Optimization of the Hot Alkali Treatment of Polyester/Cotton Fabric with Sodium Hydrosulfite

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Received 6 October 2005; accepted 6 October 2005
DOI 10.1002/app.23851
Published online in Wiley InterScience (www.interscience.wiley.com).

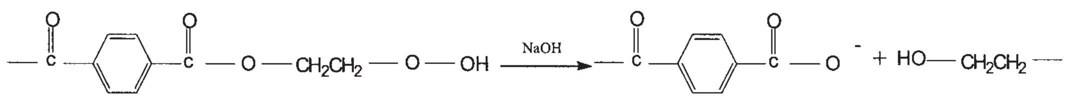
ABSTRACT: The treatment of cotton or polyester fabric in alkali media is a common modification process for producing a fabric with desirable qualities. Alkali treatment of polyester/cotton fabric could produce a fabric with better performance. In this research, polyester/cotton fabric was treated with NaOH at different temperatures and times. The results show that alkali treatment at the optimum temperature and time with NaOH could hydrolyze the polyester fiber surface and remove some of the impurities from the cotton fiber at the same time and may also improve some of the fabric properties, such as fabric regain, water drop absorbency, and fabric pilling. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 100: 5049–5055, 2006

Key words: fibers; modification; polyester; strength; tension

INTRODUCTION

The mercerization process with a concentrated solution of NaOH is well-known for the improvement of some of the physical properties of cellulose-containing fabrics. Recently, researchers have found that mercerization at a low temperature may swell the cotton fiber and produce desired properties, but this process may not be uniform, and in industrial processes, this function may be limited to the fiber surfaces.[1] As a result, the fiber core remains unaffected by the alkali; in contrast with hot alkali, it can rapidly penetrate the cotton fibers and cause more uniformity.[1–6]

The alkali treatment of polyester with NaOH at high temperatures is a conventional process for hydrolyzing the fiber surface, decreasing the fineness of the fiber, and improving the wetting, softness, and fabric comfort. The hydrolysis process of the polyester is due to the hydroxide ions attacking the fabric surface and producing carboxylate ions in the fabric surface.[7–11] This reaction is as follows:



With the increase in the commercial use of different fiber mixtures in the fabric, the effects of alkali on the physical characteristics of these fabrics have been noted. The alkali treatment of cotton/polyester fabric can be done at 15–20°C, which is the same as cotton mercerization, and may improve dyeability of the fabric.[12] Furthermore, as a result of some problems arising from cold mercerization, the hot mercerization process was developed.[13–15] Hot mercerization of the cotton/polyester fabric can impart silky softness to the polyester fibers and improve water and dye absorption on both fibers in the fabric.[14–15]

A review of the research work showed that investigations in this area have been focused on the continuous alkali process without tension. However, the effect of this process on the whiteness of the fabric has not been reported. In this study, we applied the batch method (exhaustion) and measured the changes in the whiteness of the fabrics. The use of sodium hydrosulfite for the reduction of fabric yellowing due to the alkali treatment and the effectiveness of $Na_2S_2O_4$ in the alkali treatment on the other fabric properties are reported.

EXPERIMENTAL

Materials

The cotton/polyester fabric used was a blend 50% cotton and 50% polyester fiber with a warp yarn count

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Journal of Applied Polymer Science, Vol. 100, 5049–5055 (2006)

TABLE I
Sample Coding Based on Process Conditions

Sample	Temperature (°C)	Time (s)	Treated samples with $Na_2S_2O_4$	Temperature (°C)	Time (s)
Untreated	—	—	Untreated	—	—
T 60-t 2	60	2	T 60-t 2H	60	2
T 60-t 5	60	5	T 60-t 5H	60	5
T 60-t 10	60	10	T 60-t 10H	60	10
T 60-t 20	60	20	T 60-t 20H	60	20
T 90-t 2	90	2	T 90-t 2H	90	2
T 90-t 5	90	5	T 90-t 5H	90	5
T 90-t 10	90	10	T 90-t 10H	90	10
T 90-t 20	90	20	T 90-t 20H	90	20

of 30 (Ne)/2 and a density of 59 ends/in.; also, the weft yarn count was 10 (Ne)/1, and the density was 43 ends/in. The woven fabric was desized with a weight of 190 g/m^2. The length of the polyester fiber used in the fabric was 38 mm with a fineness of 1.6 dtex and a semidull luster. The purity of NaOH used (Abadan Petrochemical, Iran) for the alkali treatment was 98%. Also, the wetting agent used in this experiment was Cottoclarin OK from Henkel (Germany), which was a stable solution in alkali media. For the neutralization of the remaining alkali on the fabric, acetic acid (56% pure) was used. The final scouring process was done with a nonionic detergent (Diadavin EWN, Bayer, Germany). Also, a white powder of sodium hydrosulfite (90% pure; Anhai Chlorine-Alkali Chemical Group, China) was used.

Methods

The alkali treatment of fabric was carried out in a stainless steel tank and a stainless steel tension frame. The exhaustion method was applied. The temperatures of the alkali treatment were 60 and 90°C for different times (2, 5, 10, and 20 min), and the concentration of the alkali was 24% on the weight of the bath. The alkali treatment for selected samples was carried out with the addition of different amounts of sodium hydrosulfite ($Na_2S_2O_4$) as an oxygen scavenger. The amount of $Na_2S_2O_4$ was dependent on the time of the process; thus, 2 g/L $Na_2S_2O_4$ was used for a 2–5 min process, 4 g/L $Na_2S_2O_4$ was used for a 10-min process, and 6 g/L $Na_2S_2O_4$ was used for a 20-min process. After alkali treatment, the samples were washed with cold water and then neutralized with 5 mL/L acetic acid. The treated samples were then washed with soap and warm water. For each treated sample, a special code was selected, which was dependant on the process conditions (Table I). There were three replications per sample per test, and all of the treated samples remained in the standard conditions room (65% relative humidity and 20°C) for 24 h before physical testing. The mean values for each sample were also recorded.

The percentage of weight loss was calculated with the following formula:

$$\text{Weight loss (\%)} = [(W_1 - W_2)/W_2] \times 100$$

where W_1 and W_2 are the weights of fabric before and after treatment, respectively.

The percentage of polyester after alkali treatment was determined by the treatment of samples with a 75% solution of sulfuric acid. In these conditions, cotton fibers were dissolved, and the polyester fibers remained unaffected in the fabric samples. By weighing the fabric samples before and after treatment, we determined the remaining percentage of polyester fibers:

$$\text{Remaining polyester in fabric (\%)} = [(W_1 - W_2)/W_1] \times 100$$

where W_1 and W_2 are the weights of the fabric samples before and after acid treatment.

The fabric regain for each sample was calculated as follows:

$$\text{Fabric regain (\%)} = [(W_w - W_d)/W_d] \times 100$$

where W_w and W_d are weights of the fabric before and after drying, respectively.

The drop absorbency of the fabric samples was measured by the dropping of 10 water drops from 1 cm of surface and the measurement of the time of complete absorption of the water drop by the sample.

The fabric strength and elongation were measured by an Instron 4202 instrument (UK) with the constant rate of elongation method. The fabric pilling was also measured by with a Martindale instrument with a rotation of 2000 according to ASTM D 3511. The whiteness index of the fabric samples was determined

TABLE II
Physical Properties of Alkali-Treated and Untreated Cotton/Polyester Fabric Samples

Sample	Weight loss (%)	Remaining polyester (%)	Regain (%)	Water drop absorption (s)	Strength at break (Kgf)	Elongation at break (%)
Untreated	—	50.00 ± 0.05	3.2 ± 0.3	Above 1200	70.5 ± 1.5	41.5 ± 1.2
T 60-t 2	2.33 ± 0.3	49.88 ± 0.04	4.3 ± 0.2	Above 1200	66.5 ± 2.2	35.8 ± 1.4
T 60-t 5	3.53 ± 0.3	49.41 ± 0.07	4.4 ± 0.3	49.0 ± 1.0	64.8 ± 1.6	33.0 ± 1.3
T 60-t 10	7.71 ± 0.4	47.99 ± 0.06	4.6 ± 0.3	18.7 ± 0.5	61.0 ± 2.0	29.7 ± 1.1
T 60-t 20	9.89 ± 0.5	47.28 ± 0.09	4.8 ± 0.2	13.8 ± 0.7	56.6 ± 2.5	27.7 ± 1.2
T 90-t 2	4.50 ± 0.3	49.00 ± 0.07	4.5 ± 0.2	9.8 ± 0.6	63.1 ± 1.7	31.8 ± 1.2
T 90-t 5	10.74 ± 0.5	46.49 ± 0.10	4.8 ± 0.3	24.0 ± 0.6	55.9 ± 2.4	27.3 ± 1.4
T 90-t 10	28.9 ± 0.7	39.38 ± 0.23	5.2 ± 0.2	11.8 ± 0.4	35.1 ± 2.2	24.5 ± 1.3
T 90-t 20	42.83 ± 1.5	26.70 ± 0.18	6.8 ± 0.2	5.6 ± 0.5	26.5 ± 1.6	22.2 ± 1.3
T 60-t 2 H	1.99 ± 0.2	49.98 ± 0.03	4.1 ± 0.2	5.4 ± 0.2	67.6 ± 2.5	38.2 ± 1.4
T 60-t 5 H	2.26 ± 0.3	49.82 ± 0.03	4.3 ± 0.4	32.0 ± 0.4	66.7 ± 2.3	36.0 ± 1.4
T 60-t 10 H	6.19 ± 0.3	48.18 ± 0.05	4.5 ± 0.3	9.6 ± 0.5	62.7 ± 1.8	31.1 ± 1.3
T 60-t 20 H	7.37 ± 0.3	47.64 ± 0.06	4.9 ± 0.4	6.5 ± 0.6	61.7 ± 2.3	30.3 ± 1.1
T 90-t 2 H	4.26 ± 0.3	49.24 ± 0.04	4.2 ± 0.3	6.2 ± 0.7	64.1 ± 2.6	32.4 ± 1.2
T 90-t 5 H	8.54 ± 0.4	47.56 ± 0.07	4.4 ± 0.2	8.8 ± 0.6	58.4 ± 1.9	29.1 ± 1.4
T 90-t 10 H	18.65 ± 0.6	43.57 ± 0.07	4.7 ± 0.3	5.5 ± 0.5	43.9 ± 1.6	25.9 ± 1.1
T 90-t 20 H	37.61 ± 0.8	30.27 ± 0.12	6.4 ± 0.3	1.5 ± 0.4	28.3 ± 2.2	23.1 ± 1.1

with a reflectance spectrophotometer (Texflash, France) with Hunter method under illuminant D_{65}.

The contact angles (θ's) of the fabric samples were measured by experimental equipment consisting of a camera, computer, and monitor. Liquid drops of 20 μm were deposited on each fabric sample with a micrometer pipette at a distance of 0.5 cm, and the image of each drop was captured by the camera and viewed on the monitor. They were photographed in less than 3 s. Five specimens of each fabric sample were tested. The same size droplets were deposited with the same syringe for all samples. The value of θ was calculated by the measurement of the diameter (d) and height (h) of the drop with the following equation:

$$\theta = 2\ \arctan\left(\frac{2h}{d}\right)$$

RESULTS

Weight loss

The results of the fabric weight loss are shown in Table II. An increase in the time of treatment from 2 to 20 min and in the temperature of treatment from 60 to 90°C caused an increase in the percentage of fabric weight loss. This could have mainly been due to the polyester hydrolysis, but some changes may have occurred in the cotton fibers that could have led to a decrease in the weight of the fabric samples. These changes could have caused a decrease in the impurities of the cotton fibers and/or the decomposition of some cotton fibers as a result of the formation of oxycellulose. Therefore, a decrease in the mass of the cotton fibers in the construction of the fabric may have occurred.

The results in Table II show a decrease in the percentage of remaining polyester in the mixture fibers fabric construction when the alkali treatment was done at 90°C. This could have been the result of more polyester fiber hydrolysis. This means that the polyester fibers were more sensitive to the temperature than the cotton fibers were during alkali treatment. Also, the addition of $Na_2S_2O_4$ sodium hydrosulfite to the alkali treatment at 60 and 90°C led to a lower decrease in the fabric weight loss. This could have been due to the consumption of part of the alkali in the alkali treatment by $Na_2S_2O_4$, which could be explained by the following mechanism:[15]

$$S_2O_4^{2-} + 4NaOH + O{=}C\!< \longrightarrow 2SO_3^{2-} + 2H_2O + {}^{-}O\text{-}C\!\lessgtr$$

Therefore, we suggest that the presence of $Na_2S_2O_4$ in the alkali media in this treatment acted as a preservative for the cotton fibers from the attack of oxygen in the surrounding area. This led to the production of a fabric free from yellowing with a higher strength:

$$Na_2S_2O_4 + O_2 \rightarrow Na_2SO_4 + SO_2$$

Fabric regain

The results of fabric regain are presented in Table II. The fabric regain increased with alkali treatment. However, this was higher for the fabric treated for a longer time and at a higher temperature. This could have resulted from the increase in the polyester hydrolysis, and the percentage of polyester fibers in the mixture fabric construction decreased at 90°C. The alkali treatment of the fabric at 60°C and for short

TABLE III
Fabric Pilling and Whiteness Index of Alkali-Treated and Untreated Fabric Samples

Sample	Pilling	Whiteness index	Contact angle (°)	Fabric code	Pilling	Whiteness index	Contact angle (°)
Untreated	2	60.4 ± 0.4	89.4 ± 2.1	Untreated	2	60.4 ± 0.4	89.4 ± 2.1
T 60-t 2	2	60.7 ± 0.3	86.2 ± 1.7	T 60-t 2-H	2–3	61.2 ± 0.4	85.3 ± 2.3
T 60-t 5	2–3	61.5 ± 0.5	84.3 ± 2.5	T 60-t 5-H	3–4	64.1 ± 0.6	77.6 ± 2.5
T 60-t 10	2–3	60.1 ± 0.5	82.6 ± 2.5	T 60-t 10-H	3	62.8 ± 0.3	75.1 ± 1.9
T 60-t 20	1–2	56.2 ± 0.7	77.7 ± 1.6	T 60-t 20-H	2–3	62.3 ± 0.5	74.3 ± 2.6
T 90-t 2	2–3	61.1 ± 0.4	85.2 ± 3.3	T 90-t 2-H	2–3	64.9 ± 0.2	76.8 ± 1.1
T 90-t 5	2	59.7 ± 0.9	79.3 ± 2.6	T 90-t 5-H	2	62.9 ± 0.4	62.9 ± 2.7
T 90-t 10	1–2	47.1 ± 0.8	61.2 ± 1.8	T 90-t 10-H	1–2	61.5 ± 0.3	<10
T 90-t 20	1	37.9 ± 1.3	52.5 ± 3.7	T 90-t 20-H	1	60.8 ± 0.3	<10

times (2 and 5 min) caused swelling of the cotton fibers, which was mainly responsible for the increase in the fabric regain.

Also, the presence of $Na_2S_2O_4$ in the alkali treatment led to a decrease in polyester hydrolysis, and therefore, the fabric regain obtained for these samples increased less than that of the alkali-treated samples in the same treatment conditions.

Fabric surface water drop absorption

The results of the water drop absorption on the fabric samples are presented in Table II. The results show that alkali treatment led to a significant decrease in the time of water drop absorption on the fabric surface. The results also indicate that alkali treatment on the polyester/cotton fabric in any conditions caused a large decrease in the water absorption time on the fabric surface. As a result of alkali treatment of the fabric, the polyester fibers were hydrolyzed, and some hydrophilic groups, including —OH and —COOH groups, on the fabric surface were produced. This led to a decrease in the time of water drop absorption. An increase in the time and temperature of the alkali treatment led to a higher hydrolysis of polyester fibers and an easier diffusion of alkali into the cotton fibers, which may have increased the fiber swelling, and thus, the time of drop absorption on the treated fabrics decreased. The addition of $Na_2S_2O_4$ in the alkali treatment produced a decrease in the drop absorption time, which could have been due to the lower hydrolysis of the polyester fibers and the conservation of cellulose fibers from the conversion to oxycellulose and probably a decrease in the separation of tiny fibers from the fabric surface.

Fabric strength and elongation at break

The results of fabric strength and elongation are presented in Table II. An increase in the time and temperature of the alkali treatment led to a decrease in the fabric strength and elongation. This could have been due to the polyester hydrolysis and cotton weakness as a result of the production of oxycellulose and the reduction of crystalline, which also may have been prolonged with the extension of the treatment time and the increase in the temperature of the process. The addition of $Na_2S_2O_4$ in the treatment bath led to a reduction in the polyester hydrolysis and prevented the cellulose from converting to oxycellulose.

Fabric pilling

The results of fabric pilling are presented in Table III. The results show that the fabric pilling was dependant on the treatment conditions. At 60°C for 2, 5, and 10 min processing times, the fabric pilling improved, but with the extension of the processing time to 20 min, the fabric pilling increased. The alkali treatment at 90°C was especially dependant on the treatment time. When the treatment time was 2 min, the fabric pilling marginally improved, but with extension of the processing time, the fabric pilling increased. Suggestions that could be considered for the improvement of the fabric pilling after the alkali treatment are (1) the reduction of the anchor fiber in the fabric surface, (2) an increase in the cotton fiber strength, and (3) an increase in the polyester fiber strength. We also suggest that an increase in the processing time at high temperatures could give rise to fabric pilling due to the addition of the anchor fiber produced in the fabric surface and the easier separation of the fibers from the fabric surface. The results in Table III also indicate that addition of $Na_2S_2O_4$ in the alkali bath could have helped to improve the fabric pilling in some conditions. This may have been due to the consumption of oxygen during the process by $Na_2S_2O_4$ that may have led to the production of free yellowing cotton fiber and also to the production of fewer weaker polyester fibers.

Fabric whiteness

The results of fabric whiteness are illustrated in Table III. The fabric whiteness increased after the alkali

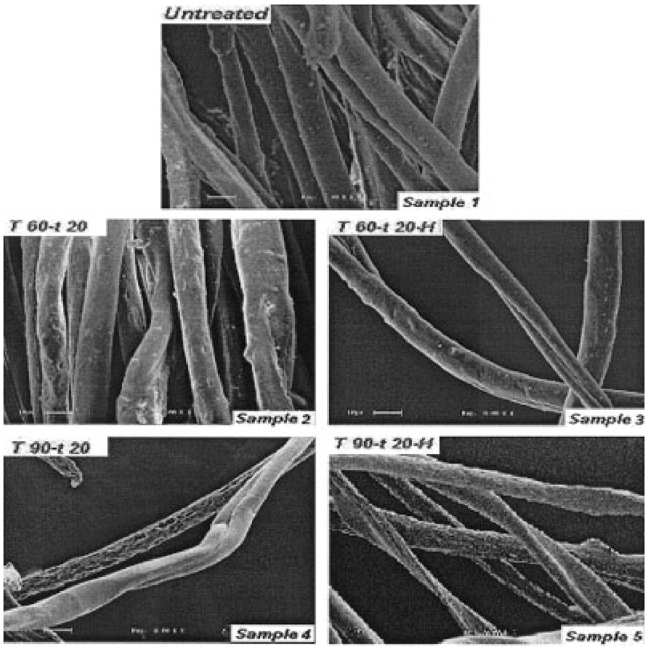

Figure 1 Scanning electron micrographs of treated and untreated samples at 8000×.

treatment, which may have been due to the removal of impurities from the cotton fibers. An increase in the processing temperature along with the extension of the processing time led to a smaller increase in the fabric whiteness index. Also, longer processing times (10 and 20 min) at high temperatures caused fabric yellowing. This happened when the alkali process was carried out without $Na_2S_2O_4$. When the alkali process was associated with $Na_2S_2O_4$, the results obtained were appreciable. This means that at high temperatures and long treatment times, fabrics with higher whiteness indices were obtained.

θ

The results of water θ on the fabric samples are displayed in Table III. According to these results, alkali treatment on the polyester/cotton fabric reduced the water θ. Severe alkali conditions produced a smaller water θ. This was due to the production of a more hydrophilic fabric surface. $Na_2S_2O_4$ in the alkali process of the polyester/cotton fabric had a positive effect as the water θ's decreased.

Microscopic surface studies of the fiber, yarn, and fabric

Fibers

For this purpose, we selected samples with higher alterations in the weight loss to study surface changes. Pictures of the sample were taken with a scanning electron microscope at 8000× and are illustrated in Figure 1. A few pores were produced on the polyester fiber surface on samples 2 and 4 that were alkali-treated at 60°C, and also, the cotton fiber cross-section changed from a dumbbell shape to a double-joint, circle-like shape. However, the size and amount of the pores produced on the polyester fiber surfaces were bigger and larger when the samples were treated at 90°C. Furthermore, in this condition, the cotton fibers swelled extensively, and some fibers collapsed. A comparison of pictures of samples 3 and 5 with samples 2 and 4 indicated that presence of $Na_2S_2O_4$ in the alkali treatment led to lower damage on the surfaces of both the polyester and cotton fibers. This meant that the formation of pores on the polyester fibers and the swelling of cotton fibers decreased when $Na_2S_2O_4$ was added to the treatment solution.

Yarns

The results of yarn density in the fabric, yarn count, and crimp are shown in Table IV (also see Fig. 2). These results indicate that the fabric density was constant for all of the fabric samples. This was due to the process conditions as the alkali treatment was carried out under tension. The fill and warp yarn count increased, and the height of the crimp decreased with alkali treatment. An increase in the process temperature led to an increase in the yarn count and a decrease in the height of the crimp. This was due to an increase in polyester hydrolysis and a reduction in impurities

TABLE IV
Yarn Count, Yarn Density, and Yarn Crimp Height of Treated and Untreated Fabric Samples

		Yarn count (Ne)	Yarn density in the fabric (yarn/in)	Yarn crimp height (mm)
Sample	Fabric code	Warp/Fill	Warp/Fill	Warp
1	Untreated	28.5/9.5	59/44	0.0930
2	T 60-t 20	31.2/10.9	59/44	0.0725
3	T 60-t 20-H	31.0/10.3	59/44	0.0735
4	T 90-t 20	40.0/20.4	59/44	0.0563
5	T 90-t 20-H	36.5/17.8	59/44	0.0625

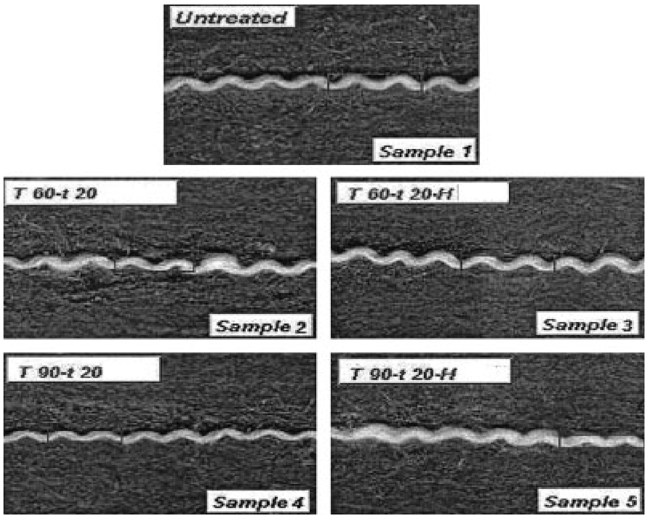

Figure 2 Pictures of yarn crimp from treated and untreated fabric samples.

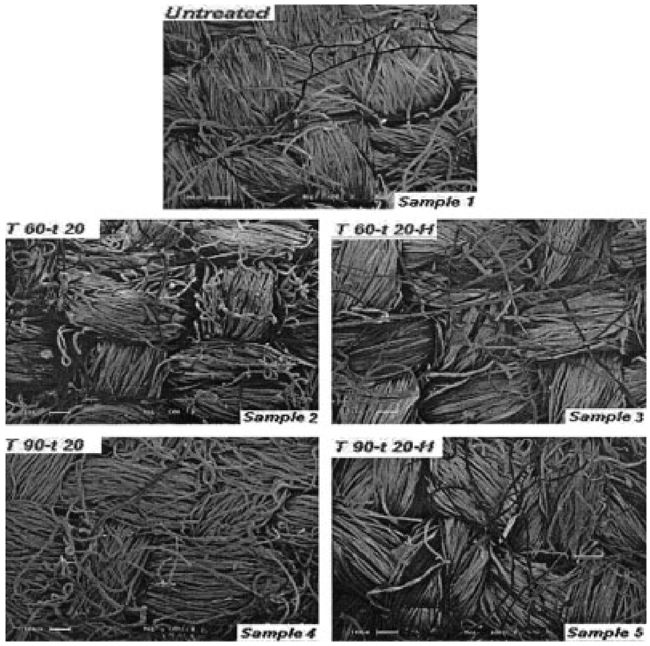

Figure 4 Scanning electron micrographs of treated and untreated fabrics at 6000×.

from the cotton fibers and/or the removal some of the immature cotton fibers from the yarn. We also concluded that pouring $Na_2S_2O_4$ in the alkali bath treatment prevented oxygen from attacking the cotton fibers and, therefore, led to a lower decrease in the yarn fineness. Therefore, we propose that the presence of $Na_2S_2O_4$ in the treatment bath resulted in a lower decrease in the height of the crimp at 60 and 90°C.

Microscopic pictures of the yarns from the untreated and treated fabric samples at 50× are presented in Figure 3. The pictures of samples 2 and 4 indicate that the yarn thickness decreased and that a few fibers as anchor fibers existed on the yarn surface. When the treatment was carried out with $Na_2S_2O_4$ (pictures of sample 3 and 5), the decrease in the yarn thickness and also the amount of anchor fibers seemed to lessen.

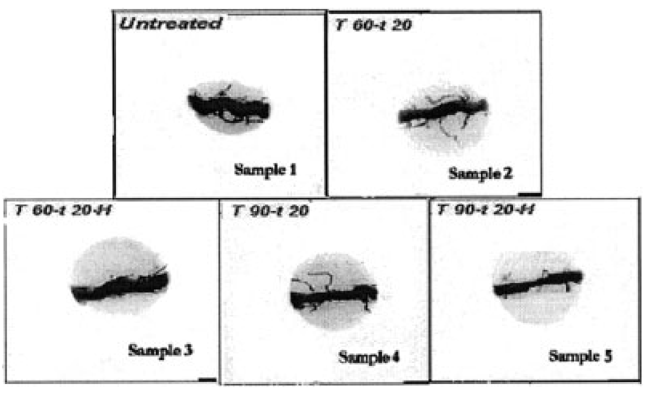

Figure 3 Pictures of yarns from treated and untreated fabric samples at 50×.

Fabric surface

Figure 4 reveals the differences in the alkali treatment conditions. This shows that the fabric surfaces changed with more severe conditions. An increase in the temperature from 60 to 90°C led to a thinner fabric. This was due to an increase in the polyester fiber hydrolysis and damage to the cotton fibers (samples 2 and 4). The addition of $Na_2S_2O_4$ to the treatment bath acted as a preservative for both cotton and polyester fibers and led to a decrease in damage to the cotton fibers and a reduction of polyester hydrolysis (samples 3 and 5).

CONCLUSIONS

This study aimed to optimize the alkali treatment conditions for cotton/polyester fabric to achieve a fabric with higher regain, lower weight loss, higher whiteness, lower loss of strength and elongation, and better fabric pilling performance.

The results of this study show that treatment of the cotton/polyester fabric at 60°C for 20 min with 24% NaOH solution decreased the weight of the fabric by about 10%. This was due to polyester hydrolysis and the removal of cotton impurities during alkali treatment. The fabric regain also increased by about 49%, which was due to an increase in the hydrophilic groups introduced to the fabric surface by the alkali treatment. The water droplet absorption time decreased from 20 min to about 10 s as a result of the purification of cotton fibers and the addition of waterloving groups to the polyester fibers. Also, with this treatment, the fabric had some defects, which included losses of fabric strength and elongation of about 14 and 33%, respectively; a decrease in fabric pilling by a half degree, and a decrease in whiteness index from 29.7 to 24.8.

To minimize the damage to the fabric, a reducing agent, $Na_2S_2O_4$, was added to the alkali treatment bath. On the basis of the treatment time, different amounts of $Na_2S_2O_4$ were added to the alkali solution. The results show that the alkali treatment at 60°C for 20 min with a 24% NaOH solution in the presence of 6 g/L $Na_2S_2O_4$ produced a fabric with optimum properties. As the fabric weight loss decreased to 7%, fabric regain rose to 51%, the loss of fabric strength decreased to 12%, elongation decreased to 27%, the fabric pilling half degree improved, and the fabric whiteness index increased from 29.7 to 34.6. This means that the presence of $Na_2S_2O_4$ had a positive effects on the alkali process of the cotton/polyester and reduced some defects of the alkali treatment, preserved the cotton fibers from oxidation, and prevented severe hydrolysis of the polyester.

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