Dyeability Improvement of Polyester Pretreated with Some Alkoxides

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ABSTRACT: The influence of some alkoxides in alcoholic media, having various dielectric constants, on the physical and mechanical properties was thoroughly studied in a previous work to attain silk-like polyester fibers. In this investigation, the dyeability behavior of this silk-like polyester is tried. Both immersion and padding techniques are applied in the treatment. The dyeability of the pretreated polyester fabric with disperse dyes shows some progressive improvements with lowering the dyeing temperature and/or decreasing the time of dyeing attained. Ethoxide is found to be more effective in enhancing the dyeability of polyester fabric than either methoxide or propoxide. Dyeing of the pretreated polyester fabric at the boil without using carriers or conducting high temperature/high pressure dyeing is also possible. Washing and crocking fastness are relatively enhanced. Physicochemical investigations of the dyeing process and a mathematical analysis for evenness are given. A decrease in the half dyeing time and an increase in the rate of dyeing of the pretreated polyester as compared with the untreated one are noticed. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 108: 7–13, 2008

Key words: polyester fibers; alkoxides; modification; dyeing kinetic; absorbency; disperse dye

INTRODUCTION

Some methods are involved to overcome the low water absorption of polyester fibers to improve its dyeability. The compactness of the structure of polyester fibers minimizes the rate of the dye diffusion. To overcome this difficulty, swelling agents or high temperature treatments are used. Three techniques are generally practiced: aqueous dyeing at the boil in presence of carriers; aqueous dyeing at elevated temperature and high pressure; and the thermosol method.[1,2] Carriers, high temperature/high pressure (HT/HP) processes are traditionally used. As an alternative, comonomers can be incorporated,[3] but it is accompanied by lowering the crystallinity and decreasing the melting temperature of the polymer upon increasing the concentration of the comonomers.[3]

The orientation of the chains in a fiber polymer determines the rate of diffusion and the dyeing rate.[4,5] The variation of the dyeing properties of polyester fibers is always given in terms of structural changes by using a two-phase theory involving crystalline and amorphous regions.[6] Preswelling and plasticization of polyester fibers promote its physico-mechanical properties,[4–6] moisture regain,[7,8] as well as dyeability characteristics.[9–11]

Treatment of polyester fabric with ethanolic sodium hydroxide solution showed a significant improvement in the absorbency behavior of the polyester fabric, such as the decrease in wicking time and the relative increase in the moisture regain percentage, as compared with the untreated mate.[12] Previous work to obtain silk-like polyester by its treatment with different alkoxides was carried out.[13,14]

Raslan and Bendak[15] studied the action of alkali treatment in both aqueous and alcoholic media, in a previous work, to attain silk-like polyester fabric. The use of alkoxide solutions is more effective on polyester fabric. Rapid loss in weight up to 15–20% occurs at ambient conditions. Immersion and padding techniques are applied. The effect of treatment on some properties of polyester fabric is given through measurements of tensile strength, draping, permeability, density gradient, crystallinity, thermogravimetric analysis, and scanning electron microscopy. Optimization of the treatment conditions is also suggested.[15]

The present work is endeavored to investigate the effect of sodium hydroxide in an alcoholic media having various dielectric constants on the improvement of dyeability of the treated polyester fabric. This also can hinder oligomer liberation from the dyed fabrics. Comparative physicochemical investigations of dyeing the pretreated and untreated poly-

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ester fabrics as well as mathematical analysis of the dyeing evenness are also tried. Further work on a comparative techno-economic and environmental investigation of the different dyeing processes (dyeing using carriers, HT/HP dyeing process, and the method applied method in this article) will be carried out in due time.

EXPERIMENTAL

Material

Polyester fabric was provided by Misr Rayon Co., Kafr El-Dawar, Egypt. All chemicals used were of reagent grade. Disperse dyes, C.I. Disperse Red 60 (based on anthraquinone structure) and C.I. Disperse Red 82 (based on monoazo structure) were applied.

Treatments

Immersion technique

Polyester samples of known weight were treated in 0.25*M* sodium hydroxide solution in alcoholic media at room temperature (~ 25°C) for 24 h to attain silk-like polyester having a loss in weight of about 15–20%.[15] This was done by soaking the substrate in alcoholic sodium hydroxide solution (liq. ratio 1 : 50) with occasional shaking. A control sample was prepared by treating the polyester fabric in the corresponding alcohol at the same conditions. The samples were then rinsed thoroughly and air dried at ambient conditions. Applying the immersion technique makes the residual solution after treatment milky because of the soluble polyester fragments, and hence the reuse of the same solution needs more than one purification process. Padding technique was tried to overcome this disadvantage.

Padding technique

Padding technique was performed by soaking the fabric in 1*M* sodium hydroxide solution in alcoholic media at room temperature (~ 25°C), followed by squeezing under pressing rollers to 100% pick up. The samples were stored in polyethylene bags overnight at room temperature. The samples were then rinsed thoroughly, air dried, and allowed to attain constant weight.

Dyeing procedure

Polyester samples of known weights were exhaust-dyed in the dye bath at different temperatures (80–130°C) for various time intervals (10–120 min). The dyeing solution was adjusted to a pH 4.5 by adding acetic acid. Reductive washing of the dyed samples was normally performed using a solution containing 0.5 g/L soda ash, 2 g/L sodium hydrosulphite, and 1 g/L of a stable surfactant for 15 min at 70°C to ensure the removal of extra dye physically adhered on the fiber surface. The dyed polyester fabric was warm and cold rinsed with water and then further rinsed with an aqueous solution acidified with 1 g/L acetic acid. The samples were then rinsed thoroughly with water and air dried at room temperature. Two different structural dye moieties (anthraquinone and monoazo) were applied in this investigation. Both dye moieties are of moderate leveling as well as having good build up on the substrates, but differ in their molecular volume and configurations. However was their energy level type (low, medium, or high), the alkoxide pretreated polyester fiber is the fiber to be investigated with respect to dyeing as compared with the untreated one by maintaining the dyestuff molecule characters and dyeing conditions constant.

Color measurements

The spectral reflectance measurements of the dyed fabric were carried out using a recording filter spectrophotometer (model ICS Texicon Ltd., Kennetside Park Newbarge, Berkshire AG 145TE, UK). The color intensity expressed as K/S values of the dyed samples was determined by applying the Kubelka–Munk equation:

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

where R is the decimal fraction of the reflectance of the dyed sample, R_o is the decimal fraction of the reflectance of the undyed sample. K is the absorption coefficient and S is the scattering coefficient.[16]

The relative color intensity is estimated by applying the following equation:

$$\text{The relative color intensity \%} = \frac{K/S \text{ of pretreated sample}}{K/S \text{ of untreated sample}} \times 100 \quad (2)$$

Color fastness

Crocking fastness

The color fastness to crocking was measured according to the AATCC test method 8, 1972. Both dry and wet crocking were measured.[17]

Washing fastness

A color fastness to washing was measured according to the AATCC test method 83-1974 using a laundrometer. Evaluation of the washing fastness was given using the Gary Scale reference for color change.[17]

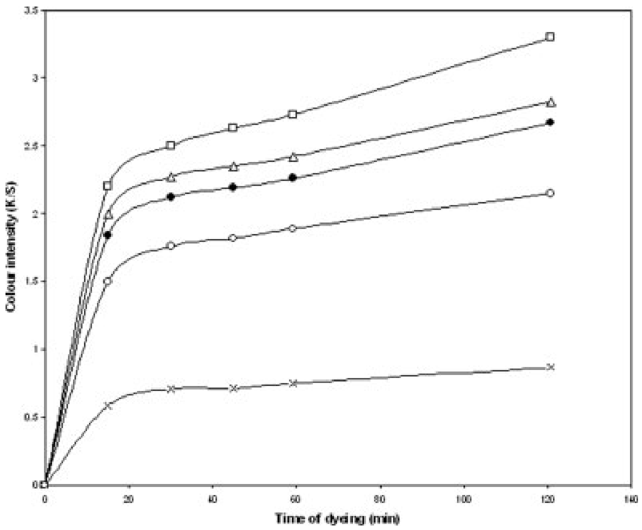

Figure 1 Dependence of the acquired color intensity by the dyed polyester and alkoxides pretreated fabrics on the time of dyeing. Treatment: 0.25*M* sodium hydroxide solution in alcoholic media, immersion technique, room temperature (~ 25°C), 24 h, liq. ratio 1 : 50, ×—× untreated, ○—○ pretreated with butoxide, ●—● pretreated with methoxide, △—△ pretreated with propoxide, □—□ pretreated with ethoxide. Dyeing: 1% (o.w.f) C.I. Disperse Red 82, 85°C, pH 4.5, liq. ratio 1 : 50.

RESULTS AND DISCUSSION

A systematic study on the influence of some alkoxides treatments on the dyeing of polyester fabric with disperse dye was performed to optimize new possibilities to dye silk-like polyester fabric at a lower temperature than the conventional dyeing one without using a carrier and/or the HT/HP routine.

Immersion technique

Polyester substrate pretreated with 0.25*M* sodium hydroxide in an alcoholic media, such as methanol, ethanol, propanol, or butanol at room temperature (~ 25°C) by immersion technique for 24 h, were exhaust dyed using disperse dye solutions. The results are illustrated in Figures 1 and 2.

Figure 1 shows the dependence of color intensity of the pretreated polyester fabric on the time of dyeing at 85°C with 1% C.I. Disperse Red 82. It can be noticed that the ethoxide solution was the most effective one in enhancing the dyeability of the pretreated polyester fabric. Figure 2 shows the dependence of color intensity of the pretreated polyester fabric on the dielectric constant (*D*) of the used alcohols in relation to the time of dyeing. The values of *D* for water, methanol, ethanol, and propanol are 78.5, 32.6, 24.3, and 19.7, respectively. Dielectric constant is defined by the relative permittivity of the matter. Physically, the dielectric effect is because of the polarization of the matter in the media.[18] It can be seen that the mentioned relation was highly enhanced. It can also be seen that the higher the $1/D$ value of the used alcohol the higher is the color intensity of the dyed polyester fabric till $1/D$ corresponding to ethanol; then the color intensity is slightly decreased by increasing $1/D$ (corresponding to propanol). The higher value of the attained color intensity corresponding to pretreatment with ethoxide holds well with previous work.[11] The decrease in color intensity of the pretreated polyester with propoxide may be because of the high volume of propoxide, which may restrict the penetration of the dye molecules between the fiber structure; besides the effect of sodium alkoxide occurred at the periphery rendering the fibers less porous, more dense, and more crystalline.[15,19]

Padding technique

Figure 3 shows the dependence of color intensity of pretreated polyester fabric with 1*M* sodium hydroxide solution in ethanol and propanol by padding technique at room temperature (~ 25°C) and stored for 24 h during the time of dyeing. It can be seen that the aforementioned treatment enhanced the dyeability of polyester fabric at 85°C when using dyeing shades of 0.5–3% (o.w.f.). The maximum color intensity ($K/S \approx 4$) was obtained by treat-

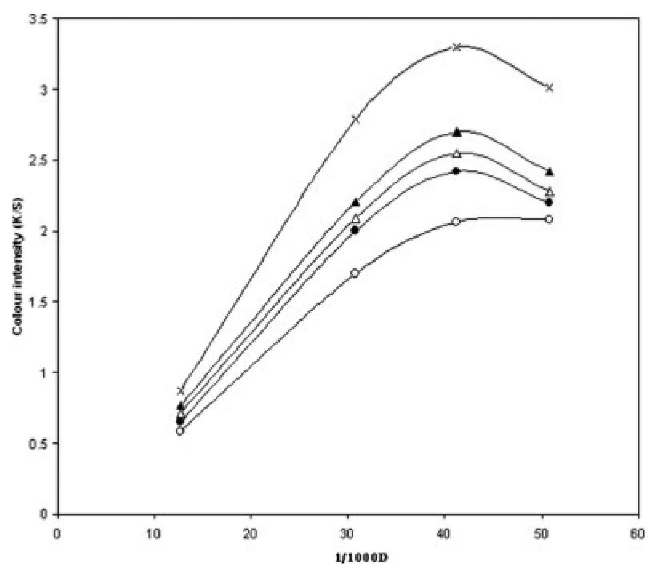

Figure 2 Dependence of the acquired color intensity by the dyed pretreated polyester fabric on the dielectric constant of alcoholic media of the used alkoxide. Treatment: 0.25*M* Na OH in alcoholic media, room temperature (~ 25°C), immersion technique, 24 h, liq. ratio 1 : 50, $1/D$ values: water, 12.7×10^{-3}; methanol, 30.7×10^{-3}; ethanol, 41.2×10^{-3}; propanol, 50.8×10^{-3}. Dyeing: 1% (o.w.f.) C.I. Disperse Red 82, pH 4.5, 85°C, liq. ratio 1 : 100, ○—○ 15 min, ●—● 30 min, △—△ 45 min, ▲—▲60 min, ×—× 120 min.

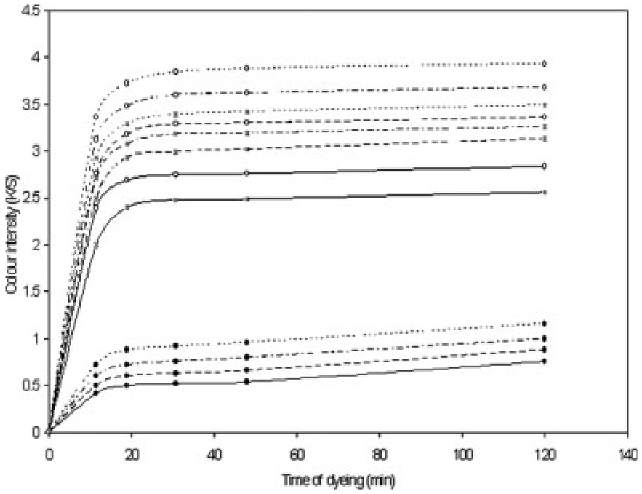

Figure 3 Dependence of the acquired color intensity by the dyed polyester and alkoxide pretreated fabric on the time of dyeing using various dye concentrations. Treatment: 1*M* sodium hydroxide in alcoholic media, padding technique, pick up 100%, stored at room temperature for 24 h, ●—● untreated, ×—× pretreated with propoxide, ○—○ pretreated with ethoxide. Dyeing: — 0.5%, - - -1%, -·-·- 2%, ···· 3% (o.w.f.) C.I. Disperse Red 82, 85°C, pH 4.5, liq. ratio 1 : 50.

ment with ethoxide compared with $K/S \approx 1$ for the untreated one and about 3.5 for pretreated polyester with propoxide.

Padding technique did not reveal an increase in sodium hydroxide in the fiber vicinity even when using 100% pick up. This corresponds to 4 g/100 g fiber; whereas in the immersion technique, using 0.25*M* solution, liquor ratio 1 : 50 it gives rise to 50 g/100 g fibers. Both techniques revealed nearly the same loss in weight[15] and consequently having nearly the same effects on dyeing behavior (Figs. 1 and 3). It seems probable that the padding technique has a privilege as compared with the immersion technique. Sodium hydroxide plays its role in the narrow vicinity in affecting the treatment significantly. In the padding technique, alcoholic sodium hydroxide solution can be reused several times without impairing its clearance.

Figure 4 illustrates the relation between the relative color intensity % of the pretreated polyester fabric dyed with C.I. Disperse Red 60 and the time of dyeing at different temperatures (80 and 90°C). It can be seen that the relative color intensity % was found to be increased. This increase in relative color intensity diminishes with increasing the dyeing time. The same trend is also given in Table II, because of increasing the rate of dye uptake (tan α) of the pretreated polyester fabric dyed with C.I. Disperse Red 82 in the early stage of dyeing.

Dyeing of pretreated polyester fabric with either ethoxide or propoxide was also performed at 100°C. The results are illustrated in Figure 5. It can be seen that the treatment of polyester fabric with either ethoxide or propoxide enhances the dyeability of polyester fabric with disperse dye at the applied dyeing shades (0.5–2% o.w.f.).

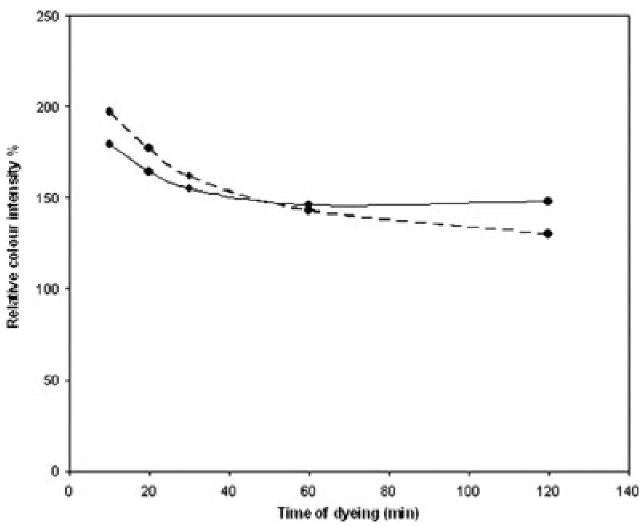

Figure 4 Effect of time of dyeing on the relative color intensity % of pretreated polyester fabric with propoxide. Treatment: 1*M* sodium hydroxide in propanol, padding technique, pick up 100%, stored at room temperature for 24 h. Dyeing: 1% (o.w.f.) C.I. Disperse Red 60, — 80°C, - - - 90°C, pH 4.5, liq. ratio 1 : 50.

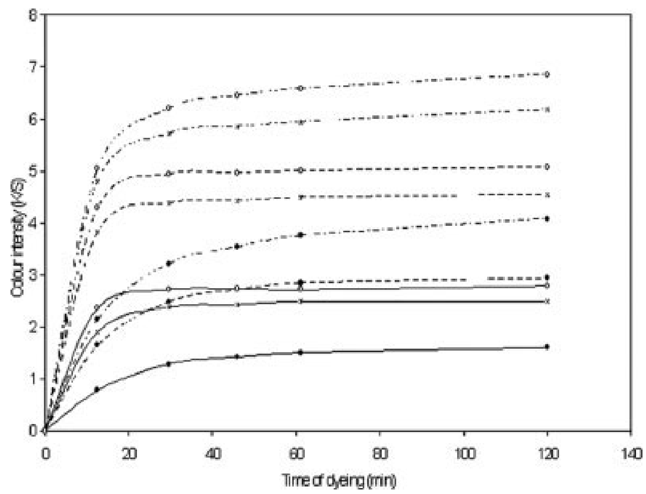

Figure 5 Dependence of the color intensity acquired by the pretreated polyester fabric with alkoxide using padding technique on the time of dyeing at the boil. Treatment: 1*M* sodium hydroxide in alcoholic media, padding technique, pick up 100%, stored at room temperature for 24 h, ●—● untreated, ×—× pretreated with propoxide, ○—○ pretreated with ethoxide. Dyeing: — 0.5%, - - - 1%, -·-·- 2% C.I. Disperse Red 82 (o.w.f.), 100°C, pH 4.5, liq. ratio 1 : 50.

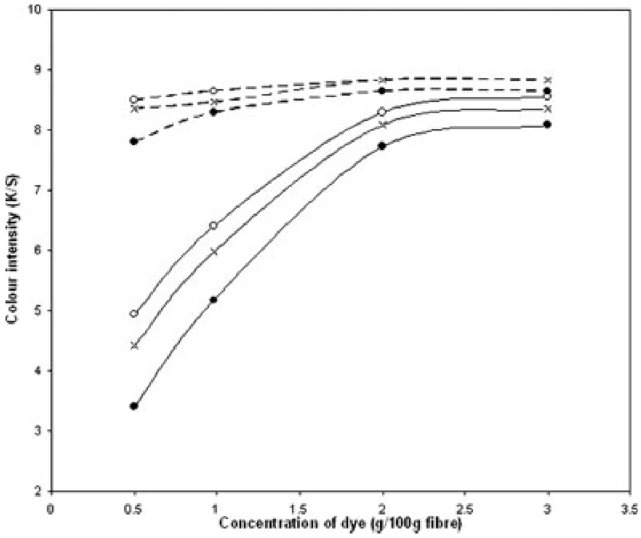

Figure 6 Dependence of the color intensity acquired by the pretreated polyester fabrics with alkoxide and dyed at HT/HP on the concentration of the used dye. Treatment: 1*M* sodium hydroxide in alcoholic media, padding technique, pick up 100%, stored at room temperature for 24 h, ●—● untreated, ×—× pretreated with propoxide, ○—○ pretreated with ethoxide. Dyeing: C.I. Disperse Red 82, 130°C, - - - 1 h, —10 min, pH 4.5, liq. ratio 1:50.

High temperature/high pressure dyeing

The untreated and pretreated polyester fabric with ethoxide and propoxide were dyed with C.I. Disperse Red 82 at 130°C by HT/HP dyeing technique for 10 and 60 min. Figure 6 represents the relation between the concentration of applied dye and the corresponding color intensity of polyester fabric. It can be seen that the color intensity of the pretreated polyester fabric was improved at the applied dyeing shades. It was also shown that the resulting color intensity of untreated polyester fabric at a given dye concentration can be attained by using lower dye concentration or by pretreating the fabric with either ethoxide or propoxide.

TABLE I
Fastness Properties of Dyed Polyester Fabric

	Washing fastness		Crocking fastness	
Type of sample	St	Alt	Wet	Dry
Untreated polyester fabric	4–5	4	3–4	4
Pretreated polyester fabric with ethoxide	4–5	4–5	4	4–5
Pretreated polyester fabric with propoxide	4–5	4–5	4	4–5

Treatment: 1*M* sodium hydroxide in alcoholic media, padding technique, pick up 100%, stored at room temperature for 24 h in polyethylene bags. Dyeing: 2% (o.w.f.) C.I. Disperse Red 82, 100°C, 60 min, pH 4.5, liq. ratio 1 : 50. St, staining and Alt, alteration.

Fastness properties

Dyed pretreated samples with moderate shade at optimum conditions were chosen to investigate the change in fastness ratings. The fastness properties including washing fastness and crocking fastness (dry and wet) for both untreated and pretreated polyester fabric with ethoxide and propoxide are shown in Table I. It can be noticed that the fastness properties of dyed pretreated fabric were relatively enhanced.

Physicochemical investigation

Figure 7 illustrates the relation between the color intensity of the pretreated polyester fabric with ethoxide and propoxide by padding technique and the square root of the dyeing time at 85°C. Straight lines are given. The slope of the straight line as indicated by tan α, where α is the degree of inclination of the straight line,[20,21] resulted in values 0.106, 0.569, and 0.663 for the untreated and pretreated polyester fabric with propoxide and ethoxide, respectively. It can be noticed that the rates of dyeing of the pretreated polyester with both propoxide and ethoxide significantly increased at a dyeing temperature of 85°C using C.I. Disperse Red 82. Ethoxide pretreatment was found to be more effective in increasing the rate of dyeing of the polyester fabric with disperse dye than the propoxide pretreatment.

The rate of dyeing of the polyester fabric pretreated with ethoxide and dyed with different shades of disperse dye at 100°C is shown in Figure 8. The half dyeing time ($t_{1/2}$) and the rate of dyeing (tan α)

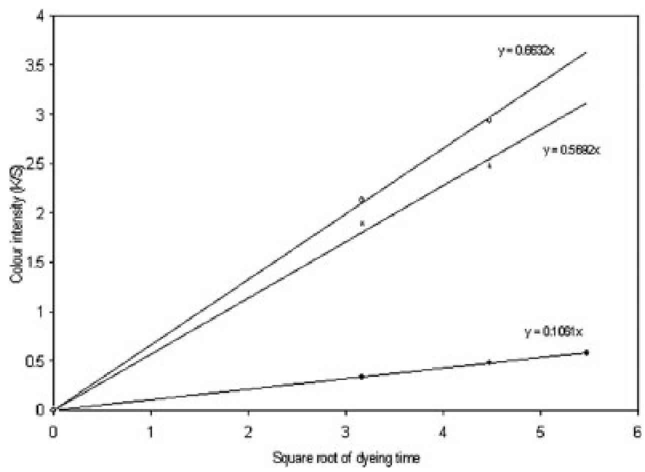

Figure 7 Dependence of the color intensity acquired by the dyed polyester and alkoxide pretreated fabrics on square root of time at the early stage of dyeing. Treatment: 1*M* sodium hydroxide in alcoholic media, padding technique, pick up 100%, stored at room temperature for 24 h, ●—● untreated, ×—× pretreated with propoxide, ○—○ pretreated with ethoxide. Dyeing: 0.5% (o.w.f.) C.I. Disperse Red 82, 85°C, pH 4.5, liq. ratio 1 : 50.

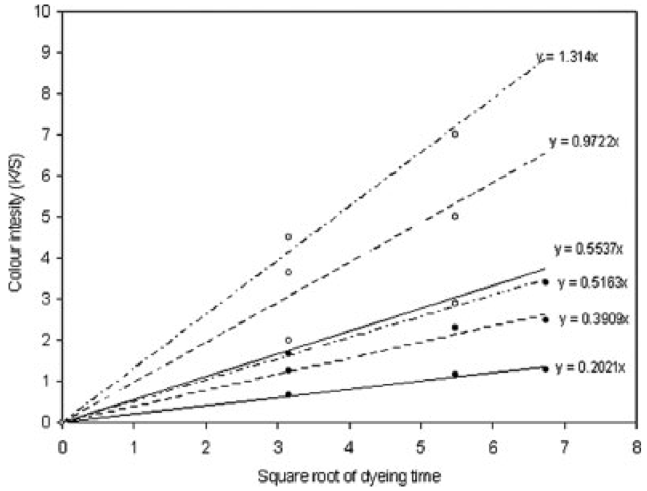

Figure 8 Dependence of the color intensity acquired by the pretreated polyester fabrics with ethoxid and dyed at the boil on the square root of time at the early stage of dyeing. Treatment: 1*M* sodium hydroxide in ethanol, padding technique, pick up 100%, stored at room temperature for 24 h, ●—● untreated, ○—○ pretreated with ethoxide. Dyeing: — 0.5%, - - - 1%, -·-·- 2% (o.w.f.) C.I. Disperse Red 82, 100°C, pH 4.5, liq. ratio 1 : 50.

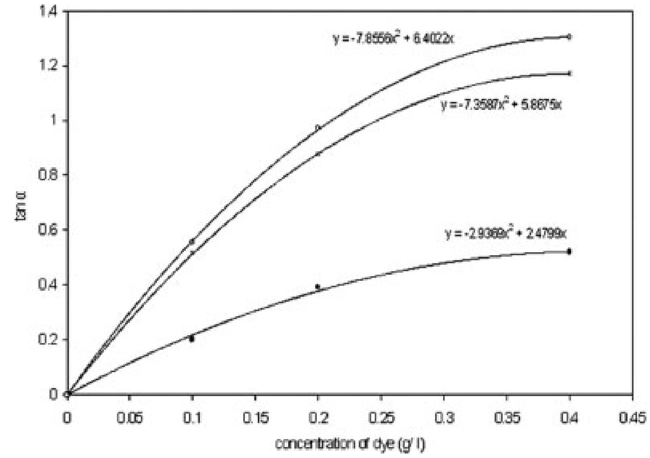

Figure 9 Dependence of tan α value on the concentration of dye used in dyeing of polyester and the alkoxide pretreated fabrics. Treatment: 1*M* sodium hydroxide in alcoholic media, padding technique, pick up 100%, stored at room temperature for 24h, ●—● untreated, ×—× pretreated with propoxide, ○—○ pretreated with ethoxide. Dyeing: C.I. Disperse Red 82, 100°C, pH 4.5, liq. ratio 1 : 50.

for the untreated and the pretreated polyester fabric were estimated and tabulated in Table II. The half dyeing time of the pretreated polyester fabric was found to decrease to about half of the untreated one, whereas the rate of dyeing (tan α) of the pretreated polyester was found to increase to more than 2–3 folds of untreated sample.

The influence of the variation of dye concentration in the solution on the extent of dyeing was studied by plotting tan α against the dye concentration.[21] This relation is illustrated in Figure 9. The resultant curve was found to rise moderately at low dye concentration up to 0.2 g/L, and then the increase of tan α was slowed down by increasing the dye concentration up to 0.4 g/L for both dyed untreated and pretreated polyester fabric. The assumed equations of the curved line in Figure 9 are:

$$Y = -2.9369x^2 + 2.4799x$$

for untreated polyester fabric,

$$Y = -7.3587x^2 + 5.8675x$$

for pretreated polyester with propoxide,

$$Y = -7.8556x^2 + 6.4022x$$

for pretreated polyester with ethoxide

By differentiation of the previous equations (y') that represent the tangent of the curve at any point, it was found that $y'_{x=0.1} = 1.9$ and $y'_{x=0.2} = 1.3$ for the untreated sample, whereas the corresponding values for the pretreated sample with propoxide are 4.4 and 2.9 and that for the pretreated polyester with ethoxide are 4.8 and 3.2, respectively. These results indicate that the rate of tan α (y') is found to decrease by increasing the dye concentration. This would enhance the evenness of the dyed pretreated polyester fabric despite increasing the rate of dyeing, as proved by the above-mentioned mathematical analysis.

TABLE II
Half Dyeing Time ($t_{1/2}$) and the Rate of Dyeing (tan α) of Untreated and Alkoxide Pretreated Polyester Fabrics

Type of sample	Dyeing shade % (o.w.f.)	$t_{1/2}$ (min)	tan α
Untreated	0.5	10	0.2021
Pretreated with propoxide		6	0.5163
Pretreated with ethoxide		5	0.5537
Untreated	1	8	0.3909
Pretreated with propoxide		5	0.8768
Pretreated with ethoxide		4	0.9722
Untreated	2	15	0.5163
Pretreated with propoxide		6	1.1886
Pretreated with ethoxide		5	1.314

Treatment: 1*M* sodium hydroxide in alcoholic media, padding technique, pick up 100%, stored at room temperature for 24 h in polyethylene bags. Dyeing: C.I. Disperse Red 82, 100°C, pH 4.5, liq. ratio 1 : 50.

CONCLUSION

Treatment of polyester fabric with some alkoxides, viz methoxide, ethoxide, propoxide, and butoxide by both immersion and padding techniques enhances the dyeability of polyester fabric with disperse dye. Ethoxide is found to be the most effective one in this respect. The rate of the acquired color intensity increases and the half dyeing time also decreases. Pretreated polyester fabric can be dyed successfully at the boil without adding carrier, without using HT/HP dyeing technique, and preventing the oligomer formation.

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