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Dyes and Pigments 66 (2005) 231–239

DYES and PIGMENTS
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A study of heterobifunctional reactive dyes on nylon fibers: dyeing properties, dye moiety analysis and wash fastness

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Received 13 May 2004; received in revised form 22 August 2004; accepted 7 October 2004

Abstract

Heterobifunctional reactive dyes provide great opportunity for efficient dye–fiber reaction due to the mixed double-anchors such as sulphatoethylsulphone and monochlorotriazinyl reactive groups, which may cover a wide range of application temperatures. Four heterobifunctional reactive dyes were applied to nylon fibers using various pH and temperature conditions. The optimized exhaustion (%E) and fixation (%F) were determined. Analysis of dye solutions being adjusted to different pH conditions was carried out to verify the structure of dye moieties. In addition, the acid dyeings having the similar color strength with reactive dyeings were prepared using color matching system to compare wash fastness properties of both the dyed samples. In the case of reactive dyeings, when compared to acid dyeings, the extent of reduction in color strength that occurred during repeated washings was very low and the shade of the dyeings was little changed.

Keywords: Heterobifunctional; Reactive dye; Nylon; LC/MS; Aftertreatment; Wash fastness

1. Introduction

In general, acid dyes have attracted much attention to nylon substrates due to their interaction mechanism and an easy method for application [1–4]. However, to achieve satisfactory levels of wash fastness, recourse is commonly required to an aftertreatment with a commercial syntan (synthetic tanning agent) and other fixing systems. While an aftertreatment of the dyed nylon substrates can improve wash fastness, not only this treatment can impart a change in shade of the ground color but also it is temporary in nature [2,5,6].

In this point of view, application of reactive dyes [7,8] to nylon substrates has attracted interests to solve those problems. Reactive dyes react chemically with amino groups within nylon fibers to form a covalent bond. Theoretically, by virtue of the covalent nature of the dye–fiber bond, reactive dyeings on nylon fibers can display excellent wash fastness without any recourse to an aftertreatment. Especially, reactive dyes containing heterobifunctional groups can provide great opportunity for efficient dye–fiber reaction due to the sulphatoethylsulphone and monochlorotriazinyl reactive systems, which may cover a wide range of application temperatures.

Thus, this paper concerns the build-up characteristics and wash fastness properties of the mixed anchor reactive dyes, namely heterobifunctional reactive dyes [9–11]. Exhaustion (%E), fixation (%F) and fixation efficiency (%FE) were examined in terms of various pH conditions and dyeing temperatures. In addition, LC/MS analysis of dye moieties in the dye solution using

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doi:10.1016/j.dyepig.2004.10.004

different pH conditions was investigated to determine the dye structures occurring from hydrolysis reaction. Finally, the level of fastness obtained by reactive dyeings on nylon fibers to extended washings was compared to acid dyeings after treated with syntan and syntan/fixing agent systems.

2. Experimental

2.1. Materials

Nylon fabric (warp 70F24, weft 140F48) was used in this experiment. C. I. Reactive Red 198, C. I. Reactive Red 176, C. I. Reactive Red 241 and C. I. Reactive Blue CL were used as heterobifunctional reactive dyes. All other chemicals used were laboratory grade reagents.

2.2. Dyeing

Nylon fabric (2 g) was dyed with 2% owf reactive dyes in sealed, stainless steel dye pots of 120 cm^3 capacity in a laboratory-scale dyeing machine (*ACE-6000T*). Samples were placed in a 40 °C dyebath of liquor ratio 30:1. After 10 min, the temperature was raised until reaching 100 °C. At this temperature, the reactive dyeing was continued for 60 min. At the end of dyeing, the dyed samples were removed, rinsed thoroughly in tap water and allowed in the open air.

2.2. Determination of exhaustion (%*E*), fixation (%*F*) and fixation efficiency (%*FE*)

To determine dyeing characteristics, absorbance measurements of the original dyebath and the exhausted dyebath were carried out using a UV/vis spectrophotometer. Using a previously established absorbance/concentration relationship at the λ_{max} of the dyes, the quantity of reactive dye in solution was calculated and the extent of exhaustion (%E) achieved was determined using Eq. (1), where D_0 and D_t are the quantities of dye initially in the bath and of residual dye in the final bath, respectively

$$\%E = \frac{(D_0 - D_t)}{D_0} \times 100 \tag{1}$$

Unfixed dye from the fabrics was extracted using 25% aqueous pyridine solution and then measured spectrophotometrically. The extent of fixation (%F) and fixation efficiency (%FE) was calculated using Eqs. (2) and (3), respectively, where D_e is the amount of extracted dye

$$\%F = \frac{(D_0 - D_t - D_e)}{(D_0 - D_t)} \times 100 \tag{2}$$

$$\%\mathrm{FE} = \frac{(E \times F)}{100} \tag{3}$$

2.3. Preparation of the acid dyeings using color matching system

The acid dyeings having the similar color strength with reactive dyeings were prepared using color matching system (*Datacolor SF 600 plus*) to compare the wash fastness properties of the both dyed samples. These acid dyeings were prepared with arbitrarily chosen dyes from the database of the color matching instrument.

2.4. LC/MS analysis

The LC/MS (*Hewlett Packard, series 1100*) instrument was used to analyze the changes of dye moieties in the dyebath [12]. Dyeings with C. I. Reactive Red 198 were carried out at alkaline conditions such as pH 5.5 and pH 12 and the dye residues in the bath then analyzed. HP Eclipse® XDB-C18 (4.6 × 150 mm, 3.5 μm) column was employed using a mixed solution (water and isopropyl alcohol, 10:90) as a mobile phase. The flow rate was 0.5 ml/min.

2.5. Aftertreatment

Samples which had been dyed using the acid dyes were aftertreated [5,6] with syntan (2% owf *MATEXIL FA-AN 25*, ICI surfactant) employing a liquor ratio 20:1. The syntan solution was adjusted to a pH of 3.5 by acetic acid. The temperature was raised to 80 °C and sustained for 30 min. The dyed and syntanned nylon fibers were consequently placed with cationic fixing agent using a liquor ratio 20:1. Samples were treated with solution of cationic fixing agent (2% owf *Lanasan® FW Liquid*, Clariant). The treatment was carried out for 30 min at 40 °C. A pH of 10 was adjusted using Na_2CO_3 after 10 min at treatment. At the end of treatment, the treated samples were rinsed in tap water and allowed to dry.

2.6. Color measurement

Colorimetric data of dyeings were determined using a *Datacolor SF 600 plus* spectrophotometer interfaced to a PC. Measurements were taken with the specular component of the light excluded and the UV component included, using illuminant D_{65} and 10° standard observer.

2.7. Color fastness

The wash fastness of the dyeing was tested using method of ISO 105 C06 B1S [13].

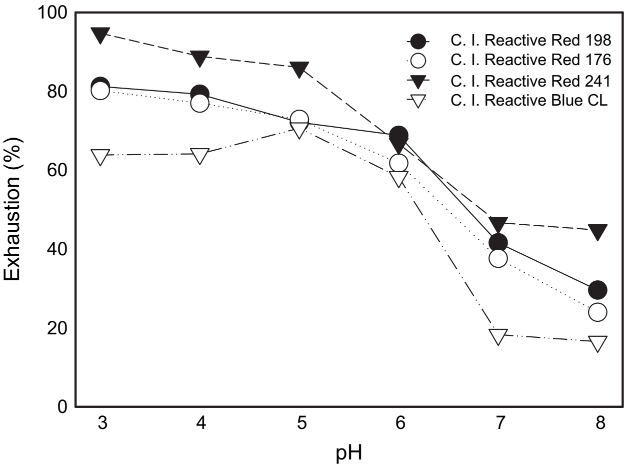


Fig. 1. Effect of application pH on exhaustion (%E).

3. Results and discussion

3.1. Effect of pH on exhaustion (%E), fixation (%F) and fixation efficiency (%FE)

The results in Figs. 1 and 2 show that exhaustion (%E) of the dyes on nylon fibers increased with decreasing pH of application and that fixation (%F) of the dyes on nylon fibers increased with increasing pH of application. Fig. 3 also displays that the highest total fixation efficiency (%FE) was achieved at pH 5.5. At lower pH values, higher extent of protonation of the amino groups within nylon fibers leads to high exhaustion and low fixation, because the ensuing low concentrations of neucleophilic amino groups in the substrates are unable to react with the dye. In addition, at low pH, the vinylsulphone form of the reactive dye is prevalent only at low concentration in the dyebath. Hence, the majority of dye–fiber interaction can be assumed to have occurred between protonated amino groups and the sulphatoethlysulphone form of the dye as in dyeing with acid dye. In contrast, at higher pH values, initial dye exhaustion is low in the absence of amino group protonation, although any dye that does exhaust undergoes reaction to give the high fixation levels seen.

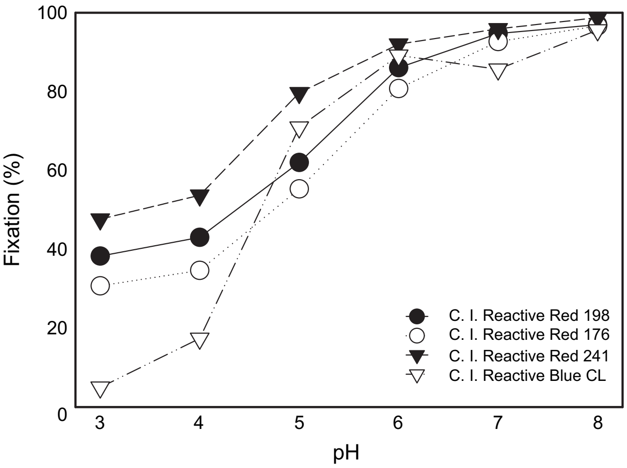


Fig. 2. Effect of application pH on fixation (%F).

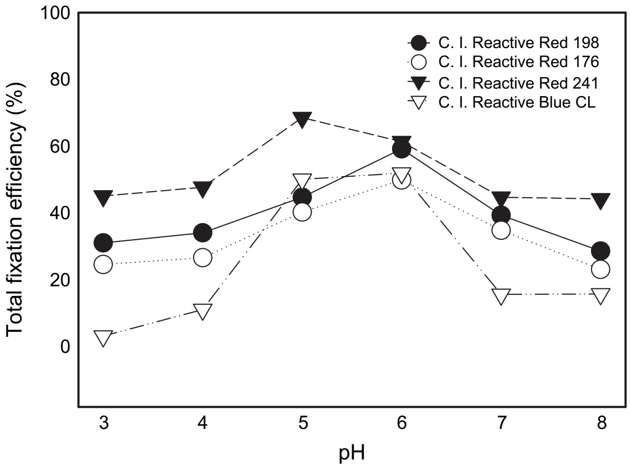


Fig. 3. Effect of application pH on fixation efficiency (%FE).

3.2. Effect of temperature

The exhaustion (%E) and fixation (%F) data obtained when different dyeing temperatures used are shown in Figs. 4 and 5. Since optimum dye exhaustion and dye fixation were achieved previously at pH 5.5 which is necessary to achieve adequate substantivity of the anionic dyes for the protonated amino end groups in the fibers, the effect of dyeing temperature was studied at this pH value. In the case of reactive dyes, due to low migrating power, careful control of dye exhaustion by means of temperature is demanded.

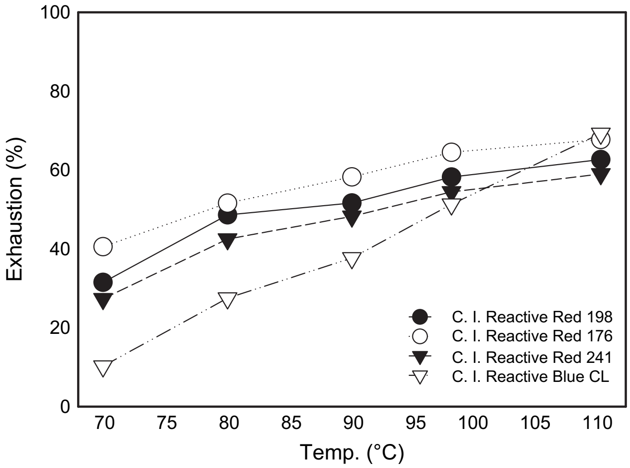


Fig. 4. Effect of application temperature on exhaustion (%E).

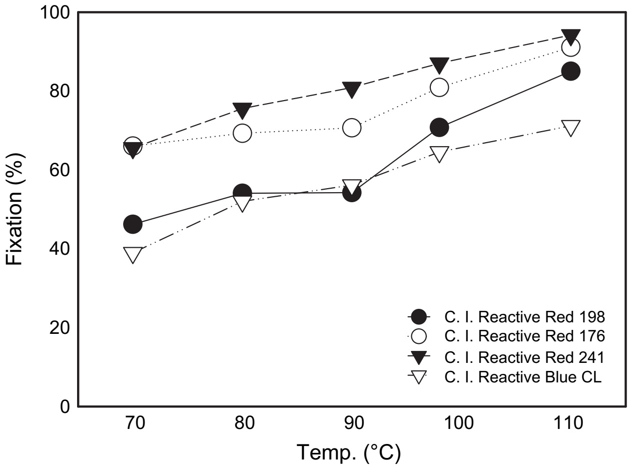


Fig. 5. Effect of application temperature on fixation (%*F*).

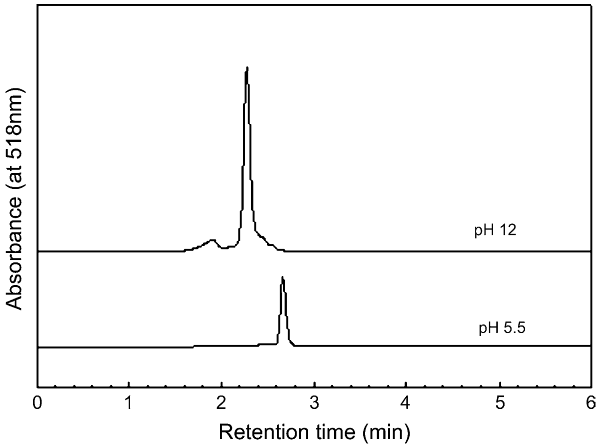


Fig. 6. HPLC analysis of C. I. Reactive Red 198 at pH 5.5 and pH 12.

Figs. 4 and 5 show that higher dye exhaustion and fixation were obtained in the range of temperatures of 100–110 °C. This efficiency can be attributed to the higher kinetic energy of the dye molecules and their consequent greater migration power within the substrates. In addition, a higher extent of fiber swelling will have contributed to increased dye exhaustion. As maximum fixation efficiency was obtained around 100 °C, this temperature was used in subsequent experiments.

3.3. *LC/MS analysis*

The previous results from Figs. 2 and 5 show that alkaline pH conditions and higher temperatures result in higher dye fixation due to high concentrations of neucleophilic amino groups. However, alkaline conditions may cause to dye hydrolysis during the dyeing progress. Especially, strong alkaline condition may expedite this undesired reaction. In this context, to confirm the structure of dye moieties, the determined optimal pH condition of 5.5 and higher pH value of 12 were used. Fig. 6 shows HPLC analysis that two marked peaks were observed at RT 2.66 min (pH 5.5) and at RT 2.27 min (pH 12). And then these separated components were subsequently injected to the mass analyzer.

The mass spectra of the dye moieties were shown in Figs. 7 and 8. The findings from the mass spectra were resulted from the two peaks of HPLC separation. In Figs. 7 and 8, the *m/z* 840 was predominantly detected. HPLC analysis showed different retention time of the dye moiety at pH 5 and pH 12, which represents the presence of different dye structure in the each dyebath. However, mass spectra showed the same value of the *m/z* 840. Herein, it is considered that the contribution of isotopes showing other mass spectra may play a decisive part to determine the exact dye structures.

In the case of C. I. Reactive Red 198 having monochlorotriazine group and sulphatoethylsulphone group, these two reactive groups can be hydrolyzed during dyeing process. Especially, sulphatoethylsulphone reactive group can be converted into vinylsulphone form or completely hydrolyzed form in the alkaline condition. Many dye structures can be considered from the diverse formation of hydrolyzed reactive groups. Possible two structures may be considered, which show exact *m/z* 840. Fig. 9 shows dye structures that in the case of structure 1, sulphatoethylsulphone group was converted to vinylsulphone form and monochlorotriazine reactive group still remained and that the hydrolyzed form of both sulphatoethylsulphone and monochlorotriazine groups was observed from the dye structure 2.

These two structures show the same mass of *m/z* 863, which was detected with *m/z* 840 (863–23, sodium atom) in LC/MS negative mode. To confirm the dye structure, a consideration of the contribution of isotopes

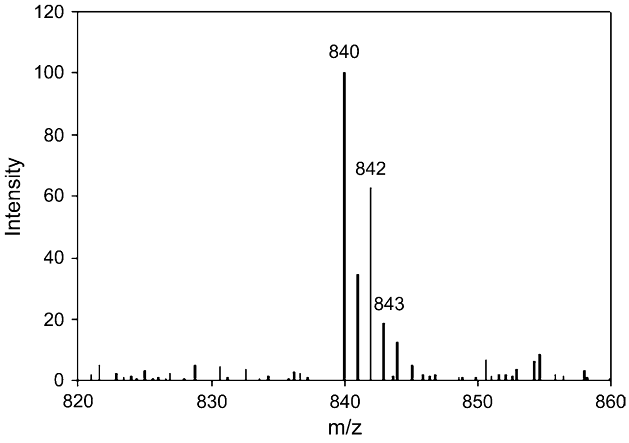


Fig. 7. Mass analysis of C. I. Reactive Red 198 at pH 5.5 (RT 2.66 min).

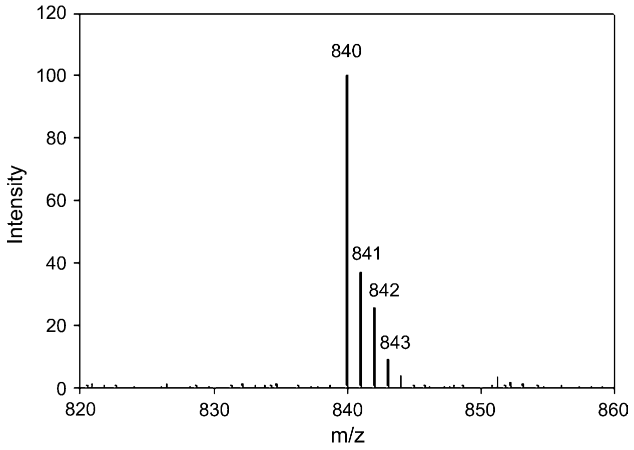
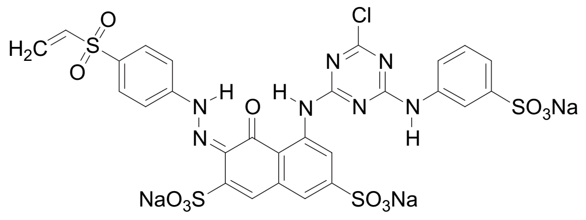


Fig. 8. Mass analysis of C. I. Reactive Red 198 at pH 12 (RT 2.27 min).

in the molecular structure is of importance. Dye structure 1 is influenced by chlorine atom but little effect of chlorine atom was considered to dye structure 2. The proportion of chlorine atoms such as ^{35}Cl and ^{37}Cl in nature corresponds to the ratio of 100% to 32%, which influences the determination of relative mass intensity. Theoretically calculated and measured relative peak intensities are shown in Table 1. It can be concluded from Table 1 and Figs. 7 and 8 that the dye moiety (RT 2.66, Fig. 7) in the dyebath of pH 5.5 represents dye structure 1 and the dye moiety (RT 2.27, Fig. 8) in the dyebath of pH 12 corresponds to dye structure 2.

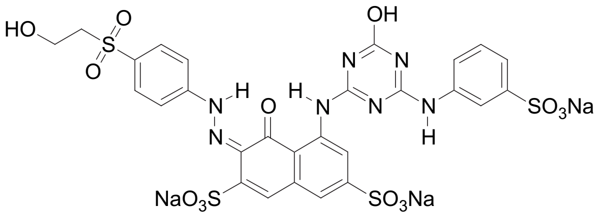
Table 1
Mass data considering isotopic contributions

Peak	*m*/*z*	Relative peak intensity (%)			
		Structure 1		Structure 2	
		Calculated	Measured	Calculated	Measured
$[M-Na]^-$	840	100.0	100.0	100.0	100.0
$[M-Na+1]^-$	841	36.4	35.7	36.6	37.0
$[M-Na+2]^-$	842	58.5	60.9	27.0	25.9
$[M-Na+3]^-$	843	19.6	18.9	8.1	8.9
$[M-Na+4]^-$	844	11.5	12.2	3.1	3.5



$C_{27}H_{17}ClN_7Na_3O_{12}S_4$
Exact Mass: 862.92

Structure **1**



$C_{27}H_{20}N_7Na_3O_{14}S_4$
Exact Mass: 862.96

Structure **2**

Fig. 9. The moieties of C. I. Reactive Red 198 having molecular mass 863.

3.4. Comparison of wash fastness of reactive dyes and acid dyes

Theoretically, reactive dyeings on nylon fibers should, by virtue of the covalent nature of the dye–fiber bond, display excellent fastness to washing without recourse to an aftertreatment. In this context, the aim of this experiment was to determine the level of fastness displayed by reactive dyeings on nylon substrates to extended washings and to compare this to that achieved using nylon fibers which had been dyed with acid dyes and aftertreated with a commercial fixing system. The acid dyeings having the similar color strength with reactive dyeings were prepared using color matching system. Table 2 displays the colorimetric data of both the dyeings using reactive dyes and acid dyes.

It is proposed that when compared to the colorimetric data of both samples, virtually similar color strength values (*fk*) using color matching system were obtained by the dyeings of acid dyes. Thus, these samples are appropriate for the comparison of wash tests. Figs 10–13 show the reduction in color strength ($\Delta fk\%$) that occurred when both reactive and acid dyeings were subjected to five repeated wash tests.

Table 2
Colorimetric data of reactive dyeings and color matched acid dyeings

	L^*	a^*	b^*	C	$h°$	*fk*
C. I. Reactive Red 198	41.53	64.9	6.87	65.3	6.04	173.7
Acid dye 1	42.44	66.85	5.90	67.11	5.05	168.6
C. I. Reactive Red 176	76.20	29.95	90.55	95.37	71.70	175.6
Acid dye 2	75.76	28.99	88.95	93.56	71.95	180.5
C. I. Reactive Red 241	44.23	70.34	2.23	70.37	1.82	150.2
Acid dye 3	43.67	68.38	3.07	68.45	2.57	145.8
C. I. Reactive Blue CL	23.80	−0.20	−13.55	13.55	269.17	312.7
Acid dye 4	24.09	−1.58	−13.47	13.56	263.32	318.7

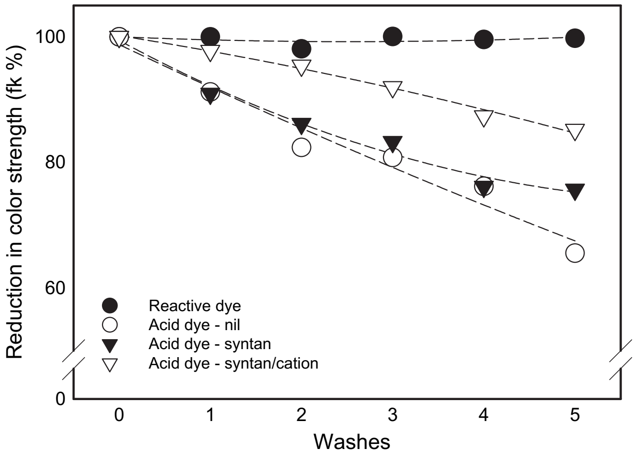


Fig. 10. Comparison of color loss of reactive dyeing (C. I. Reactive Red 198) and acid dyeing.

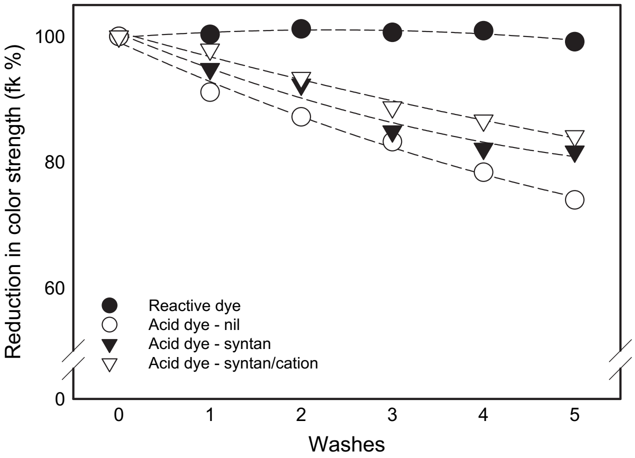


Fig. 12. Comparison of color loss of reactive dyeing (C. I. Reactive Red 241) and acid dyeing.

It is evident that the color strength of the acid dyeings decreased with increasing number of washes and that dye desorption from the dyed samples occurred progressively as the number of washes increased. The results displayed in Figs. 10–13 also reveal that aftertreatment with the syntan and the syntan/cation fixing system reduced both the rate and extent of dye loss that occurred as a result of extended washings. This observed improvement in the wash fastness can be attributed to the presence of a large molecular size complex formed by syntan and syntan/cation fixing system, situated at the periphery of the dyed substrates. In addition, Figs. 10–13 show the reduction in color strength ($\Delta fk\%$) of reactive dyeings. It is evident that for each of the four reactive dyes, the extent of dye loss that occurred during repeated washings was very low and the shade of the dyeings was little changed. The considerable difference in wash-down observed between the reactive dyeings and the acid dyeings can be attributed to the difference in the nature of dye–fiber interaction, namely covalent, in the case of the reactive dyes and non-covalent in the case of the acid dyes. The corresponding assessments of the extent of staining to the adjacent multifiber strip after five wash tests (Tables 3–6) support the findings displayed in Figs. 10–13.

Tables 3–6 show that the reactive dyeings displayed very good fastness properties to the five multiple washes in terms of shade change and that very little staining to the adjacent multifiber strip occurred. This result can be also attributed to the nature of the covalent bond characteristics.

4. Conclusions

Reactive dyeing was investigated to achieve higher wash fastness properties. While heterobifunctional

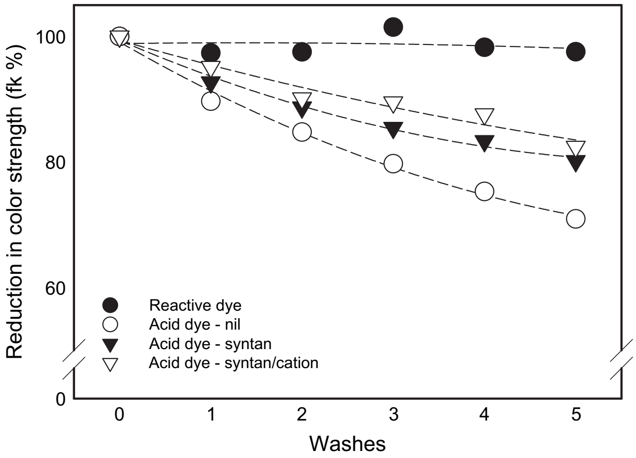


Fig. 11. Comparison of color loss of reactive dyeing (C. I. Reactive Red 176) and acid dyeing.

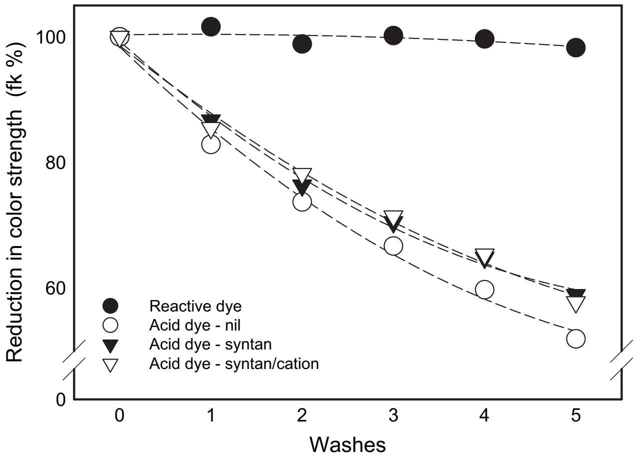


Fig. 13. Comparison of color loss of reactive dyeing (C. I. Reactive Blue CL) and acid dyeing.

Table 3
Grey scale assessments for reactive dyeing (C. I. Reactive Red 198) and acid dyeing

	Washes	Change in color	Staining					
			Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
C. I. Reactive Red 198	1	–	5	5	5	5	5	4/5
	2	–	5	5	5	5	5	4/5
	3	–	4/5	5	4/5	5	5	4/5
	4	–	5	5	5	5	5	5
	5	5	5	5	5	5	5	5
Acid dye (nil)	1	–	4	4	3/4	5	5	3/4
	2	–	4/5	4/5	4	5	5	4
	3	–	4/5	4/5	4/5	5	5	4
	4	–	4/5	5	4/5	5	5	4
	5	3/4	5	5	4/5	5	5	4
Acid dye (syntan)	1	–	4/5	4/5	4	5	5	4
	2	–	5	4/5	4/5	5	5	4/5
	3	–	5	4/5	4/5	5	5	4/5
	4	–	5	5	4/5	5	5	4/5
	5	4	5	5	4/5	5	5	4/5
Acid dye (syntan/cation)	1	–	5	4/5	4/5	5	5	4/5
	2	–	5	4/5	4/5	5	5	4/5
	3	–	5	4/5	4/5	5	5	5
	4	–	5	5	4/5	5	5	5
	5	4/5	5	5	4/5	5	5	5

reactive dyes provide the opportunity for efficient dye–fiber reaction, it is determined that exhaustion (%E) of the dye on nylon fibers increased with decreasing application pH and that fixation (%F) of the dye on nylon fibers increased with increasing application pH. The findings on the effect of temperature show that higher dye exhaustion and fixation were obtained at the ranges of 100–110 °C.

In the case of C. I. Reactive Red 198, HPLC analysis shows that two marked peaks were observed at RT 2.66 min (pH 5.5) and RT 2.27 min (pH 12). It can be concluded that the dye moiety (RT 2.66 min) in the dyebath of pH 5.5 represents dye structure 1 and the dye moiety (RT 2.27 min) in the dyebath of pH 12 corresponds to dye structure 2. In the case of structure 1, sulphatoethylsulphone group was converted to

Table 4
Grey scale assessments for reactive dyeing (C. I. Reactive Red 176) and acid dyeing

	Washes	Change in color	Staining					
			Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
C. I. Reactive Red 176	1	–	5	5	4/5	5	5	4/5
	2	–	5	5	4/5	5	5	4/5
	3	–	5	5	5	5	5	4/5
	4	–	5	5	5	5	5	4/5
	5	5	5	5	5	5	5	4/5
Acid dye (nil)	1	–	2	4/5	2	4/5	5	
	2	–	3	5	2	4/5	5	4
	3	–	3/4	5	2/3	4/5	5	4
	4	–	4	5	3	5	5	4
	5	4	4	5	3	5	5	4
Acid dye (syntan)	1	–	3	4/5	2	4/5	5	4
	2	–	3/4	5	2/3	5	5	4
	3	–	4	5	2/3	5	5	4
	4	–	4	5	3	5	5	4
	5	4/5	4	5	3	5	5	4
Acid dye (syntan/cation)	1	–	3/4	4/5	2/3	5	5	4
	2	–	4	5	2/3	5	5	4
	3	–	4	5	3	5	5	4
	4	–	4	5	3	5	5	4/5
	5	4/5	4/5	5	3/4	5	5	4/5

Table 5
Grey scale assessments for reactive dyeing (C. I. Reactive Red 241) and acid dyeing

	Washes	Change in color	Staining					
			Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
C. I. Reactive Red 241	1	–	5	5	5	5	5	4/5
	2	–	5	5	4/5	5	5	4/5
	3	–	5	5	5	5	5	4/5
	4	–	5	5	5	5	5	4/5
	5	5	5	5	5	5	5	4/5
Acid dye (nil)	1	–	5	4	3	5	5	3/4
	2	–	5	4/5	3	5	5	4
	3	–	5	4/5	3/4	5	5	4
	4	–	5	4/5	4	5	5	4
	5	4	5	4/5	4/5	5	5	4
Acid dye (syntan)	1	–	5	4/5	4	5	5	4/5
	2	–	5	4/5	4	5	5	4
	3	–	5	5	4/5	5	5	4
	4	–	5	5	4/5	5	5	4
	5	4/5	5	4/5	4/5	5	5	4
Acid dye (syntan/cation)	1	–	5	5	4	5	5	4/5
	2	–	5	5	4	5	5	4
	3	–	5	5	4/5	5	5	4
	4	–	5	5	4/5	5	5	4/5
	5	4/5	5	5	4/5	5	5	4/5

vinylsulphone form and monochlorotriazine reactive group still remained and that the hydrolyzed form of both sulphatoethylsulphone and monochlorotriazine groups were determined from the dye structure 2.

It is evident that for each of the four reactive dyes, the extent of dye loss that occurred during repeated washing was very low and the shade of the dyeings was little changed. The considerable difference in wash-down observed between the acid dyeings and the reactive dyeings can be attributed to the difference in the nature of dye–fiber interaction, namely covalent. The reactive dyeings displayed very good fastness properties to the five multiple washes in terms of shade change and very little staining to the adjacent multifiber strip occurred.

Table 6
Grey scale assessments for reactive dyeing (C. I. Reactive Blue CL) and acid dyeing

	Washes	Change in color	Staining					
			Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
C. I. Reactive Blue CL	1	–	5	4/5	5	5	5	4/5
	2	–	5	4/5	5	5	5	4
	3	–	5	4/5	5	5	5	4
	4	–	5	4/5	5	5	5	4
	5	5	5	4/5	5	5	5	4
Acid dye (nil)	1	–	3	3	2	4	5	2/3
	2	–	3/4	3/4	2/3	4/5	5	3
	3	–	3/4	4	3	4/5	5	3
	4	–	3/4	4/5	3/4	4/5	5	3
	5	3	4	4/5	3/4	4/5	5	3
Acid dye (syntan)	1	–	3/4	3/4	2	4	5	3
	2	–	4	4	3	4	5	3
	3	–	4	4	3	4	5	3
	4	–	4/5	4/5	3/4	5	5	3
	5	3/4	4/5	4/5	3/4	5	5	3
Acid dye (syntan/cation)	1	–	4	4	2	4	5	2/3
	2	–	4	4	3	4	5	3
	3	–	4	4	3	4	5	3
	4	–	4/5	4/5	3/4	5	5	3
	5	3/4	4/5	4/5	3/4	5	5	3

Acknowledgement

This work was supported by Korea Research Foundation Grant (KRF-2002-003-D00466).

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