used method to determine the extent of mercerization is the barium activity number. Table 3shows the barium activity number of both two mercerized fabrics. In comparison between two mercerized fabrics combined scouring, bleaching and mercerizing gives lower value of barium activity number. Though the value of barium activity number is lower it is still lying between the standard (130-150). This lower value can be explained by the higher temperature and lower concentration of caustic.

Table 3 Barium activity number of two mercerized fabrics

Fabric type	Barium activity number
Scoured and bleached and separately mercerized	146
Combined scoured, bleached and mercerized	141.5

3.4 Bursting strength

Table **4**displays the bursting strength of differently treated fabric. The bursting strength of both two mercerized fabrics is higher than the conventional scoured and bleached fabric. The increase in strength can be described by the alleviation of internal stresses and de-twisting of normal ribbon like structure of fiber due to the swelling process. The bursting strength of separately mercerized fabric is higher than the combined scoured, bleached and mercerized fabric. The reason behind this is the lower concentration of caustic and the tension during mercerization. Higher degree of orientation can be achieved if the fabric is kept under tension during mercerization. High temperature is also slightly related to the strength because at high temperature high concentration alkali can cause harm to the fiber. It is well established that mercerization increases the strength of cotton fabric. In both two process strength of fabric increased significantly which clearly evident that effectiveness of combined scouring, bleaching and mercerization.

Table 4 Bursting strength of different fabrics

Fabric type	Bursting strength kg/cm2
Scoured and bleached	10.33
Scoured and bleached and separately mercerized	11.98
Combined scoured, bleached and mercerized	10.68

3.5 Change in K/S value

Figure shows the K/S value of all three fabrics dyed with vinylsulfone reactive dye at the concentration of 1%, 2% and 3%. For all shade percentages, both mercerized fabrics show higher K/S value than the un-mercerized fabric. X-ray diffraction diagram and scanning electron microscopy show that there are significant changes occurred in the structure of the fabrics due to the treatment. These structural changes have remarkable effect on the color strength of dyed cotton fabric. As the crystallinity of both mercerized fabrics decreased, dyes molecule can easily penetrate into the fiber.

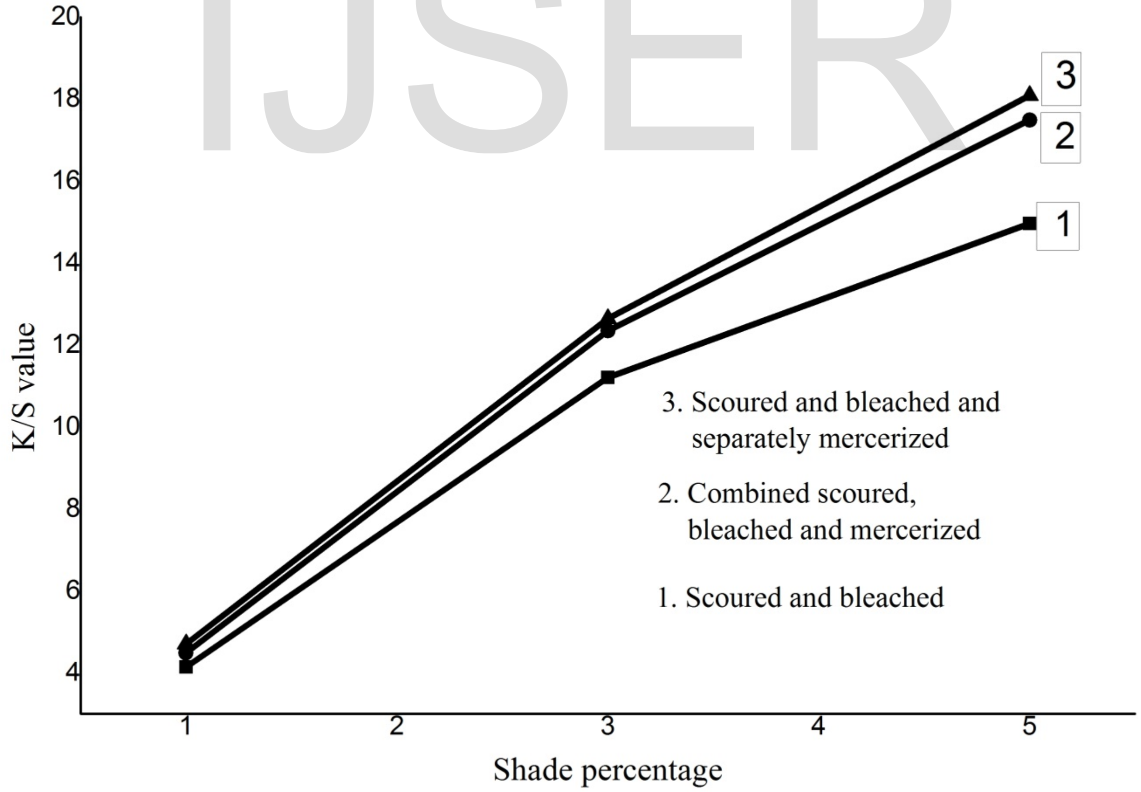

Figure 6 K-S value of different fabrics

In comparison between two mercerized fabrics, the separately mercerized fabric shows higher K/S value though crystallinity is higher than the other. This can be described as the crystallite size and crystalline phase that means the orientation of fiber structure. Separately mercerized fabric has higher crystallite size and higher amount of cellulose II in the structure of fiber than the other one. Though there is little difference between the two mercerized fabrics both show higher K/S value than the un-mercerized one. This is a strong evidence that mercerization can also achieved by using high concentration caustic during scouring and bleaching.

4 Conclusion

Cotton single jersey knitted fabric is mercerized in a new process called combined scouring, bleaching and mercerizing. The process is carried out in exhaustion method by using high concentration caustic soda in scouring and bleaching bath. As a part of investigation another two samples are prepared; one is by only scouring and bleaching and another one is by scouring and bleaching and then mercerizing. For proofing the effectiveness of combined scouring, bleaching and mercerizing surface morphology, fine structure (crystallinity, crystallite size, crystalline phase), barium activity number of both two mercerized fabrics, bursting strength of all three fabrics are investigated. The K/S value of all three fabrics dyed with vinylsulfone reactive dyes in different concentration is also measured. By analyzing the surface images of all three fabrics it can be said that the surface of the two mercerized fabrics exhibits smoother, lustrous and round like structure than the scoured and bleached fabric. The decrease in crystallinity, conversion of cellulose I to cellulose II and crystallite size confirm the effectiveness of the process. Barium activity number of two mercerized fabrics is lying between the standard values. The improve in bursting strength and K/S value of dyed fabrics prove that combined scouring, bleaching and mercerizing enhance the physical properties same as the separately mercerized fabric.

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