

# Exhaust Dyeing of Polyester-Based Textiles Using High-Temperature–Alkaline Conditions

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**ABSTRACT:** The factors affecting the dyeability of polyester-based textiles with disperse dyes in an alkaline medium were studied. It was found, for a given set of dyeing conditions, that (a) the appropriate conditions for attaining a higher color yield were 45 min at 130°C with pH 9 using a material-to-liquor ratio of 1/10; (b) increasing the Diaserver® AD-95 concentration to 2% ows (based on weight of substrate) as well as including triethanolamine to 2% ows in the dyeing formulations bring about a significant improvement in the dye uptake; (c) both a preheat setting from 160 to 200°C/30 s and an alkaline weight reduction have a positive impact on postdyeing with the used disperse dye; (d) the extent of dye uptake as well as the color strength are governed by the type of substrate, that is, knitted fabric > spun yarn > woven fabric, nature of the dye stabilizer,

that is, EDTA > Diaserver® AD-95 > Tinoclorite® CBB > citric acid > none, as well as kind of the disperse dye; (e) direct reuse of the disperse dyebaths, without reconstitution, in the dyeing of the used substrates was shown to be feasible in a single shade and in the reverse-order dyeings (dark → light); (f) one-bath, one-step exhaust dyeing of polyester/cotton-knitted fabric using selected disperse reactive dyes combinations under high-temperature alkaline conditions is feasible; and (g) the color and fastness properties of the resultant dyeings depend on the type of the used auxiliaries, in addition to the nature of disperse/reactive dyes combinations as well as compatibility with other ingredients. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 89: 3563–3573, 2003

**Key words:** blends; dyes; polyester

## INTRODUCTION

The use of polyester fibers in many textile applications is growing very rapidly due to their high strength, good elastic recovery, dimensional stability after heat setting, as well as suitability for blending with natural fibers.<sup>1,2</sup> However, the main drawbacks in polyester-based textiles, for example, low moisture content, static accumulation, soiling, uncomfortable feel, pilling tendency, and difficulty in dyeing, attributed to their high crystallinity, compactness, hydrophobic nature, and absence of chemically reactive groups.<sup>3,4</sup> Therefore, considerable efforts and technical developments have been done to upgrade their quality and usefulness, for example, antistatic finish, soil-release finish, water-repellent finish, antipilling finish, flame-retardant finish, and silklike finish,<sup>4–11</sup> as well as to enhance their dyeing properties, for example, carrier-free dyeing, package dyeing, incorporation of anionic or cationic active sites in the fiber, thermosol dyeing, and dyeing in an alkaline medium.<sup>12–19</sup> So far, there have been only a few studies concerned with the alkaline dyeing system for polyester fibers.<sup>12–16</sup> On the other hand, many attempts and technological efforts

have been made to develop continuous dyeing of polyester/cotton blends using disperse/reactive dyes, but they have had little success in industrial applications.<sup>20–24</sup>

Accordingly, the main tasks of the present work were (i) to evaluate the impact of dyeing conditions and formulations, heat setting, and alkaline weight reduction on the extent of exhaustion, (ii) to maximize the extent of fixation of disperse dyes onto the polyester-based textiles in an alkaline medium, (iii) to examine the feasibility of disperse dye bath reuse as an effective method for pollution-source reduction and materials saving, in addition (iv) to study the technical feasibility of exhaust dyeing of polyester/cotton blend using disperse/reactive dyes combinations under high-temperature alkaline conditions.

## EXPERIMENTAL

### Materials

Commercially available 100% polyester substrates: woven fabric (70 den/34 filament, 210 g/m<sup>2</sup>), a circular knit Jersey fabric (50/34 textured yarn, 160 g/m<sup>2</sup>), and spun yarn (40S, twofold, 2.0 dtex), were used throughout this work. A mill-scoured and bleached polyester/cotton blend (50/50) knitted fabric (circular knitted interlock, 40 S × 40 S, 265 g/m<sup>2</sup>) was used.

The commercial disperse dyes used were Dianix® Navy Blue AD-G, Dianix® Yellow AD-G, Dianix® Red

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AD-2B, Dianix® Orange AD-R, Dianix® Rubbin AD-B, and Dianix® Blue AD-R from Dystar (Frankfurt, Germany). The commercial reactive dyes used in this work were Procion® Sapphire HEXL, Procion® Flavine HEXL, and Procion® Red HE3B from Dystar.

Textile auxiliaries used were Diaserver® AD-95 (has many functions, such as in stabilizing dyestuffs, sequestering agents, buffering, and dissolving oligomers; Dystar), Eganal® PSN (linear polycondensate, leveling and dispersing agent for disperse dyes; Clariant, Muttenz, Switzerland), Dispersogen® P (sulfonate of a formaldehyde condensation product, dispersing agent mainly for disperse dyes; Clariant), Dodigen® 226 (quaternary ammonium compound; Clariant), as well as Tinoclorite® CBB (based on gluconic acid, peroxide stabilizer; Ciba, Basel, Switzerland). Hostapal® CV-ET (a nonionic wetting agent based on alkyl aryl poly(glycol ether); Clariant), in addition to Cibapone® R (a nonionic detergent with powerful wetting and dispersing properties, based on alkyl aryl sulfonate; Ciba). All the chemicals such as sodium hydroxide, soda ash, sodium chloride, borax, citric acid, ethylenediamine tetracetate (EDTA), triethanolamine (TEOHA) and acetic acid were of commercial grade.

## Methods

### Alkali dyeing process

Samples of polyester substrates were dyed at a material-to-liquor ratio (LR) of 1/10, with a 2% ows (based on weight of the substrate) commercial disperse dye solution. The standard dyeing profile is shown in Figure 1.<sup>16</sup> Washing-off was carried out according to the manufacturer's instructions to remove unfixed dissolved oligomers as well as residual auxiliaries.

### Heat setting

Heat setting of the polyester fabric samples were carried out in a hot-air curing oven in a taut condition for 30 s at 160–200°C.

### Weight reduction

Woven fabric samples were treated at 80°C for 30 min with a liquor ratio of 20:1, including small quantities of Dodigen® 226 (0–1 g/L) as an accelerant, in an aqueous sodium hydroxide solution (10 g/L). The treated samples were then rinsed in water to remove any residual (alkali, accelerator, as well as hydrolysis products), neutralized with acetic acid, then thoroughly rinsed again with distilled water until the rinse water was neutral, and, finally, dried under standard conditions. Weight loss was determined gravimetrically.<sup>5,6,25</sup>

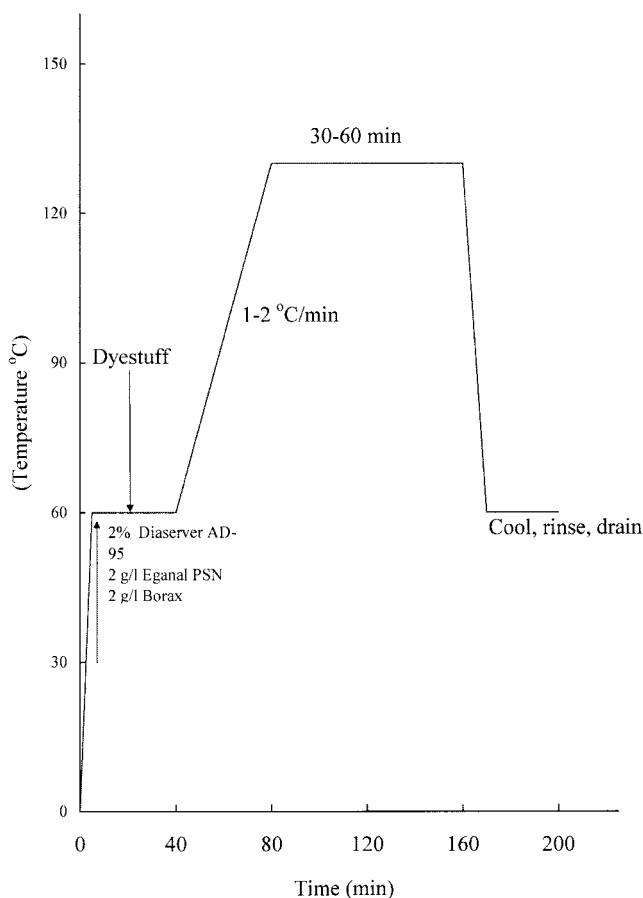


Figure 1 Dyeing cycle used in the present study.

### Dyebath reuse

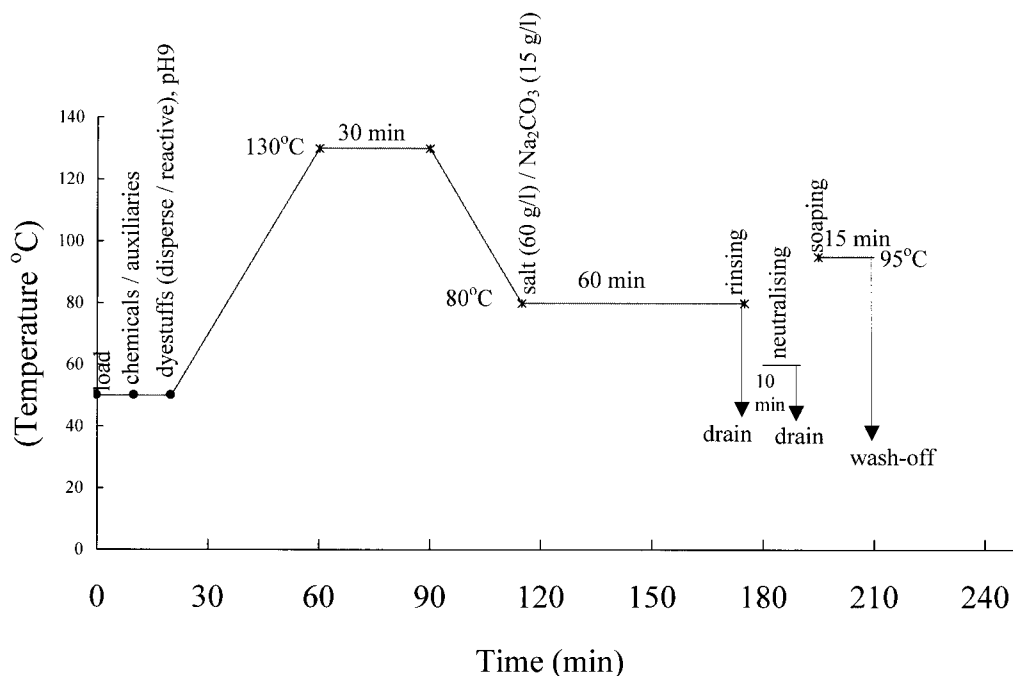
The exhausted hot dyebaths were reused to dye a number of batches of material, without analysis, to determine the concentrations of residual dyestuffs and auxiliaries, that is, without reconstitution, keeping the LR fixed to obtain lighter shades for economical and ecological reasons.

### Disperse/reactive dyeing of polyester/cotton knitted fabric

The standard dyeing profile is shown in Figure 2. Washing-off was carried out according to the manufacturer's instructions to remove unfixed and/or hydrolyzed dyes as well as residual salts, fixing agents, and other auxiliaries.

### Testing

The color intensity, expressed as the *K/S* value, of the dyed substrate was measured at the wavelength of maximum absorbance using an automatic filter spectrophotometer and calculated by the Kubelka–Munk equation<sup>26</sup>:



**Figure 2** Dyeing cycle used in the present study (rapid dyeing/one bath). Chemical/auxiliaries: borax (2 g/L); Dispersogen® P (0.5 g/L); Eganal® PSN (2 g/L); Diaserver® AD-95 (2% owf, based on weight of fabric), unless otherwise notified; with a liquor ratio of 10:1, in presence or absence of TEOHA (0.5% owf). Dyestuffs: The disperse/reactive dyes combination (2% owf), pH 9.

$$K/S = (1 - R)^2/2R$$

where  $K$  is the light-absorption coefficient;  $S$ , the light-scattering coefficient; and  $R$ , the reflectance of the dyed sample. The higher the  $K/S$  value, the greater is the color intensity and, hence, the better is the dye uptake.

Weight loss by alkaline hydrolysis was determined gravimetrically and calculated by the equation<sup>6</sup>

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

where  $W_1$  and  $W_2$  are the weight before and after treatment, respectively. Fastness properties to washing, crock, as well as light of the dyed samples were assessed according to AATCC Test Methods (61-1972), (8-1972), and (16A-1972), respectively.

## RESULTS AND DISCUSSION

### Factors affecting disperse dyeing of polyester-based textiles in an alkaline medium

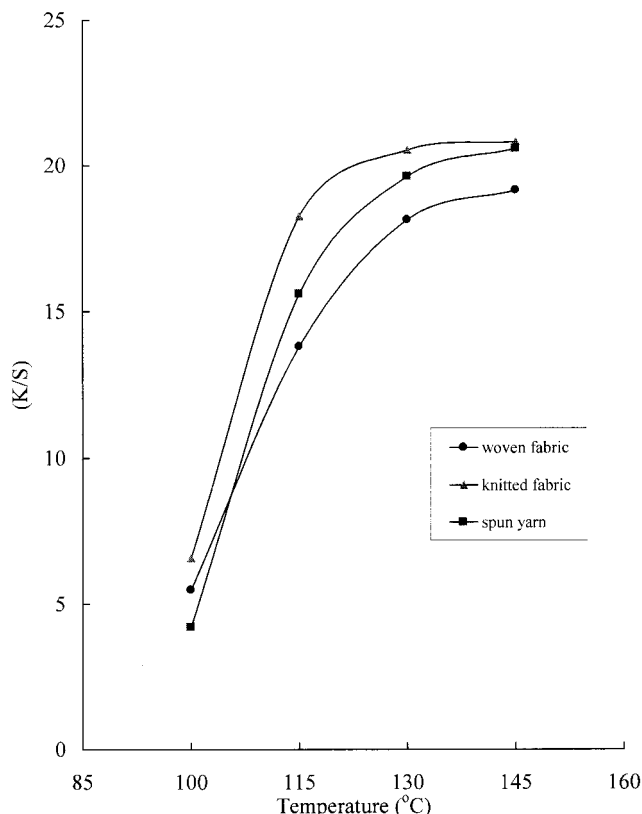
Since the primary objective of the present work was to study such factors affecting the dyeability of polyester with disperse dyes in an alkaline medium as well as to evaluate the possibility of disperse dyebath reuse, a wide range of parameters such as dyeing conditions, that is, temperature and time, LR, type and concentration of auxiliaries and additives, pH, preheat-setting,

alkaline weight reduction, in addition to reuse of the disperse dyebath were examined.

### Dyeing temperature

Figure 3 shows the effect of the dyeing temperature on the extent of dyeing the polyester substrates (woven and knitted fabrics as well as spun yarns) with the used disperse dye (Dianix® Navy Blue AD-G, 2% ows), expressed as  $K/S$  values. It is clear that, for given dyeing conditions, increasing the dyeing temperature to 130°C gives rise to a remarkable increase in the  $K/S$  values of the obtained polyester dyeings, regardless of the used substrate, which is a direct consequence of (i) enhancing the swellability and minimizing the compactness of the polyester structure, thereby facilitating the segmental mobility of the chains to exhibit enhance dye uptake<sup>27-28</sup>; (ii) helping the disaggregation, solubility, as well as mobility of the dye molecules, thereby speeding up and enhancing the diffusion and penetration of the dye molecules in the amorphous domain and ultimately promoting dye uptake<sup>29</sup>; along with (iii) overcoming the activation energy barrier of the dyeing process, thereby enabling and improving the extent of exhaustion and dye uptake.<sup>19,30</sup>

On the other hand, further increase in the dyeing temperature above 130°C does not seem to significantly affect the dye uptake, expressed as an improve-



**Figure 3** Variation of the  $K/S$  values with the dyeing temperature. Dyeing bath constituents: Dianix® Navy Blue AD-G (2% ows); Diaserver® AD-95 (2% ows); Eganal® PSN (2 g/L); Dispersogen® P (0.5 g/L); borax (2 g/L); pH 9; LR, 1/10; dyeing time, 30 min.

ment in the  $K/S$  values, which may be explained in terms of lower stability of the disperse dye dispersion at a higher dyeing temperature, which, in turn, gives rise to a higher tendency of the aggregation of the disperse dye molecules,<sup>31</sup> shortage in the dye bath components via exhaustion, as well as blocking or inaccessibility of the so-called amorphous regions of the polymer.

It is also clear (Fig. 3) that the  $K/S$  values of the dyed substrates follows the descending order

knitted polyester > spun yarn > woven polyester

which would be expected to rely on the fiber structure, fiber cross section, surface area, degree of orientation, location and accessibility of dyeing sites, textile form, fabric construction, as well as proportion of the dye distribution among the aforementioned substrates.<sup>32</sup>

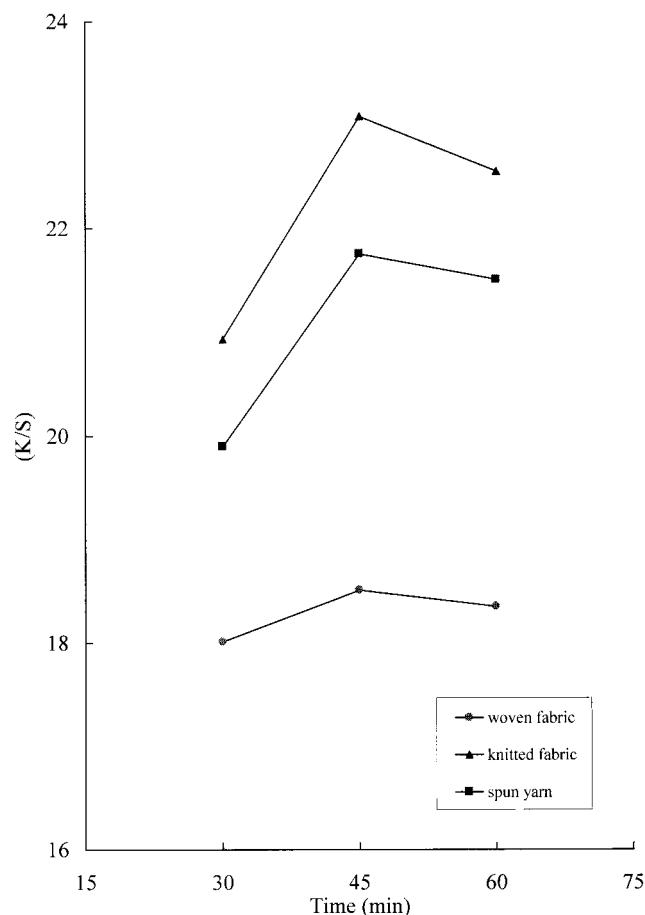
#### Dyeing time

Figure 4 shows the effect of the dyeing time on the extent of dye uptake, expressed as the  $K/S$  value, of the dyed polyester substrates. As is evident, prolonging the dyeing duration to 45 min at 130°C brings about a

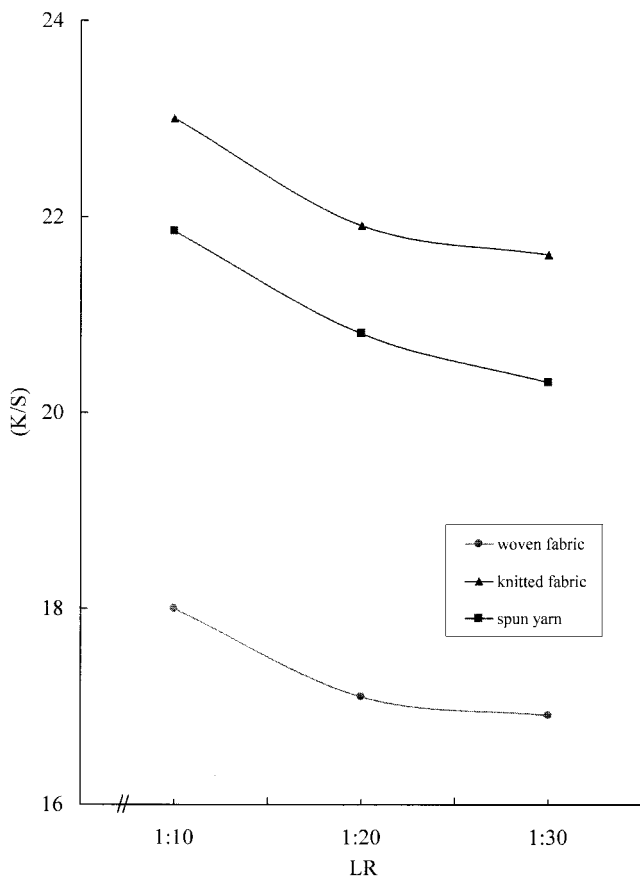
noticeable increase in the  $K/S$  value, whereas further dyeing time, to 60 min, has a negative impact on the dye uptake, regardless of the used substrate.

It is understandable that the proper dyeing time is required for (i) opening up and swelling of the polyester structure, (ii) dissolution and redissolution of the disperse dye, (iii) transferring of the dissolved dye from the bulk of the dye liquor to the fiber surface, (iv) adsorption of the dye at the fiber surface, and, then, (v) diffusion monomolecularly into the fiber structure under the influence of thermal energy.<sup>30,33</sup> The net effect of the aforementioned steps is an enhancement in the extent of dye uptake as well as an improvement in the  $K/S$  values of the dyed polyester substrates. However, the  $K/S$  value is governed by the nature of the polyester substrate.

On the other hand, the decrease in the  $K/S$  values beyond 45 min at 130°C could be ascribed to a partial aggregation of the disperse dye molecules and/or increasing the amount of saponified and dissolved cyclic trimers migrated into the dye bath during dyeing at a longer time, thereby minimizing the extent of dye



**Figure 4** Variation of the  $K/S$  values with the dyeing time. Dyeing bath constituents: Dianix® Navy Blue AD-G (2% ows); Diaserver® AD-95 (2% ows); Eganal® PSN (2 g/L); Dispersogen® P (0.5 g/L); borax (2 g/L); pH 9; LR, 1/10; dyeing temperature, 130°C.



**Figure 5** Variation of the  $K/S$  values with the dyebath LR. Dyeing bath constituents: Dianix® Navy Blue AD-G (2% ows); Diaserver® AD-95 (2% ows); Eganal® PSN (2 g/L); Dispersogen® P (0.5 g/L); borax (2 g/L); pH 9; dyeing at 130°C for 45 min.

penetration and fixation into the compact structure of the polyester polymer, regardless of the used substrate.<sup>16,31</sup>

#### LR

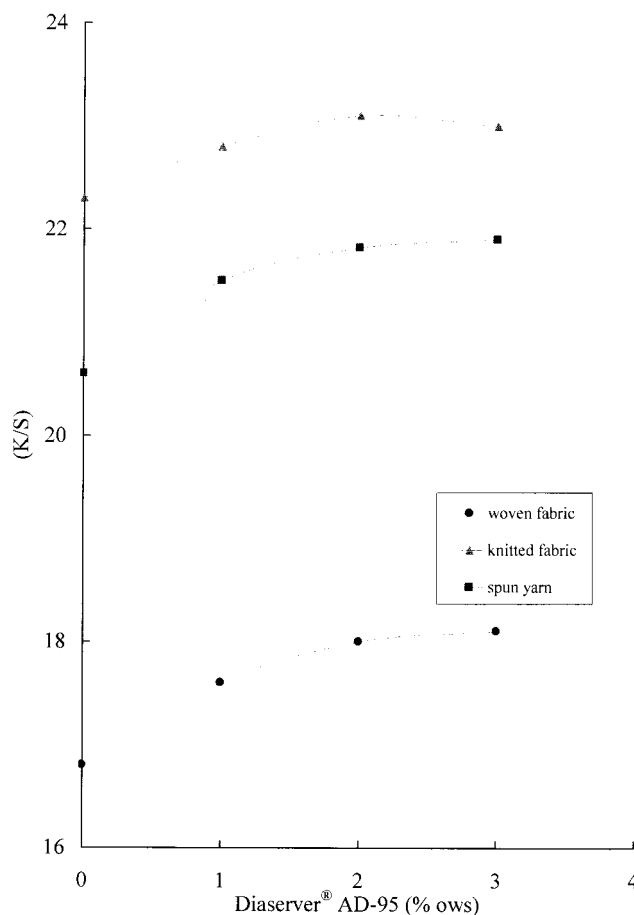
Figure 5 shows the change in the  $K/S$  values as a function of the LR ranging from 1/10 to 1/30 at 130°C for 45 min. It is clear that decreasing the LR to 1/10 is accompanied by a gradual increase in the  $K/S$  value regardless of the used substrate, that is, the lower the LR, the higher is the dye uptake. The enhancement in the  $K/S$  value of the dyed polyester substrates as the LR decreases could be attributed to the greater swellability and accessibility of the polymer structure along with a greater availability of dye molecules and active ingredients in the vicinity of the so-called amorphous regions of the polyester polymer.

#### Dyestuff stabilizer

Figure 6 shows the effect of the Diaserver® AD-95 concentration on the dye uptake of the used polyester

substrates. It is evident that, within the range examined and for given dyeing conditions, increasing of the Diaserver® AD-95 concentration to 2% ows improves the dye uptake, expressed as  $K/S$  values, regardless of the used substrates. Further increase in the concentration, beyond 2% ows, has practically no or only a slight effect on the  $K/S$  values.

This indicates that incorporation of Diaserver® AD-95 (up to 2% ows) in the dyeing formulation seems to perform many functions, such as stabilizing and protecting the disperse dye under alkaline conditions, working up as a sequestering agent for metal ions, as a buffering effect, as well as for the capability of saponifying oligomers and dissolving trimers, thereby enhancing the dye uptake, that is, higher  $K/S$  values.<sup>13,16</sup> On the other hand, leveling off the dye uptake, above 2% ows, can be explained in terms of shortage in and/or inaccessibility of the dyeing sites or may be due to the dispersing action of the used dyestuff stabilizer, especially at higher concentrations, thereby leading to the retardation of dyeing.<sup>13,34</sup>



**Figure 6** Variation of the  $K/S$  values with Diaserver® AD-95 concentration. Dyeing bath constituents: Dianix® Navy Blue AD-G (2% ows); Eganal® PSN (2 g/L); Dispersogen® P (0.5 g/L); borax (2 g/L); pH 9; LR, 1/10; dyeing at 130°C for 45 min.

**TABLE I**  
Effect of Using Different Dye Stabilizers

Stabilizer (2% ows)	None	Diaserver® AD-95	EDTA	Tinoclorite® CBB	Citric acid
K/S	16.75	18.00	18.35	17.93	17.57
	—	(19.14)	(19.63)	(18.76)	(18.24)

Substrate: woven fabric; Dianix® Navy Blue AD-G (2% ows); Eganal® PSN (2 g/L); Dispersogen® P (0.5 g/L); borax (2 g/L); TEOHA (1% ows); pH 9; LR (1/10); dyeing at 130°C for 45 min. The values in parentheses are the *K/S* values of the polyester dyeings in the presence of TEOHA (1% ows) along with other auxiliaries.

Table I shows the effect of the full replacement of Diaserver® AD-95 added to the dyeing formulation as a multifunctional agent, using other common sequestering agents, on the extent of dyeing of the woven fabric samples. It is clear that, for a given set of dyeing conditions, the *K/S* value of the dyed samples follows the descending order

EDTA > Diaserver® AD-95

> Tinoclorite® CBB > citric acid > none

The differences in the *K/S* values, among the used auxiliaries, are probably due to differences in the (i) chemical composition, (ii) functionality, (iii) binding capacity, as well as effectiveness against hardness salts as well as heavy metals under alkaline conditions, (iv) buffering action, (v) compatibility with other dyebath components, (vi) dispersing action, as well as (vii) acting as a dye stabilizer.<sup>13,16</sup>

On the other hand, incorporation of TEOHA (1% ows) along with the aforementioned stabilizers results in a significant improvement in the extent of dyeing regardless of the used stabilizer, which is a direct consequence of the positive impact of TEOHA on modifying the polyester structure as discussed earlier in addition to the synergistic action of TEOHA along with other auxiliaries under investigation. Nevertheless, the *K/S* value is determined by the nature of the dye stabilizer

#### Dyebath pH

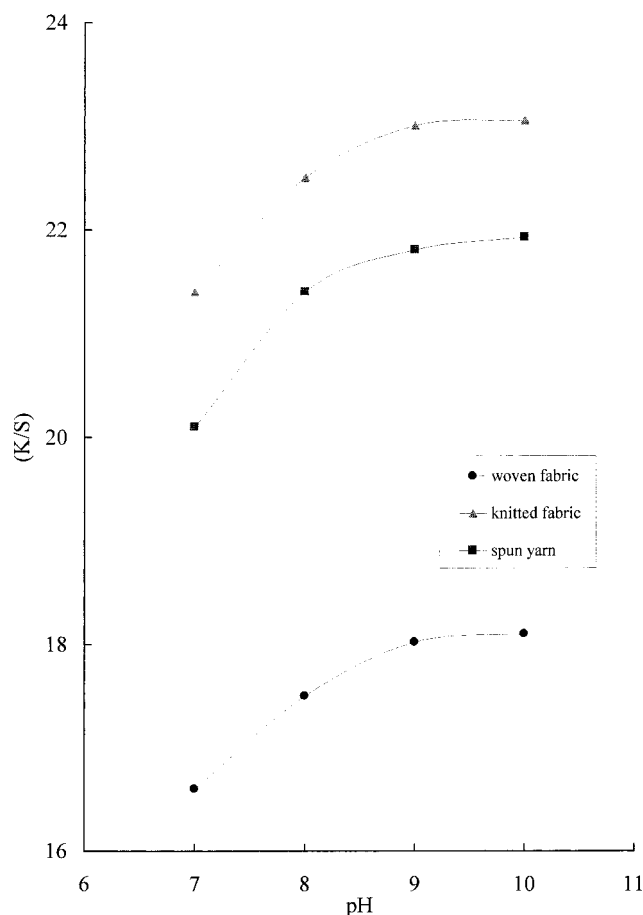
It is evident (Fig. 7) that, within the range examined, increasing the pH to 9 at 130°C for 45 min in the presence of Diaserver® AD-95 (2% ows) along with other additives brings about a noticeable increase in the *K/S* values of the dyed samples, then it levels off with a further increase in pH to 10, reflecting the favorable impact of a proper dyebath pH on (i) loosening the fiber structure, (ii) releasing and dissolution of oligomers, and (iii) enhancing and facilitating easy penetration of dye molecules into the polymer structure, thereby giving rise to higher dye uptake.<sup>13,34</sup>

On the other hand, a further increase in pH, that is, beyond 9, has practically no effect on the *K/S* values. Most probably, dye instability accounts for this, thereby leveling off the extent of dye uptake.<sup>13</sup>

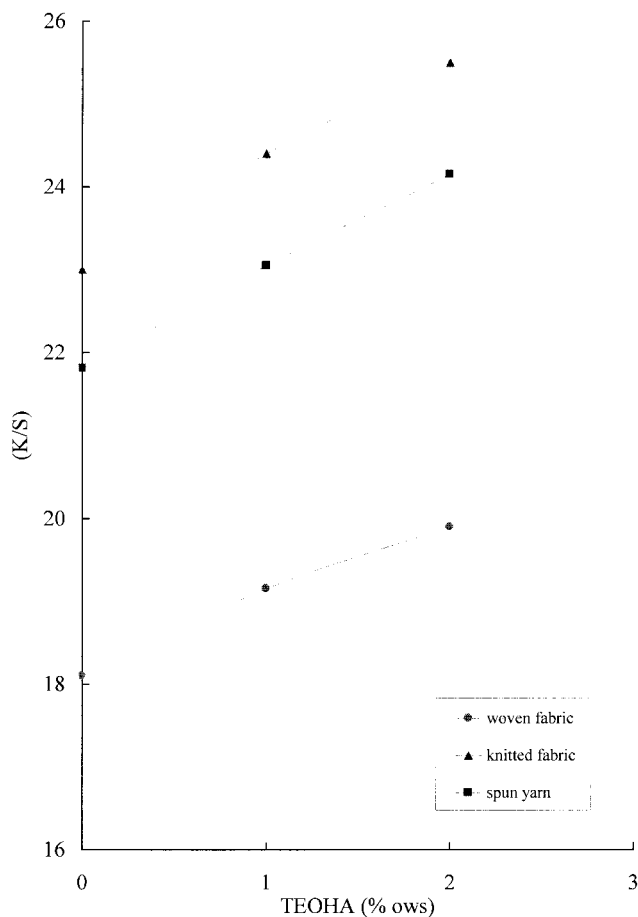
#### Amine concentration

The effect of incorporation of TEOHA in the dyeing bath along with other ingredients on the *K/S* values is shown in Figure 8. It is obvious that increasing the TEOHA concentration to 2% ows brings about a gradual increase in the *K/S* values, regardless of the used substrate.

It is understandable that the addition of TEOHA tends to (i) diffuse into and react with the polyester fiber, (ii) rupture the original intermolecular bonds, and (iii) cause internal changes in the fiber structure, thereby loosening it, thus enhancing the dye uptake.<sup>35,36</sup> Nevertheless, the changes in the *K/S* values are determined by the nature of the polyester substrate.



**Figure 7** Variation of the *K/S* values with the dyebath pH. Dyeing bath constituents: Dianix® Navy Blue AD-G (2% ows); Diaserver® AD-95 (2% ows); Eganal® PSN (2 g/L); Dispersogen® P (0.5 g/L); borax (2 g/L); LR, 1/10; dyeing at 130°C for 45 min.



**Figure 8** Variation of the  $K/S$  values with TEOHA concentration. Dyeing bath constituents: Dianix<sup>®</sup> Navy Blue AD-G (2% ows); Diaserver<sup>®</sup> AD-95 (2% ows); Eganal<sup>®</sup> PSN (2 g/L); Dispersogen<sup>®</sup> P (0.5 g/L); borax (2 g/L); pH 9; LR, 1/10; dyeing at 130°C for 45 min.

#### Heat-setting temperature

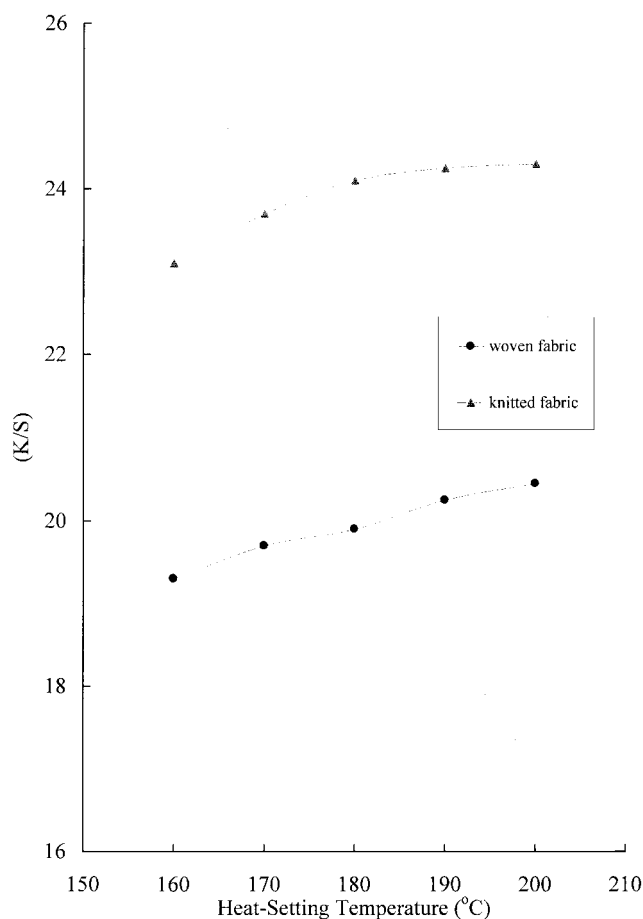
Figure 9 shows the relationship between the Dianix<sup>®</sup> Navy Blue AD-G disperse dye (2% ows) uptake on the woven and knitted polyester fabrics and heat-setting temperature. It is evident that, for a given dyeing condition, increasing the thermosetting temperature from 160 to 200°C for 30 s gives rise to a gradual increase in the dye uptake, regardless of the used substrate, which is a direct consequence of the (i) formation of larger crystals, (ii) reduction in the chain molecules' orientation, as well as (iii) increasing the free volume available to each crystal. The net effect of the aforementioned changes in the polymer structure is a gradual increase in dye uptake with increase in the heat-setting temperature.<sup>28,37</sup>

#### Alkaline weight reduction

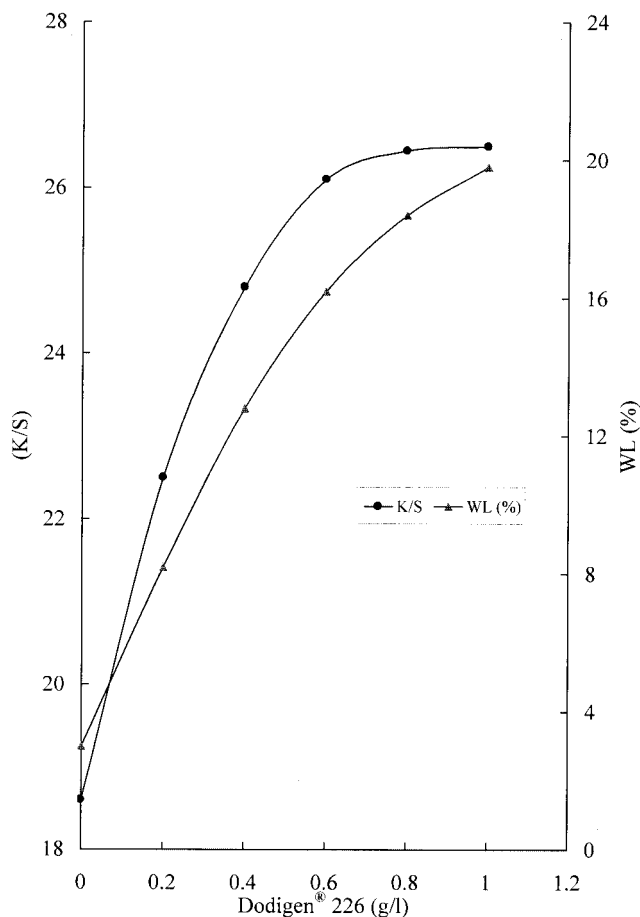
Figure 10 shows the effect of alkaline hydrolysis of polyester woven fabric in the presence and absence of a quaternary ammonium compound (Dodigen<sup>®</sup> 226)

on the weight loss as well as on the postdyeing with the Dianix<sup>®</sup> Navy Blue AD-G disperse dye. It is clear that adding Dodigen<sup>®</sup> 226 (to 1 g/L) to the alkaline bath (NaOH: 10 g/L) is accompanied by a great increase in the weight loss as a direct consequence of (i) facilitating the accessibility of the reactant OH anions to the reactive ester linkages in the fiber substrate as well as (ii) increasing the stoichiometric concentration of the reactant OH anions in the vicinity of the polyester immobile macroradicals, thereby accelerating alkaline hydrolysis, that is, enhancing the extent of attack of amorphous regions followed by chain scission and, finally, a slower attack on both the semicrystalline and crystalline regions.<sup>5,6,25</sup>

On the other hand, the enhancement in dye uptake by increasing the Dodigen<sup>®</sup> 226 concentration (to 0.8 g/L) in the alkaline bath could be interpreted in terms of (i) higher and more rapid dye adsorption, (ii) improving hydrophilicity, (iii) removing of oligomers, (iv) opening up and modifying the fiber structure, as well as (v) enhancing the segmental chain mobility



**Figure 9** Variation of the  $K/S$  values with the heat-setting temperature. Dyeing bath constituents: Dianix<sup>®</sup> Navy Blue AD-G (2% ows); Diaserver<sup>®</sup> AD-95 (2% ows); Eganal<sup>®</sup> PSN (2 g/L); Dispersogen<sup>®</sup> P (0.5 g/L); borax (2 g/L); pH 9; LR, 1/10; dyeing at 130°C for 45 min; heat-setting time, 30 s.



**Figure 10** Variation of the weight-loss and  $K/S$  values with Dodigen® 226 concentration in alkaline weight-reduction bath. Dyeing bath constituents: Dianix® Navy Blue AD-G (2% ows); Diaserver® AD-95 (2% ows); Eganal® PSN (2 g/L); Dispersogen® P (0.5 g/L); borax (2 g/L); pH 9; LR, 1/10; dyeing at 130°C for 45 min.

inside the fiber, thereby speeding the diffusion of the dye into the fabric and increasing its dye uptake.<sup>5,35,36</sup> Further increase in the accelerator concentration, beyond 0.8 g/L, has practically no effect on the dye uptake.

### Color and fastness properties of polyester dyeing

The results (Table II) reveal that (i) the  $K/S$  values are determined by the nature of the substrate, that is, knitted fabric > spun yarn > woven fabric, for given dyeing conditions and irrespective of the used disperse dye, (ii) the dye uptake as well as the change in the fastness properties are governed by the nature of the disperse dye regardless of the used substrate, (iii) the differences in dyeing properties upon using the disperse dyes under investigation could be interpreted in terms of differences among them with respect to molecular weight, chemical structure, energy levels, extent of aggregation and agglomeration, extent of solubility as well as degree of stability, diffusion characteristics, migration power, in addition to compatibility with other ingredients,<sup>27,33,38</sup> and (iv) polyester substrates dyed with Dianix® Yellow AD-G had excellent wash and crock fastness along with a very good light fastness.

On the other hand, Table III shows that incorporation of the aforementioned dye stabilizers in dyeing formulations bring about significant changes in the  $K/S$  values of the polyester dyeings without affecting their fastness properties. This was observed in the presence or in the absence of TEOHA.

### Feasibility of direct reuse of the disperse dyebaths

The feasibility of the direct reuse of the disperse dye-baths, in a single shade and without reconstitution of both the dyestuff and auxiliary chemicals, in the coloration of 100% polyester substrates to accomplish energy/materials savings for economical and ecological reasons is shown in Table IV. The results reveal that (i) the  $K/S$  values of the dyed samples before reusing are far greater than in the first and second runs, regardless of the used dyestuff, (ii) the extent of exhaustion and the change in color yield are governed by the type of substrate and the nature of the disperse dye, (iii) for a given dyeing procedure, direct reuse,

**TABLE II**  
Color and Fastness Properties of Polyester Dyeings

Disperse dye	Substrate	$K/S$	WF (CC)	LF	CF	
					d	w
Dianix® Navy AD-G ( $\lambda_{max}$ : 600 nm)	Spun yarn	21.75	4-5	5-6	5	4-5
	Knitted fabric	23.01	4-5	5-6	5	4-5
	Woven fabric	18.00	4-5	5-6	5	4-5
Dianix® Yellow AD-G ( $\lambda_{max}$ : 420 nm)	Spun yarn	15.26	5	6-7	5	5
	Knitted fabric	16.007	5	7	5	5
	Woven fabric	14.98	5	7	5	5
Dianix® Red AD-2B ( $\lambda_{max}$ : 520 nm)	Spun yarn	14.53	4-5	5-6	5	4-5
	Knitted fabric	15.98	4-5	6	5	4-5
	Woven fabric	14.96	4-5	6	5	4-5

Dye (2% ows); Diaserver® AD-95 (2% ows); Eganal® PSN (2 g/L); Dispersogen® P (0.5 g/L); borax (2 g/L); pH 9; LR (1/10); dyeing at 130°C for 45 min. WF: washing fastness; CC: color change; LF: light fastness; CF: crock fastness; d: dry; w: wet.



**TABLE III**  
Effect of Using Different Dye Stabilizers on Fastness Properties of Dyed Woven Fabrics

Dye stabilizer	K/S	WF (CC)	LF	CF	
				d	w
Diaserver® AD-95	18.00 (19.14)	4-5 (4-5)	5-6 (5-6)	5 (5)	4-5 (4-5)
EDTA	18.35 (19.63)	4-5 (4-5)	5-6 (5-6)	5 (5)	4-5 (4-5)
Citric acid	17.57 (18.24)	4-5 (4-5)	5-6 (5-6)	5 (5)	4-5 (4-5)
Tinoclorite® CBB	17.93 (18.76)	4-5 (4-5)	5-6 (5-6)	5 (5)	4-5 (4-5)

Dianix® Navy Blue AD-G (2% ows); dye stabilizer (2% ows); TEOHA (1% ows); Eganal® PSN (2 g/L); Dispersogen® P (0.5 g/L); borax (2 g/L); pH 9; LR (1/10); dyeing at 130°C for 45 min. The values in parentheses are the color and fastness properties of woven dyeings in the presence of TEOHA (1% ows) along with other auxiliaries. WF: washing fastness; CC: color change; LF: light fastness; CF: crock fastness; d: dry; w: wet.

especially the first one, of single shades was found feasible in the reverse-order dyeings (dark → light), (iv) the *K/S* values in case of the second reuse are negligible, and (v) the colorless and spent dyebaths left after successive reuses could be reused to perform polyester dyeing, save energy, water, as well as auxiliaries, that is, cost reduction, in addition to reduce the mass of pollutants actually discharged.

### One-bath, one-step disperse/reactive dyeing of polyester/cotton blend

With a view to study the technical feasibility of a high-temperature alkaline process for the single-bath, one-step exhaust dyeing of a polyester/cotton blend knitted fabric with disperse/reactive combinations, attempts have been made to modify the dyeing formulation via inclusion of certain dye stabilizers, that is,

Diaserver® AD-95, EDTA, citric acid, and Tinoclorite® CBB, along with TEOHA, in addition to define the proper disperse/reactive combinations.

As far as the changes in the color and fastness properties of polyester/cotton blend dyeings as a function of the type of the disperse dye stabilizer, the presence or absence of TEOHA as an additive, as well as the nature of disperse/reactive dyes combination (Tables V–VII) reveal that

1. The dye uptake, expressed as *K/S* values, is determined by the nature of disperse dye stabilizer and follows the descending order

EDTA > citric acid > Tinoclorite® CBB

> Diaserver® AD-95

most probably due to the differences among the aforementioned stabilizers in their chemical structure, functionality, buffering action, compatibility with other ingredients, binding capacity, in addition to effectiveness against hardness salts and heavy metals under the given dyeing conditions.<sup>13,16</sup>

2. Incorporation of TEOHA (0.5% ows) along with the aforementioned stabilizers brings about a significant improvement in the *K/S* values, regardless of the dye stabilizer used as well as the type of the disperse/reactive dyes combination, reflecting its positive impact on modifying both the polyester and cotton components, enhancing swellability and wettability of the used substrate, improving the extent of the diffusion and penetration of the dyes within the fabric structure, as well as accelerating and increasing the extent of the reactive dye–cellulosic fiber interaction according to the following scheme of reactions<sup>35,36,39–41</sup>:

**TABLE IV**  
Feasibility of Reusing the Dyebath Without Reconstitution

Polyester substrate	Disperse dye	<i>K/S</i>	<i>K/S</i> <sub>1</sub>	<i>K/S</i> <sub>2</sub>
Woven fabric	Dianix® Yellow AD-G ( $\lambda_{\max}$ : 420 nm)	14.977	0.269	0.011
	Dianix® Orange AD-R ( $\lambda_{\max}$ : 440 nm)	12.206	0.366	0.014
	Dianix® Red AD-2B ( $\lambda_{\max}$ : 520 nm)	14.960	0.371	0.015
	Dianix® Rubbin AD-B ( $\lambda_{\max}$ : 540 nm)	16.062	0.503	0.020
	Dianix® Blue AD-R ( $\lambda_{\max}$ : 600 nm)	21.162	0.366	0.014
	Dianix® Navy Blue AD-G ( $\lambda_{\max}$ : 600 nm)	18.001	0.809	0.032
Knitted fabric	Dianix® Yellow AD-G	16.007	0.295	0.012
	Dianix® Red AD-2B	15.980	0.430	0.017
	Dianix® Navy Blue AD-G	23.010	1.038	0.041
Spun yarn	Dianix® Yellow AD-G	15.263	0.148	0.006
	Dianix® Red AD-2B	14.530	0.437	0.017
	Dianix® Navy Blue AD-G	21.750	0.773	0.031

Initial dyebath components: disperse dye (2% ows); Diaserver® AD-95 (2% ows); Eganal® PSN (2 g/L); Dispersogen® P (0.5 g/L); borax (2 g/L); pH 9; LR (1/10); dyeing at 130°C for 45 min. *K/S*: color strength (before reusing); *K/S*<sub>1</sub>: color strength (first run); *K/S*<sub>2</sub>: color strength (second run). Reusing of the dyebath was carried out without reconstitution.

**TABLE V**  
**Color and Fastness Properties of Dyed Samples Using Dianix® Blue AD-R/Procion® Sapphire HEXL Combination**

Dye stabilizer	K/S*		Increase in K/S (%)	WF (CC)	CF		LF
	Without	With			d	W	
Diaserver® AD-95	6.72	8.25	22.77	4-5	5	4-5	6
EDTA	10.63	12.14	14.20	4-5	5	4-5	6
Citric acid	10.30	11.79	14.47	4-5	5	4-5	6
Tinoclorite® CBB	10.09	11.60	14.96	4-5	5	4-5	6

Disperse dye/reactive dye combination (2% ows); dye stabilizer (2% ows); K/S\*: color intensity of dyed samples; without: in absence of TEOHA; with: in presence of TEOHA (0.5% ows). WF: washing fastness; CC: color change; CF: crock fastness; d: dry; w: wet; LF: light fastness. Fastness properties: for samples dyed in presence of TEOHA (0.5% ows). Dyeing conditions: see Figure 2.



- The K/S values of the resultant dyeings, the percent increase in the K/S values, as well as the fastness properties of these dyeings are determined by the nature of disperse/reactive combination, that is, the molecular weight, chemical structure, extent of aggregation and agglomeration, extent of solubility, diffusion and penetration, degree of stability, energy level of the disperse dye, dye affinity, migration power, in addition to compatibility with each other as well as with other ingredients in dyeing formulations.<sup>27,33,38,42,43</sup>
- Generally speaking, the fastness properties' ratings of the dyed samples, in the presence of TEOHA (0.5 ows), are rated quite highly, especially in the case of using Dianix® Blue AD-R/Procion® Sapphire HEXL and Dianix® Yellow AD-G/Procion® Flavine HEXL combinations.
- Fabrics dyed with a Dianix® Red AD-2B/Procion® Red HE3B combination resulted in a slightly lower wash and light fastness properties ratings.

- This dyeing procedure also produces level polyester/cotton dyeings.

## CONCLUSIONS

In this study, trials have been carried out to

- Study factors governing the dyeability of polyester-based textiles with disperse dyes in an alkaline medium,
- Evaluate the feasibility of the reuse of the dye-baths, as well as
- Demonstrate the technical feasibility of dyeing polyester/cotton blend with disperse/reactive dyes at high-temperature alkaline conditions.

The results obtained led to the following conclusions:

- For a given set of dyeing conditions, dyeing at 130°C for 45 min constitutes the optimal condi-

**TABLE VI**  
**Color and Fastness Properties of Dyed Samples Using Dianix® Yellow AD-G/Procion® Flavine HEXL Combination**

Dye stabilizer	K/S*		Increase in K/S (%)	WF (CC)	CF		LF
	Without	With			d	w	
Diaserver® AD-95	3.46	5.01	44.80	4-5	5	4-5	6
EDTA	5.05	5.57	10.30	4-5	5	4-5	6
Citric acid	4.84	6.38	31.82	4-5	5	4-5	6
Tinoclorite® CBB	4.71	6.21	31.85	4-5	5	4-5	6

Disperse dye/reactive dye combination (2% ows); dye-stabilizer (2% ows); K/S\*: color intensity of dyed samples; without: in absence of TEOHA; with: in presence of TEOHA (0.5% ows). WF: washing fastness; CC: color change; CF: crock fastness; d: dry; w: wet; LF: light fastness. Fastness properties: for samples dyed in presence of TEOHA (0.5% ows). Dyeing conditions: see Figure 2.

TABLE VII  
Color and Fastness Properties of Dyed Samples Using Dianix® Red AD-2B/Procion® Red HE3B Combination

Dye-stabilizer	K/S*		Increase in K/S (%)	WF (CC)	CF		LF
	Without	With			d	w	
Diaserver® AD-95	6.44	7.98	23.91	4	5	4-5	5-6
EDTA	9.45	10.99	16.30	4	5	4-5	5-6
Citric acid	9.01	10.55	17.09	4	5	4-5	5-6
Tinoclorite® CBB	8.85	10.37	17.18	4	5	4-5	5-6

Disperse dye/reactive dye combination (2% ows); dye stabilizer (2% ows); K/S\*: color intensity of dyed samples; without: in absence of TEOHA; with: in presence of TEOHA (0.5% ows). WF: washing fastness; CC: color change; CF: crock fastness; d: dry; w: wet; LF: light fastness. Fastness properties: for samples dyed in presence of TEOHA (0.5% ows). Dyeing conditions: see Figure 2.

tions for attaining higher K/S values, regardless of the used substrate.

- Lowering the LR, incorporation of Diaserver® AD-95 to 2%, and/or inclusion of TEOHA to 2% along with other ingredients enhance the dye uptake with a subsequent improvement in the K/S values.
- Increasing the thermosetting temperature to 200°C for 30 s or an alkaline weight reduction has a positive impact on the K/S values.
- The extent of dye uptake is governed by both the type of substrate as well as the nature of the dye stabilizer.
- Reuse of disperse dyebaths, without reconstitution and in single shades, was investigated in dyeing different polyester substrates and was found to be feasible in the reverse (dark → light) direction.
- Proper disperse/reactive dyes combinations as well as dyeing formulations that are suitable for a single-bath/one-step exhaust dyeing of a polyester/cotton blend at high-temperature alkaline conditions are given without adversely affecting the levelness.

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