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Effects of Resin Treatments on the Quality of Cotton Fabric Dyed with Reactive Dye

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Abstract
In this study, we analysed different resin finishing agents on cotton fabric dyed with reactive dye. Different tests were carried out on the resin treated fabric, such as the wrinkle recovery, tensile strength, dimensional stability, stiffness, abrasion resistance and colour strength (K/S) of the dyed fabrics. Melamine formaldehyde resin treated fabric gave a higher crease recovery angle, better smoothness and higher bending length because of higher crosslinking with cellulose. The colour strength decreased after applying the resin finish, and fastness properties were unaffected by the resin treatments.

Key words: *cotton, formaldehyde resin, crease, tensile strength, abrasion, pilling, dimensional stability.*

Introduction

Among all the natural and synthetic fibres, cotton is still the most popular fibre because of its unique properties, such as strength, absorbency and comfortability [1-2]. Cotton fibre consumes 48% of the total market share around the world [3]. However, one of the major drawbacks of cotton fabric is its poor crease recovery property. A number of studies have been carried out on the surface modification of cotton fibre prior to dyeing through employing several finishing agents with a view to improving the dye uptake and fastness properties [4-5]. Crease formation in cotton fibre is a complex mechanism because of intermolecular hydrogen bonding of primary and secondary hydroxyl groups of the polymer chains. These weak hydrogen bonds in the amorphous regions can easily break down by folding. In the folding state, the broken hydrogen bonds get reformed at different new positions, and thus the crease gets stabilised [6]. Fulfilling the satisfactory comfort of cotton fabric, different durable press finishes have been used for many years [7]. Resins comprise two types: one is deposited on the fabric surface as surface coating, where no reaction will take place between the fibre and resin, and the other type of resin chemically reacts with the fibre. Various chemicals have been employed to impart protection against crease recovery to cotton fabric, among which formaldehyde-based cross-linking agents are dominantly used in the coloration industry. Many researchers have reported that the harmful effects of free formaldehyde content resin, which reduces the strength of cotton fabric, increases the stiffness and also reduces the abrasive properties of treated fabrics [8-10]. N-methylol compounds such as dimethylol dihydroxyethylene urea (DMDHEU) react readily with the hydroxyl groups of cellulose chains [11-15].

In this study, we analysed the physical properties of different resin treated and untreated fabrics, such as the formaldehyde content, crease recovery, tensile strength, smoothness of appearance, bending length and shrinkage (%). The subsequent section describes the colour strength, colour difference and colour fastness properties of both fabrics.

Experimental

Materials

Scoured & bleached cotton (100%) woven fabric (117 g/m^2, number of threads: warp 150/inch, weft 80/inch, warp & weft 40^s cotton count) was obtained from Beximco Textile Mills Ltd (Dhaka, Bangladesh). The reactive dye (CI Reactive Red 3, see ***Figure 1***) and six different resin finishes (***Table 1***) were supplied through the generosity of BASF, Huntsman, and the Orient Chemical Company (Dhaka, Bangladesh). Dyes and resin finishes were used without purification. $MgCl_2$ was used as a catalyst.

Dyeing

Scoured & bleached cotton fabrics were dyed in a laboratory-dyeing machine at a liquor ratio of 1:20. A 50 ml dye bath consisting of C.I. Reactive Red 3 and $Na_2S_2O_4$. 10 H_2O (50 g/l) was prepared. The cotton scoured fabric (2.0 g), reactive dyes (1% owf) and salt were immersed in the dye bath, and then the temperature was raised to 80 °C at a rate of 1.5 °C/min. Na_2CO_3 was added to the dye bath after 30 mins. Dyeing was carried out at this temperature for 40 min, after which all of the samples were rinsed and dried at 60 °C.

Padding

Six different types of resin finishing agent (200 g/l) and catalyst (triethylenetetramine) (50 g/l) were added, and pH 4.5 was maintained by acetic acid. Cotton fabric was padded by a Werner Mathis AG (made in Switzerland) padding machine. The fabric speed and pressure between the padding rolls were controlled carefully. The wet pick up percentage was 75%. The padded fabric was dried at 120 °C for 2 min, after which the fabric was cured at 170 °C for 1 min.

Measurement of formaldehyde content

The formaldehyde content on the fabric was measured by BS EN ISO 14184-1:1999 Part I using a dynamic double beam DB-20 UV-Vis spectrophotometer (Dynamica GmbH, Switzeraland). 5 ml of the filtered test specimen was added with 5 ml of formaldehyde and 5 ml of acetyl acetone reagent solution, and then shook. Then the test tubes were placed in a water bath at (40±2) °C for (30±5) min. To measure the absorbance, a 10 mm absorption cell was used.

Measurement of crease recovery

A rectangular piece of fabric was folded and kept under pressure of a certain load for a certain period of time. The ability of the fabric to resist and recover from this deformation after releasing this load to the initial wrinkle-free surface is expressed as the crease recovery property. The capability of the fabric to regain its initial state is measured by the angle between the pre-folded halves, termed as the crease recovery angle. Standard AATCC 66 was used to measure the crease recovery properties. The load used was (500±5) g for 5 min at standard room temperature. The samples for crease recovery measurement were typically cut according to a special standard rectangular shape. The value of the crease recovery angle recorded is the average of ten measurements.

Measurement of tensile strength

The tensile strength was measured by the ISO test method 12676-1989 at room temperature. The samples were cut into strips of 5cm width and 20 cm length, and every data point was the average of five measurements. A universal strength tester Titan[5] (James Heal, UK) was used employing a crosshead speed of 20 m/min.

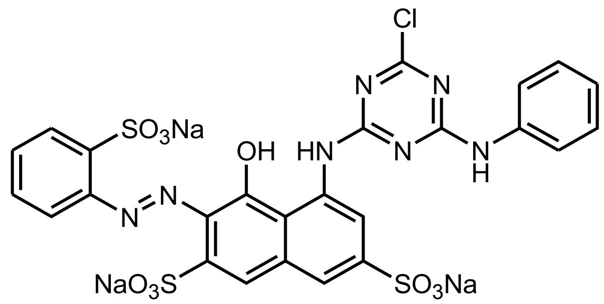
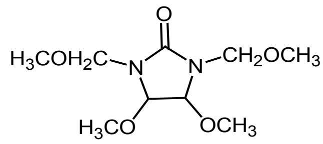
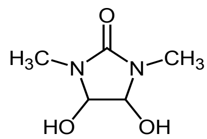
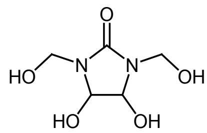
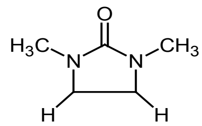
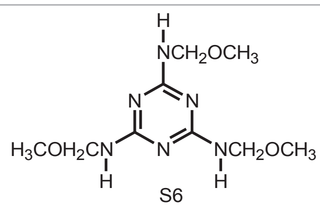


Figure 1. C.I. Reactive Red 3.

Table 1. Different resin finishing agents.

Chemical name	Chemical structure
1,3-Bis-hydroxymethyl-urea	O ‖ HOCH$_2$-N-C-NH-CH$_2$OH S1
4,5-Dimethoxy-1,3-bis-methoxymethyl-imidazolidin-2-one	 S2
4,5-Dihydroxy-1,3-dimethyl-imidazolidin-2-one	 S3
4,5-Dihydroxy-1,3-bis-methoxymethyl-imidazolidin-2-one	 S4
1,3-Dimethyl-imidazolidin-2-one	 S5
N,N,N-Tris-methoxymethyl-[1,3,5]triazine-2,4,6-triamine	 S6

Measurement of stiffness

In principle, cloth stiffness is the resistance of a fabric to bending, which can be determined by measuring the bending length. Bending length is the length of the fabric that bends under its own weight to a definite extent. Standard ASTM (D-1388-19988) was used to measure stiffness by means of a Shirley Stiffness Tester (TESTEX, China). Samples were cut into strips of 25 cm length and 2.5 cm width. When two inclined lines (inclined plane making an angle of 41.5 with the horizontal plane) of the tester coincided, the length of the overhanging portion from the scale was recorded.

Measurement of dimensional stability

Dimensional change is measured in terms of the shrinkage percentage and spirality in the angle, for which the ISO 63301 test method was used. A fabric sample of

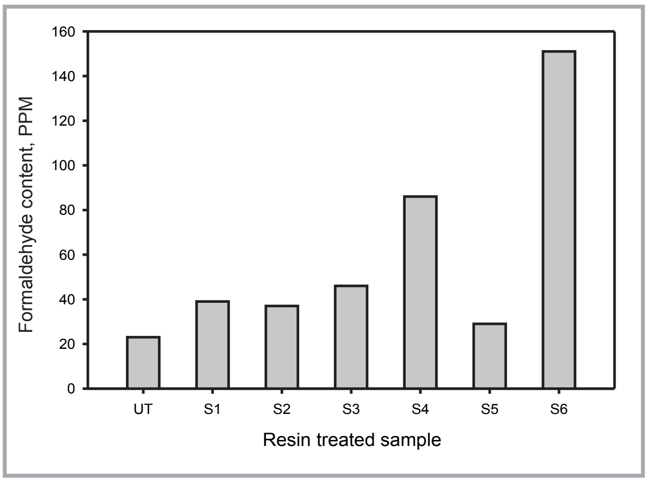


Figure 2. *High molecular weight resin formation with a most complex structure of cross-linking.*

50 cm X 50 cm was taken and an initial length of 35 cm was marked both in the warp and weft direction. The fabric sample was soaked in soap solution of 2 g/l at room temperature for 1 hr. The sample was then rinsed in cold water and dried. The final distance was measured and the change in the dimension was expressed in percent.

$$S = (L_o - L_a) \text{ X } 100/L_o \qquad (1)$$

Where L_o = initial length, L_a = final length, S = shrinkage percentage, %.

Measurement of abrasion

Abrasion is the wearing away of any part of a material by rubbing it against another surface. The specimen was abraded by rubbing it multi-directionally against an abrading cloth. For measurement the ASTM standard testing method D 4060-2006 was used along with a Martindale abrasion tester (SDL Atlas, USA). Samples were cut into specimens of 13.5 cm diameter. The value of abrasion measurements recorded is the average of five measurements.

Measurement of pilling

To assess the pilling of the fabrics, a Martindale Pilling Tester (SDL Atlas, USA) was used and the test method stipulated by ASTM D 4970. Samples were cut to 140 mm diameter. The machine was run for 500 cycles, 1000 cycles and 2000 cycles, and the sample tested was assessed with a photographic replica.

Table 2. *Crease recovery angle of different resin finished fabrics.*

Sample	Recovery angle, °
UT	100
S1	145
S2	158
S3	155
S4	148
S5	145
S6	163

Measurement of smoothness

To measure the fabric smoothness, the specimen was subjected to repeated washing with (3 and 5 times), for which the AATCC test method 143-1992 was used. The sample tested was assessed with six plastic replicas. An expert observer assigned a numerical grade to the specimen corresponding to the replica through observing the specimen's smoothness in appearance.

Measurement of colour intensity

The color strength (K/S) of the dyed fabrics was measured using a spectrophotometer (X-rite 8000 series, UK, standard light D65, 10^o standard observer, specular component included) that was interfaced with a personal computer. The L^*, a^* and b^* system used was based on the CIE-Colour triangle. The colour difference ΔL^*, Δa^* and Δb^* was measured. The color strength of the dyed fabrics before and after finishing was calculated according to the following equation:

$$K/S = (1 - R)^2/2R, \text{ where R is the reflectance}$$

$$\Delta L^* = U_{ntreated} - L_{(S1\sim S6)},$$
$$\Delta a^* = a_{untreated} - a_{(S1\sim S6)},$$
$$\Delta b^* = b_{Untreated} - b_{(S1\sim S6)} \qquad (2)$$

Scanning electron microscope (SEM)

The surface morphology of the untreated and resin treated cotton fabrics was examined by SEM. Micrographs were taken with a JSM-5400 instrument (JEOL, Japan). A sputter coater was used to pre-coat conductive gold onto the surface before observing the microstructure at 30 KV.

Results and discussion

Formaldehyde content results

Results of the formaldehyde content on the treated and untreated fabric are shown in ***Figure 2***. Melamine formaldehyde resins S6 & S4 showed a higher value of formaldehyde content. On the other hand S1, S2, S3 and S5 showed a low amount, below the tolerable range (<30 ppm). Hence it can be explained that the more electron donating group (-OH, $-NHCH_2OCH_3$) enhances the resonance in cyclic structures; thus S4 and S6 showed higher formaldehyde content.

Crease recovery angle results

The crease recovery angle of the different resin finished fabrics are shown in ***Table 2***. Many researchers have reported the crease recovery properties of cotton fabrics [16]. All the fabric samples treated with resin finishes showed a higher recovery angle than the untreated ones. The samples with a high formaldehyde content showed a higher recovery angle. The maximum recovery was exhibited by sample S6. Therefore we can say that melamine formaldehyde resin imparts a higher recovery property than the other resin finishes. It is considered that the higher molecular wt. of resin enhances the formation of most complex crosslinks with cellulose (***Figure 2***), and also acts as cellulosic anti-swelling. The molecular weight of different resin finishes are (S1 = 120.11, S2 = 234.252, S3 = 206.198, S4 = 146.14, S5 = 114.15 & S6 = 216.205). High molecular weight resin of the treated fabric increases the crystalline region and decreases the amorphous region. Thus it shows a greater improvement in the recovery angle.

Smoothness and appearance results

Figure 3 illustrates the smoothness and appearance of resin treated and untreated fabrics. From the figure it can be explained that resin makes crosslinks with adjacent cellulose, thus preventing the movement of fibre molecules during stress, which hinders swelling as well as shrinkage and wrinkle formation. Sample S6 showed a higher smoothness rating than the other samples. The chemical composition of this resin is melamine formaldehyde; this product has mostly three to six reactive n methylol groups, which leads to high cross-linking. Cellulose chains are bonded with primary and secondary hydroxyl groups, thus enhancing the crystalline region. However, some

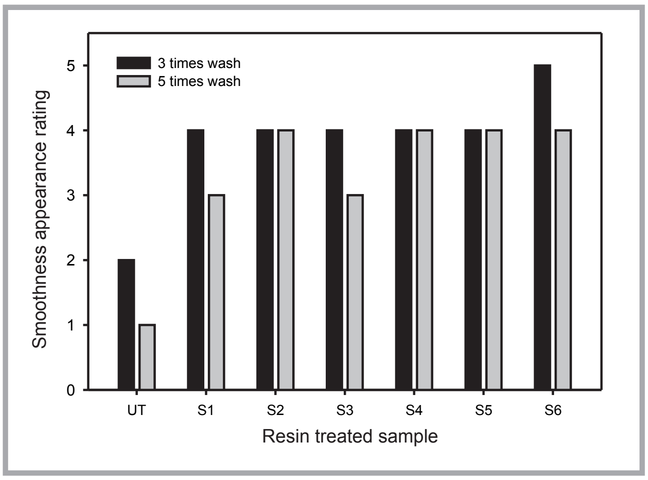


Figure 3. Effect of resin treatment on smoothness appearance of cotton fabric.

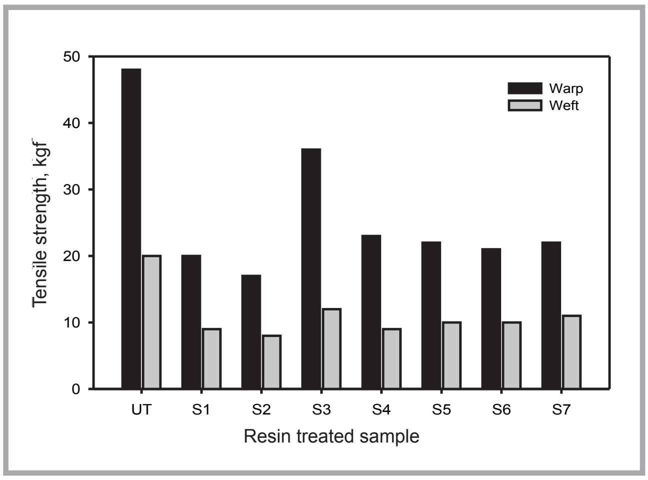


Figure 4. Effect of resin treatment on tensile strength.

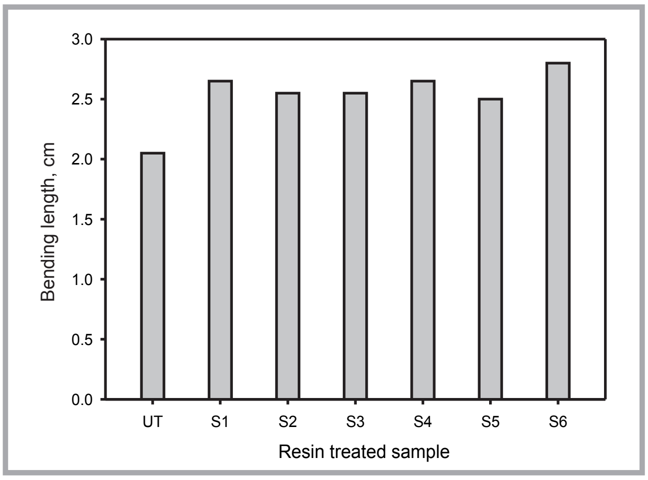


Figure 5. Effect of resin treatment on bending length, cm.

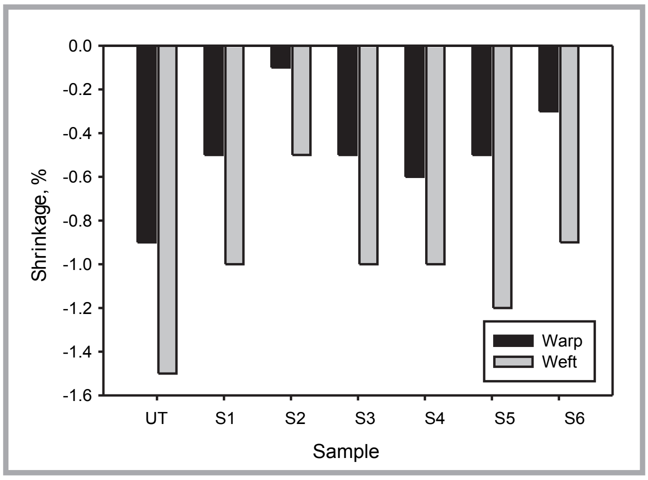


Figure 6. Effect of resin treatment on shrinkage, %.

chains do not get bonded with hydrogen groups, which creates an amorphous region of the cellulose. Melamine formaldehyde resin performs bonding with the "not bonded" portion of cellulose and makes a more crystalline region with the whole chain; thus smoothness increases.

Tensile strength results

The tensile strength of resin treated and untreated samples was measured in the kilogram force unit. ***Figure 4*** illustarates that the tensile strength in both the warp and weft direction was reduced after applying different resin finishes. Previous studies showed that when cotton fabrics are treated with durable press finishes, this reduces the fabric strength [17-19]. The figure shows that after applying the resin finish, the tensile strength of the cotton fabric decreased to almost half of that of the untreated fabric, both in the warp and weft direction. Hence it can be said that resin treatments create cross-links with adjacent cellulose, thus forming a three-dimensional branched network and blocking the free hydroxyl groups, resulting in lower molecular movement and displacement of the hydrogen bonding; thus the strength is reduced.

Bending length results

The bending lengths of all resin finished and untreated samples are compared in ***Figure 5***, which states that the resin finished fabric showed a higher bending length than the untreated fabric. The fabric treated with melamine formaldehyde resin, which has a reactive n methylol group, showed the highest bending length. The results demonstrate that the resin treatment creates a three- dimensional branched network replacing the hydrogen bonds of primary and secondary hydroxyl groups in cellulose. which enhances the crystallinity of the cellulosic polymer. It also imparts very a stiff and firm handle to the fabric as well as increases the bending length.

Shrinkage (%) results

The present study verified the shrinkage (%). ***Figure 6*** shows that untreated fabric gave poor shrinkage properties than the treated fabric, due to the three-dimensional network of resin finished fabric restricting molecular movement because of the blockage of free hydroxyl groups; the filler-matrix hinders the swelling of fabric. S2 showed better shrinkage percentage because the modified DMDHEU and methylol group creates a cross-linked three-dimensional network which cannot easily be broken down by water molecules.

Abrasion resistance results

Figure 7 shows that the untreated fabric exhibited lower weight loss than the

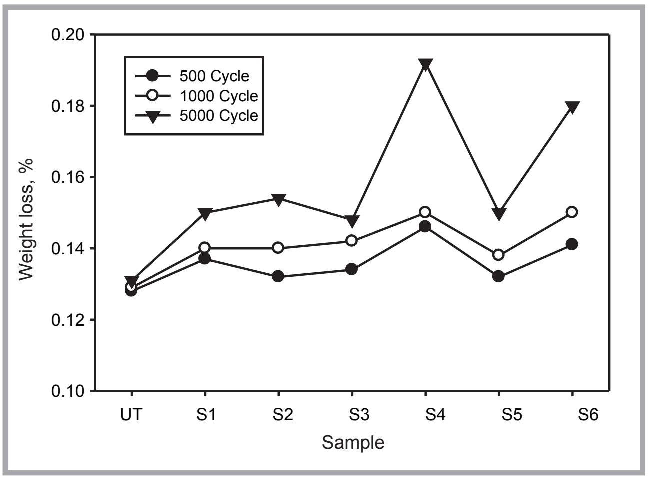


Figure 7. Abrasion resistances with the weight loss (%) of the resin treated fabric.

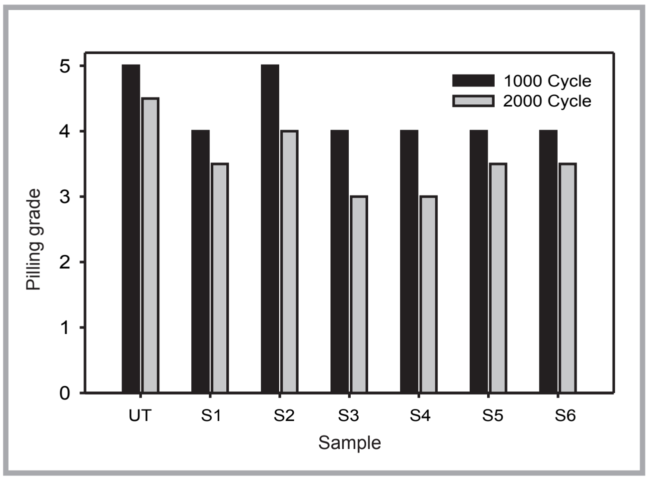


Figure 8. *Effect of resin treatment on pilling performance. (1 = no, 2 = slight, 3 = moderate, 4 = severe, 5 = very severe pilling).*

treated one. It clearly indicates that resin treatment increased the weight loss of the fabric, and hence the abrasion resistance was reduced. The crosslinking of the three-dimensional network of cellulose and resin creates a stiffer fabric; thus molecular movement of the cellulose chain is reduced. S4 and S6 formed a higher crosslinking with cellulose.

Pilling performance results

Pilling has been a problematic phenomenon for a long time in the textile industry. It is the formation of pills or knots on the surface of woven or knitted fabric caused by friction [20-21]. The pilling performance of untreated and treated fabrics is shown in ***Figure 8***. The figure illustrates that resin treated fabric showed a lower pilling rate than the untreated fabric. Hence it can be demonstrated that resin treatment reduces the molecular movement of the cellulose structure; thus when rotational stress is applied, the cellulose chain can be degraded.

Colour strength results

Colour strength is a term used to describe color efficiency on the surface of a fabric. The effect of simultaneous dyeing and resin finishing of cotton fabrics has been studied [2] by several researchers. The K/S value of the resin treated fabric was slightly lower than that of the untreated fabric, as shown in ***Table 3***. Resin finish is applied on fabric at high temperature, and thus colour may bleed out at high temperature. The colour difference showed slight changes after resin finishing.

Table 3. *K/S & Colour difference value of different fabrics.*

Sample	K/S Values (1% owf C.I. Reactive Red 3)	ΔL^*	Δa^*	Δb^*
UT	17.5	–	–	–
S1	16	1.50	-0.23	-0.58
S2	16.5	1.13	-0.14	0.05
S3	16	0.88	0.09	0.48
S4	15.5	0.95	0.03	0.28
S5	16	1.01	-0.16	-0.35
S6	16.2	0.93	-2.51	-2.30

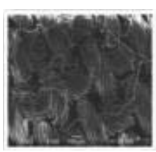
Scanning electron microscope results

The SEM micrograph shown in ***Table 4*** depicts that twisting of the fibre of the untreated fabric was at a greater extent as compared to the resin treated fabric. Hence we can say that resin treatment on cotton fabric forms a cross-linked structure with the free hydroxyl groups of cellulose chains, and consequently it is quite stable with respect to wrinkling or twisting.

Colour fastness results

Appropriate color fastness for many end users can be achieved through correct dye recipe selection and optimised finishing route design. Also color fastness varies with changes in the fibre structure and properties. Color fastness to washing, light, rubbing and perspiration are compared in ***Table 5***. The data presented for washing, light and perspiration fastness shows that there was no significant change in the resin treated sample as compared with the untreated sample. Dye molecules tend to move out from the outer surface of the resin treated fabric because of rubbing, from which it can be explained that some unfixed dyes also interact with resin on the surface of the fabric; thus the dye fastness rating slightly gets decreased.

Table 4. *SEM micrograph of untreated and resin treated fabric.*

UT	S1	S2	S3	S4	S5	S6
						

Conslusions

Resin treatment on cotton fabric enhances the physical properties, such as crease recovery, dimensional stability and abrasion resistance. Among the many resin finishes, melamine formaldehyde resin provides the highest formaldehyde content; moreover it shows a better crease recovery angle, a higher level of smoothness and bending length. Since the higher molecular weight of resin enhances the formation of most complex crosslink structures with cellulose, the crease recovery angle increases. The primary and secondary hydroxyl groups of cellulose enhance the crystalline region, and the "not yet bonded" portion yields an amorphous region. Melamine formaldehyde resin activates the "not yet bonded" portion of cellulose and forms a strong crystalline region; thus smoothness increases. However, resin treatment on cotton fabric reduces the fabric strength because resin treatment creates three-dimensional branched network crosslinks which block the free hydroxyl group, resulting in lower molecular movement and displacement of the hydrogen bonding. On the other hand, resin treated fabric shows better shrinkage (%), abrasion resistance and pilling performance because of the formation of three-dimensional crosslinks with cellulose. No significant changes in color strength can be achieved by different resin treatments. Also colour fastness properties are unaffected by resin treatment, except rubbing fastness; wet rubbing fastness is reduced due to the application of resin treatment.

Table 5. *Colorfastness property of dyed fabric.*

Sample	Wash fastness rating	Light fastness rating	Rubbing fastness		Perspiration fastness	
			Dry	Wet	Acidic	Alkaline
UT	4-5	5	4-5	3-4	4-5	4-5
S1	4-5	5	4	2-3	4-5	4-5
S2	4-5	5	4	2-3	4	4
S3	4-5	5	4-5	2-3	4-5	4-5
S4	5	5	4-5	2-3	4-5	4-5
S5	4-5	4-5	4-5	2-3	4-5	4-5
S6	5	5	4-5	2-3	4-5	4-5

Acknowledgements

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