
**DYEING OF ALKALI TREATED POLYESTER AND POLYESTER/
VISCOSE BLEND FABRICS AND CHARACTERIZATION OF THEIR
PROPERTIES**

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Abstract

The alkaline weight reduction of polyester and polyester/viscose blend fabrics and determination of the fabrics dyeing characteristics were evaluated. An alkali treatment of polyester and blend fabrics was carried out in a pilot scale mini jet machine using dilute caustic soda (7 g/L) at 1:20 liquor ratio, in the presence of cetyl trimethyl ammonium bromide, a cationic surfactant (1 g/L), as a reaction accelerator, at 120°C. The weight reduction obtained was 16 % for polyester fabric and 7.5 for the blend (7.5% weight loss at 95 °C). The alkali treated polyester fabric surface was examined using a scanning electron microscope.

The polyester fabric which has been subjected to alkaline hydrolysis was dyed in alkaline (pH 8.5) and acidic medium (pH 4.5), liquor ratio 1:20, using different Dianix, commercial disperse dyes, in a mini jet machine at and 120°C. The results indicated that the alkaline dyed samples exhibited an increase of color strength and slightly better anti-crease property compared to the acid dyed and untreated control fabrics.

Dyeing of polyester/viscose blend fabrics was carried out in a single bath at 120°C, in a mini jet dyeing machine using the conventional Forosol dyeing at pH 4.5. Alkaline weight reduction process improved the fabrics handle properties. Excellent to very good fastness results were observed for the dyed fabrics.

Key words: alkaline reduction; alkaline and acidic disperse dyeing; polyester; polyester/viscose blend; single bath blend dyeing.

Introduction

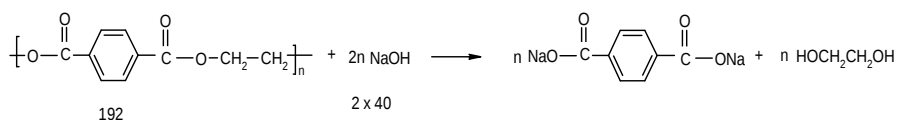
Nowadays, the market demands for silky-like polyester and polyester blend fabrics (viscose or cotton blends) is increased due to the rise in price of natural fibers such as cotton and silk. The textile industry was directed to the production and development of apparel and furniture garments made of polyester and cellulose blends.

Research studies were undertaken in the world for the alkali finishing and dyeing of polyester and blend fabrics to acquire a soft, light, silky handle, to prevent pilling

and decrease antistatic effects of the fabric surface. Sheth *et al.*¹ have investigated the characteristic changes of polyester and polyester/cotton blend fabrics during weight reduction using lab., batch, continuous and jet dyeing methods, for the alkaline treatment in presence of a cationic surfactant. The different factors studied include weight loss, alkali concentration, hydrophilicity, pill formation, denier and tear reduction. Wicker *et al.*² reported on the concepts for after treatment and dyeing of polyester/ cellulose yarns. Montazer *e al.*³ has treated cotton/polyester fabric (50/50 %) with 24 % NaOH at 60 and 90°C for different periods. This treatment improves the fabric properties such as moisture regain, water droplet absorbed and fabric pilling. Huseyin *et al.*⁴ worked on the dye selection for the alkaline, one step disperse/reactive dyeing of polyester/cotton blends. The effectiveness and environmental impact for the alkaline polyester dyeing and reductive clearing have been investigated⁵⁻⁶.

Gawish *et al.*⁷⁻¹⁴ have studied the alkaline finishing of polyester fabrics (also called alkaline saponification, hydrolysis or deweighing) in absence and presence of different specially synthesized cationic surfactants and polymers. The cationic surfactant was found to accelerate the alkaline reaction with nearly complete alkali consumption. The weight loss of fabrics, efficiency of the hydrolysis reaction, fibers tenacity as a function of temperature and reaction time in the presence of different cationic surfactants were evaluated.

A weight loss of 24% was attained¹⁵⁻¹⁹ for polyester fabric at a lab. scale, hydrolysed at 130°C in 40-60 minutes using 10% NaOH (owf), 1% cationic surfactant, which was prepared and compared to cetyl trimethyl ammonium bromide as a control cationic surfactant. Every terephthalate unit requires two moles of sodium hydroxide for the complete saponification reaction and the weight loss is equivalent to the quantity of alkali consumed in the reaction as follows:



On the other hand, the effect of the alkaline weight reduction process on the dyeing of polyester has been studied. Imaruku²⁰, Ibrahim *et al.*²¹ and Youssef *et al.*²² have investigated at a lab. scale the alkaline dyeing system of polyester and polyester/cotton blend fabrics using sodium edetate. Boyadzhev²³ reported that the

treated polyester with different degrees of weight reduction showed a slight decrease in color strength of the dyed and printed fabrics. Needles et al²⁴ investigated that the weight reduced polyester fabrics gave a darker color and a slight color change compared to the untreated fabrics. Becheva²⁵ demonstrated that by increasing the level of weight loss, the color strength was increased but the crock fastness of prints was reduced by 0.5-1 grey scale unit.

In the present investigation we have proceeded for the alkaline hydrolysis, in a HT mini jet machine at a pilot scale Fig.1, of polyester fabric and blends, using dilute sodium hydroxide (7 gm/l) in presence of 1g/l cetyl trimethyl ammonium bromide at 120 °C and 1:20 liquor ratio. A new alkaline dyeing process was adopted using a series of Dianix disperse dyes (DyStar Co.) at pH 8.5 (with a mixture of borax /potassium dihydrogen phosphate), in the presence of Diaserver AD-85 as a stabilizing and buffering agent. Acidic dyeing of polyester at pH 4.5 was done and compared to the alkaline dyeing.

Alkaline hydrolysis at 95 °C and dyeing of polyester/viscose blend fabric (65/35%) was also investigated in a single step at 120°C and pH 4.5 using Forosol dyes (Clariant Co.). The weight loss, fastness properties, dry crease recovery angle, surface roughness, pilings, electrostatic charges and mechanical properties were evaluated for polyester and blend fabrics.

Experimental

Materials

Scoured woven crepe polyester fabric 100% (167 g/m²) have been supplied by Egyptian spinning and weaving Co., Mehala El-Kobra, and polyester/viscose (65/35%) twill blend fabric (270 g/m²), was supplied by Stia Co., Alexandria, Egypt, were used.

Chemicals such as sodium hydroxide, borax, potassium dihydrogen phosphate, citric acid, glauber salt were all laboratory reagent grade.

Cationic surfactant, cetyl trimethyl ammonium bromide was supplied by Amresco Co., USA. Nonionic surfactant, nonyl phenol ethoxylate used for washing was supplied by ICI Co., England.

Dyes for polyester fabric (DyStar Co. Egypt)

Commercial alkali stable disperse dyes used are Dianix Yellow S-G, Dianix Rubine S-3B (CI Disperse Red 35), Dianix Blue S-BG and Dianix Orange S-G 200%.

Dyes for polyester /viscose blend fabric (Clariant Co., Egypt)

Forosol Yellow PC, Forosol Rubinol PC, Forosol Grey PC, Forosol Navy HE and Forosol Brown PC.

Dyeing auxiliaries:

- Diaserver AD 85 , stabilizing and buffering agent, for the soft alkaline polyester dyeing at pH 8.5.(DyStar Co.)
- Sera Gel P-LP, anionic dispersing agent (DyStar Co.)
- Alba fluid, anti-crease (Ciba Co.)
- Respumit NF, antifoaming agent (Bayer Co.)
- Sand acid DSB, anionic, pH adjustment for the acid dyeing (Clariant Co.)
- Indosol ESO, cationic fixing agent for Forosol dyes (Clariant Co.)

Methods

Pretreatment

The fabrics were first washed with 2gm/L nonionic surfactant at 60°C and liquor ratio 1:20 for 30 min , rinsed , dried and pre-set at 190°C for 30 seconds on a stenter.

Alkaline weight reduction process

Weight reduction of polyester and polyester/viscose was carried out in a mini HT jet Longclose dyeing machine (Fig.1) at 120°C and liquor ratio 1:20 (100 liters/5Kg fabric), using 7 g/l sodium hydroxide and 1 g/l cetyl trimethyl ammonium bromide, a cationic surfactant for 60 min. The treated samples were thoroughly rinsed with water, neutralized with 5% citric acid solution and dried. The fabric properties were tested prior to dyeing.

Polyester dyeing

Dyeing was carried out in a Longclose mini HT jet dyeing machine using various dye recipes at 120°C and liquor ratio 1:20. The soft alkaline dyeing of polyester fabric was carried out at pH 8.5 using the following recipe: X% (owf) Dianix disperse dye, 1.5% Sera Gal PLP, 2% of Diaserver AD-85, 2g /L Alba fluid anti-crease, 0.2g/L Respumit NF antifoaming agent, 0.5 g/L odour free carrier Sera Gal PEW, a mixture of 2gm/L borax and 1.5 gm/l potassium dihydrogen phosphate. The polyester fabric was introduced into the dyebath at 60°C and the temperature was

raised at a rate of 2°C/min to 120°C, and the dyeing continued for 30 min, after which, the dye bath temperature was reduced to 70°C, and then the samples were neutralized with 5% citric acid rinsed thoroughly with water and dried (Fig.2).



Fig. 1 Mini jet Longclose dyeing machine for alkaline reduction and dyeing of polyester and polyester/viscose blend fabrics

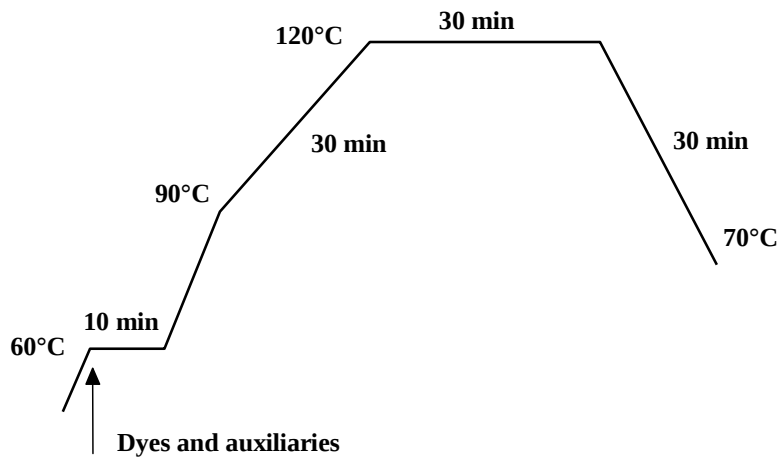


Fig. 2 Dyeing profile for alkaline reduction and dyeing of polyester and polyester/viscose blend fabrics

Following the dyeing profile shown in Fig. 1, the conventional dyeing method of polyester fabric at pH4.5 with 2gm/L sand acid PSB was similarly performed using x% owf Dianix disperse dyes, 1.5% Sera Gal PLP, 2gm/L Alba Fluid anti-crease , 0.2 gm/L Respumit NF antifoaming agent and 0.5gm/L Sera Gel PEW carrier.

The alkaline and acidic dyeing processes were done for the alkaline hydrolyzed fabric (16 % weight loss) and the properties compared to the untreated control polyester fabrics with the following four Dianix recipes (Khaki, Grey, Rubine and Blue colors):

X% (owf) Kaki, combined Dianix dyes recipe:

0.39% Dianix Yellow S-G, 0.04% Dianix Rubine S-3B and 0.02% Dianix Blue S-BG

X% (owf) Grey, combined Dianix dyes recipe:

0.026 % Dianix Orange S-G 200%, 0.03 % Dianix Rubine S-3B and 0.04 % Dianix BlueS-BG

1.37 % (owf) Dianix Rubine S-3B (Rubine), and 0.9 % (owf) Dianix Blue S-BG .

Dyeing of polyester/viscose blend

The blend fabric (alkali reduced to 7.5 % weight reduction) was dyed at pH 4.5 in a single bath, adjusted with 2 g/l Sand acid (Clariant Co.), at 120°C for 30 min as shown in Fig. 2. Dyeing was conducted with x % owf Forosol dyes, 4gm/L Glauber's salt, 2 g/L Alba Fluid anti-creasing agent, 1.5g/L Sera Gal P-LP, 0.2gm/L Respumit NF , 0.5 gm/L Sera Gel PEW. The dyed samples were rinsed, treated with 1% Indosol fixing agent at 70°C for 30 min, after which the fabric is rinsed with water and dried at 120°C.

Four combined Forosol dyes were used as follows:

x% (owf) beige, Forosol dye recipe:

0.034% Forosol Yellow PC, 0.022% Forosol Rubinol PC, 0.04% Forosol Grey PC

x% (owf) grey, Forosol dye recipe:

0.004% Forosol Rubinol, 0.006% Forosol Yellow PC, 0.06% Forosol Gray PC

x% (owf) blue marine, Forosol dye recipe:

0.9% Forosol rubinol PC, 2.9% Forosol Navy HE

x% owf Kaki, Forosol dye recipe:

0.06% Forosol brown PC, 0.06% Forosol Grey PC

Testing

Color measurements

The color strength (K/S) of the dyed samples were measured spectrophotometry. (Data color International SF 600 plus, D65) at the maximum absorption wave length for each dye (λ_{max}).

CIELAB color system

The CIELAB system uses three coordinates L^* , a^* and b^* where L^* stands for lightness and ranges from 0 for black to 100 for white. A color (hue) can also be specified either in terms of the coordinates a^* and b^* (for grey and dull colors),

Fastness properties

The dyed samples were tested according to ISO standard methods.²⁶ The specific tests were ISO 105-C02 (1989) for the washing fastness, ISO 105-E04 (1989) for color fastness to perspiration and ISO 105-B01 (1994) fastness to light using Sun test GPS, Atlas apparatus (Germany).

Crease recovery angle

It was done in the warp and weft directions using the iron recovery apparatus type FF-07 (Metrimpex).

Surface roughness

The surface roughness of the fabric was done using the surface roughness measuring instrument SE1700.

Mechanical Properties

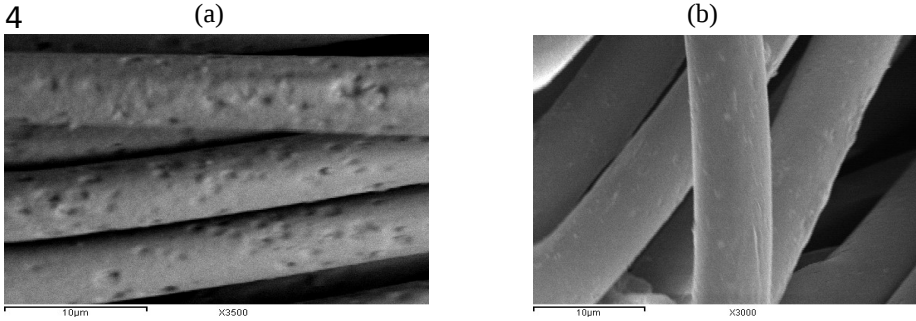
Tensile strength (kg) and percentage elongation were tested on the Instron instrument according to ASTM D 3822.

Scanning Electron Microscopy (SEM)

SEM was done for the untreated and alkaline treated polyester fabric (16%weight loss) Fig.3. The samples were mounted on an aluminum device and sputter-coated with gold for microscopic examination at 3000-3500 X magnification

Determination of pilings

The fabric piling was determined on a ICI piling tester. The fabric sample was sewed on a rubber rod and allowed to rotate in the piling box for ten hours and the average view number of pilings was determined by a magnification lenses and compared to a standard image.



SEM

Fig.3 (a) alkali reduced PET fabric (16% weight loss), and (b) control fabric.

Determination of static charges

The fabric static charges were determined using a hand collect electricity potentiometer type having a narrow scale which does not exceed 10 K V.

Results and Discussion

Effect of the alkaline reduction on the fabric properties

The weight reduction, tensile strength loss, crease recovery angle and surface roughness of the polyester and polyester/viscose blend fabrics treated with 7 g/l sodium hydroxide concentration in the presence of 1 gm/L cationic surfactant, cetyl trimethyl ammonium bromide, at 120°C are listed in Table 1. The surface roughness of the fabrics has decreased as expected with the alkaline treatment and the percentage weight loss increased. The alkali treated fabrics show a loss in tensile strength for polyester fabric which is accompanied by a decrease in the fiber diameter as shown in Figure 3. A more pronounced decrease in tensile strength loss was observed for polyester/viscose blend at 95° and 120 °C compared to that of polyester fabric at 120 °C. This is mostly due to the increase in the weight loss of the viscose component in the blend. Also, the hydrophilicity of the blend fabric would be improved by the alkaline treatment as a result of increase of the viscose swelling and thus speeding up the surface hydrolysis of polyester component, resulting in an increase in the fiber softness.

The results of Table 1 also indicate that the percentage tensile strength loss is twice the fabric weight loss for polyester fabric. In case of polyester/viscose blend fabric, the tensile strength loss is fourth the percentage fabric weight reduction.

Table 1

Effect of alkali treatment of polyester and polyester/viscose blend on weight reduction, tensile strength loss, crease recovery angle and surface roughness.

Fabric	Weight reduction %	Tensile strength loss %	Crease recovery angle*	Surface roughness* um
PET	16.4	30.6	301 (295)	17.9 (20.6)
PET/viscose blend	7.5	32.0	287(276)	18.68(22.3)

(*) = untreated fabrics

Dyeing and fastness properties of the alkali treated polyester fabric

The color strength K/S of the alkaline treated and untreated polyester dyed fabrics with Dianix dyes are shown in Table 2. The results revealed that the alkaline dyeing of the treated fabric give a higher color strength than the untreated samples. However, the untreated and conventionally dyed samples no 6 and 10 show better color strength values than those of the treated fabrics 8 and 12, respectively.

As a result of greater surface reflectance and better dye penetration, alkaline treated fabrics tend to exhibit higher color strength than untreated fabric. Also, the interior of the fiber becomes more accessible and dyes are able to diffuse more easily following the weight reduction treatment. An exception to this occurs under conventional dyeing of untreated fabric and the bigger diameter of the untreated fibres may suppress the dye to diffuse completely into the fiber.²⁷

The colorimetric (L^*) refers to brightness–darkness with values from 100 to 0 representing white to black. The (a^*) values represent negative (green) to positive (red). The (b^*) values are from negative (blue) to positive (yellow). The colorimetric data of the alkali treated and untreated polyester fibers dyed with Dianix Rubine S-3B (Rubin color , CI35) at pH 4.5 (Conventional) and pH 8.5 (alkaline dyeing) are listed in Table 3. It can be seen that the (L^*) values of alkali treated/alkali dyed sample 7 and untreated/alkali dyed sample 5 decreased compared with those of the conventionally dyed samples 8 and 6, respectively. Additionally, the (a^*/b^*) values are noticeably increased for the former samples 7 and 5. This means that the color of such dyed samples become darker.

Table 2: Color strength (K/S) of the alkali treated 100% PET (16% weight loss), dyed with Dianix dyes at pH 8.5 compared to the control, untreated and conventionally dyed fabrics

Dianix dye (color)	λ_{\max} (nm)	Sample No.	PET fabric	K/S
Kaki	470	1	untreated	6.80
		2	treated	6.91
Gray	640	3	untreated	0.92
		4	treated	0.98
Rubine	530	5	untreated	16.53
		(6)	"	(16.07)*
		7	treated	17.89
Blue	640	(8)	"	(15.88)*
		9	untreated	14.89
		(10)	"	(15.23)*
		11	treated	15.72
		(12)	"	(14.86)*

* untreated and treated PET fabrics conventionally dyed at acidic pH 4.5

Table 3 Colorimetric CIE L*a*b* of the untreated and treated polyester fabrics dyed using 1.37 % (owf) Dianix Rubine S-3B (Rubin color, CI 35) at pH 4.5 (Conv.) and pH 8.5 (alkali) dyeing

Treatment	Dyeing	Sample No.	L*	a*	b*
Untreated	Conv.	6	34.29	49.38	-4.17
	alkali	5	34.12	49.92	-4.03
Treated	Conv.	8	33.87	47.95	-3.30
	alkali	7	33.51	48.23	-2.95

Table 4 shows the effect of alkali treatment on the fastness properties of the alkali and conventionally acid dyed polyester fabrics compared to untreated samples.

The results indicate that the alkali hydrolysis had no effect on the washing, perspiration and light fastness. Excellent to very good washing and perspiration fastness were detected. The light fastness obtained was also similar irrespective of the treatment and the dyeing process.

Table 4 Fastness properties of untreated and alkali treated polyester (16% weight loss) which are dyed with Dianix dyes under both alkaline and conventional acid conditions

Dianix dye (color)	PET * Sample No.	Washing fastness PET/C	Fastness to perspiration		Light fastness
			Acidic PET/C	Alkaline PET/C	
Khaki	1	5/5	5/5	5/5	7/8
	2	5/5	5/5	5/5	7/8
Gray	3	5/5	5/5	5/5	5/8
	4	5/5	5/5	5/5	5/8
Rubin	5	4/5	4-5/4-5	4-5/4-5	7/8
	(6)	4/5	4/5	4-5/5	6/8
	7	4/5	4-5/4-5	5/5	7/8
	(8)	4/5	4/5	4-5/5	6/8
Blue	9	4-5/4-5	4-5/4-5	5/5	5/8
	(10)	4-5/4-5	4/5	5/5	5/8
	11	4-5/4-5	4-5/4-5	5/5	5/8
	(12)	4-5/4-5	4-5/4-5	4-5/4-5	5/8

* For key see Table 2

Dyeing and fastness properties of polyester/viscose blend fabric

The color strength K/S values of the blend fabric dyed with four different Forosol dyes combinations in a single step dyeing at pH 4.5 and 120°C for 30 min are shown in Table 5. The alkali treated fabrics exhibited higher color strength values than the untreated samples. The increase in weight reduction may contribute to increase the hydrophilicity of the fabric. As a result, the dyes can diffuse more easily, improving the dye uptake of the alkali treated fabrics. These effects are pronounced with the different Forosol dyes combinations studied even with the darker blue marine color.

Table 5 Color strength (K/S) of the alkali treated and untreated polyester/viscose blend fabric conventionally dyed with different Forosol dye combinations at pH 4.5

Forosol dye (colo)	λ_{\max} (nm)	Sample No.	OET/Viscose blend fabric	K/S
Beige	460	13	untreated	0.65
		14	treated	0.66
Gray	470	15	untreated	2.78
		16	treated	2.91
Kaki	640	17	untreated	0.63
		18	treated	0.71
Blue Marine	560	19	untreated	19.92
		20	treated	20.76

Similarly, the colorimetric data of the alkali treated and untreated polyester/viscose blend fibers dyed with Forosol dyes at pH 4.5 are listed in Table 6. It can be seen that the (L^*) values of alkali treated and dyed samples 14 and 20 are marginally decreased compared with those of the untreated dyed samples 13 and 19, respectively. Additionally, the (a^*/b^*) values are slightly increased for the treated samples. This means that the dye uptake of the treated samples marginally increased.

The fastness results of untreated and alkali treated polyester/viscose blend dyed fabrics are shown in Table 7. The results indicate that the alkaline treatment had no effect on the washing, perspiration and light fastness of the dyed polyester/viscose blend fabrics. The washing fastness and perspiration fastness are excellent for all samples with exception of the Kaki color which showed very good ratings and very good light fastness is observed for all samples.

Table 6: Colorimetric CIE $L^*a^*b^*$ of the untreated and treated polyester/viscose blend fabrics dyed using Forosol dyes (CI undisclosed) at pH 4.5.

Treatment	Sample No.	L^*	a^*	b^*
Untreated	13	70.74	4.60	8.94
Treated	14	69.88	4.68	9.06
Untreated	19	21.18	7.01	-14.88
Treated	20	20.85	6.94	-14.51

Table 7 Fastness properties of the alkali treated and untreated polyester/viscose blend fabric dyed with different Forosol dye combinations at pH 4.5

Forosol dye (color)	PET/Viscose Sample No. *	Washing fastness PET/C	Fastness to perspiration		Light fastness
			Acidic PET/C	Alkaline PET/C	
Beige	13	5/5	5/5	5/5	6-7/8
	14	5/5	5/5	5/5	7/8
Gray	15	5/5	5/5	5/5	7/8
	16	5/5	5/5	5/5	6-7/8
Kaki	17	4/4-5	4/4-5	4/4-5	6-7/8
	18	4/4-5	4/4-5	4/4-5	6-7/8
Blue Marine	19	4/5	5/5	5/5	6/8
	20	4/5	5/5	5/5	7/8

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* For key see Table 5

Pilings and static charges measurements

The alkali treated and dyed 100 % polyester fabric (blue and Kaki) showed no pilings at the fabric surface and this finding is in agreement with other published articles which are cited in the literature review⁹ The untreated and alkali treated polyester exhibited static electricity over 10 KV and cannot be well determined due to the narrow range of the potentiometer (0-10 KV).

The alkali treated and dyed beige PET/Viscose fabric (65/35 %) shows a medium pilings effect compared to the untreated sample (high pilings) and there was no detection of static electricity.

Conclusion

Alkali hydrolysis of polyester fabric in HT mini jet machine using sodium hydroxide (7 g/l) in the presence of 1 gm/L cationic surfactant as an accelerator at 120°C was accompanied by 16% weight reduction and 30% loss in the tensile strength. In this procedure the sodium hydroxide concentration is low and the procedure is inexpensive compared to the conventional method using 170-180 gm/L caustic concentration and an accelerator , padding to 60-70% steaming 3-5 min at 103 to 110° C⁶. It has been shown that the weight reduction process improves the surface roughness, prevent the pilings in case of 100% polyester, improves the pilings and prevents the static charges for PET/Viscose blend .Under alkali and conventional acid dyeing, the alkali treated polyester fabrics exhibited higher color strength compared to the untreated fabrics using different Dianix dye combinations. The dyed fabric is for thought to benefit from the increase in hydrophilicity and decrease in fiber diameter. The fastness results were all satisfactory for the different dye recipes examined.

Low levels of weight reduction for PET / Viscose bland (7.5%) and tensile strength (32%) were observed at 95°C and were dyed using four different dye combinations of Forosol dyes in single step process at pH 4.5 and 120°C. Increased levels of dye uptake were observed for the alkali treated blend fabrics and the fastness properties are high.

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