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Synthesis and application of temporarily solubilised azo disperse dyes containing β-sulphatoethylsulphonyl group

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Abstract

A series of temporarily solubilised azo disperse dyes containing β-sulphatoethylsulphonyl group in their structures were synthesised for dispersant-free dyeing of polyester. Diazo components having nitro or bromo group were prepared and coupled with corresponding coupling components to give 4-diethylamino-4′-(2-sulphatoethylsulphonyl)azobenzene dyes. The absorption maxima of the dyes were dependent upon the substituents in the diazo and coupling components and varied from 464 to 528 in DMF. Polyester fabric was successfully dyed with the synthesised dyes without using any dispersing agent. The dyes showed good build-up, leveling and fastness properties on polyester.

Keywords: Temporarily solubilised azo disperse dye; β-Sulphatoethylsulphonyl group; Dispersant-free dyeing; Polyester; Absorption maxima

1. Introduction

In the conventional disperse dyeing of polyester, dispersing agents (dispersants) are usually added to increase the dispersion stability and solubility of disperse dyes. However, after the dyeing process is finished, they are not adsorbed onto polyester and discharged as effluents with the residual dyeing liquor, which increases the COD and BOD values of the effluent [1].

In an effort to overcome some of the environmental problems associated with the use of dispersants, we prepared some temporarily solubilised disperse dyes containing β-sulphatoethylsulphonyl group and investigated the feasibility of dispersant-free polyester dyeing [2,3]. The terminal sodium sulphate group of the dye confers sufficient water solubility at room temperature without the aid of dispersants. During the dyeing procedure, the soluble dye is gradually converted to insoluble form as the β-sulphatoethylsulphonyl group is hydrolysed into vinylsulphone group through β-elimination reaction (Scheme 1). Then the water-insoluble form of the dye having substantivity to hydrophobic polyester fiber is adsorbed onto polyester. The temporarily solubilised disperse dyes were successfully dyed on polyester without using dispersants and they exhibited moderate to good fastness properties.

Recently, we have attempted to synthesise the temporarily solubilised disperse dyes which have more variety of colour range. Thus, we prepared a series of temporarily solubilised disperse dyes of azopyridone [4] and azoindole [5] derivatives for the yellow colour shade. Those dyes gave yellow, greenish yellow and yellowish orange hues on polyester. For the preparations of disperse dyes of orange, red, violet and blue shade, aminoazobenzene dyes having electron-withdrawing

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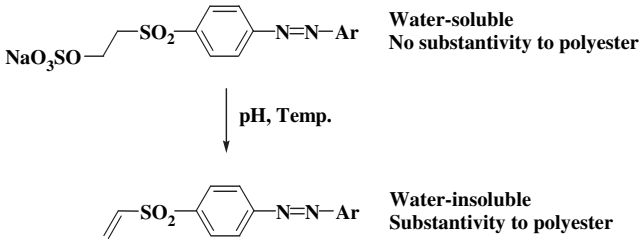


Scheme 1. Conversion of β-sulphatoethylsulphonyl group into vinylsulphone group of the temporarily solubilised disperse dyes.

groups in the diazo component and electron-donating groups in the coupling component, are commonly used.

In this paper, we prepared the diazo components having nitro or bromo group and synthesised six temporarily solubilised azo disperse dyes (**8**–**13**), 4-diethylamino-4′-(2-sulphatoethylsulphonyl)azobenzene derivatives using the diazo components and two coupling components. The correlation between the dye structure and spectral properties were discussed. Dispersant-free dyeing and fastness properties of the dyes on polyester fabric were also investigated.

2. Experimental

2.1. Materials and apparatus

Sodium 4-(aminophenyl)sulphonylethylsulphate obtained from Kyung-In Synthetic Co., 4-(2-hydroxyethylsulphonyl)acetoanilide from ShinWon Chem. Co. and 3-(diethylamino)acetoanilide from M. Dohmen Korea Co. were used without further purification. All other chemicals used were of synthesis grade purchased from Aldrich Chemical Company. Melting points were determined using Electrothermal 9100, Dong-Bo Chem. Co. and are uncorrected. IR spectra were recorded on a Prospect-IR infrared spectrometer using KBr discs. ^{1}H NMR spectra were recorded with Jeol lambda series instrument at 300 MHz using DMSO-d_6 as the solvent and TMS as the internal standard. mass spectra were recorded in fast atom bombardment ionisation mode using Jeol JMS-AX505WA/HP 5890 Series II GC–Mass Spectrometer. Elemental analyses were recorded on an EA 1110. The visible spectra were measured using an HP 8452A spectrophotometer.

2.2. Synthesis of dye intermediates (Scheme 2)

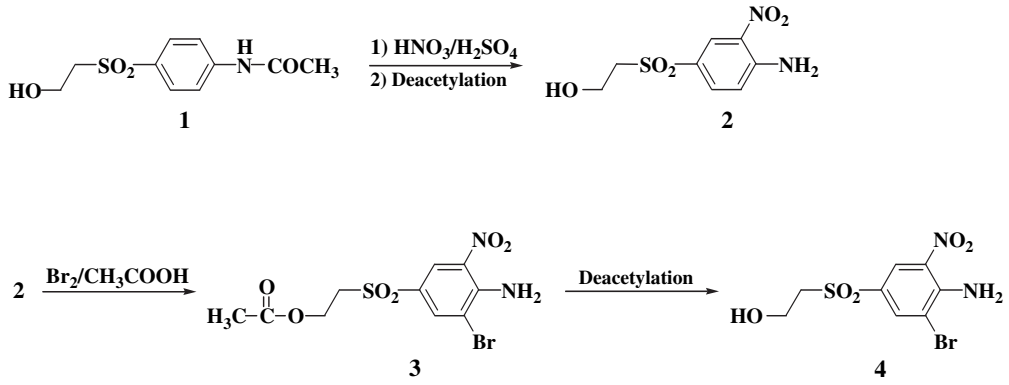
2.2.1. 4-(2-Hydroxyethylsulphonyl)-2-nitroaniline (**2**)

4-(2-Hydroxyethylsulphonyl)acetoanilide (compound **1**, 48.6 g, 0.2 mol) was added at 5 °C to the acid mixture of 95% sulphuric acid (200 ml) and fuming nitric acid (40 ml). The whole mixture was stirred for 2 h at 0–5 °C and then poured into ice. The precipitate was filtered off, washed with cold water and dried overnight. A solution of the dried solid in water (750 ml) and 95% sulphuric acid (80 ml) was stirred for 3 h under reflux. The solution was filtered hot, cooled to room temperature and neutralized with 40% NaOH solution. It was allowed to stand overnight, then filtered, washed with cold water and dried. After recrystallisation from water, light yellow product (**2**, 4-(2-hydroxyethylsulphonyl)-2-nitroaniline) was obtained.

Yield: 29.0 g (59%). m.p. 150–152 °C; FTIR (KBr) 3510(–OH), 1515 and 1350 (–NO_2), 1298 and 1147 (–SO_2) cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 3.38 (t, $J = 7.2$ Hz, 2H, CH_2SO_2), 3.65 (t, $J = 7.0$ Hz, 2H, CH_2O), 7.10 (d, 1H, ArH), 7.71 (d, 1H, ArH), 8.37 (s, 1H, ArH) ppm ; EA calcd.; $C_8H_{10}N_2O_5S$ (C, 39.02; H, 4.09; N, 11.38), found; (C, 39.05; H, 4.08; N, 11.32); m/z; 247 (MH^+).

2.2.2. 2-Bromo-4-(2-hydroxyethylsulphonyl)-6-nitroaniline (**4**)

4-(2-Hydroxyethylsulphonyl)-2-nitroaniline (compound **2**, 24.6 g, 0.1 mol) was dissolved in glacial acetic acid (200 ml). Bromine (5.63 ml, 0.11 mol) was added to the solution and the mixture was stirred for 4 h under reflux. After cooling down, it was filtered off, washed with cold water and dried giving the yellow solid (compound **3**).



Scheme 2. Schemes of diazo components synthesis.

FTIR (KBr) 1741 (−C═O), 1515 and 1360 ($-NO_2$), 1320 and 1151 ($-SO_2$) cm^{-1}; ^1H NMR (DMSO-d_6): δ 1.78 (s, 3H, CH_3CO), 3.78 (t, $J = 7.2$ Hz, 2H, CH_2SO_2), 4.27 (t, $J = 7.0$ Hz, 2H, CH_2O), 8.21 (s, 1H, aromatic), 8.43 (s, 1H, ArH) ppm; EA calcd.; $C_{10}H_{11}BrN_2O_6S$ (C, 32.71; H, 3.02; N, 7.63), found; (C, 32.64; H, 2.95; N, 7.53); m/z; 367 (MH^+), 369 ($[M + 2]H^+$).

The dried solid (**3**) was added to water (1.4 l) and 35% hydrochloric acid (400 ml) and the mixture was stirred for 2 h under reflux. The solution was filtered hot, cooled to room temperature and neutralized with 40% NaOH solution. It was then filtered, washed with cold water and dried giving yellow product (**4**, 2-bromo-4-(2-hydroxyethylsulphonyl)-6-nitroaniline).

Yield: 22.9 g (70%). m.p. 162–164 °C; FTIR (KBr) 3513(−OH), 1517 and 1358 ($-NO_2$), 1318 and 1152 ($-SO_2$) cm^{-1}; ^1H NMR (DMSO-d_6): δ 3.48 (t, $J = 7.2$ Hz, 2H, CH_2SO_2), 3.68 (t, $J = 7.0$ Hz, 2H, CH_2O), 8.20 (s, 1H, ArH), 8.43 (s, 1H, ArH) ppm; EA calcd.; $C_8H_9BrN_2O_5S$ (C, 29.55; H, 2.79; N, 8.62), found; (C, 29.67; H, 2.77; N, 8.46); m/z; 325 (MH^+), 327 ($[M + 2]H^+$).

2.3. Synthesis of dyes (Scheme 3)

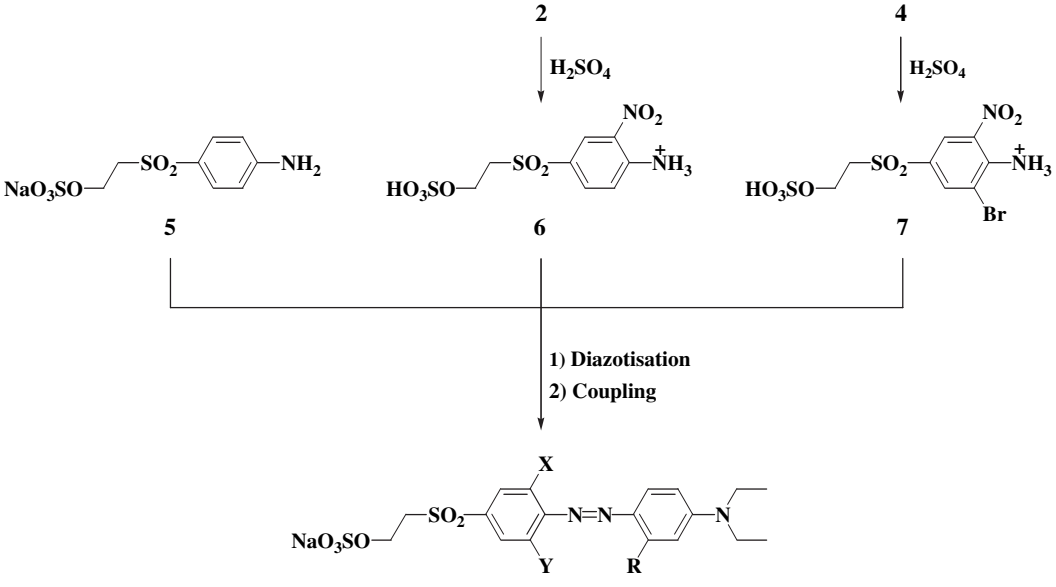
2.3.1. Diazotisation

Sodium 4-(aminophenyl)sulphonylethylsulphate (compound **5**, 2.81 g, 0.01 mol) was dissolved in water (200 ml) and concentrated hydrochloric acid (2.4 ml) at room temperature. The solution of sodium nitrite (0.69 g, 0.01 mol) in water (20 ml) was added slowly to the above solution at 0–5 °C and the diazotisation was continued for 3 h.

The compound **2** (2.46 g, 0.01 mol) was added over 1 h to 95% sulphuric acid (10 ml) and the solution was stirred for 4 h at 50 °C. Nitrosylsulphuric acid (1.97 ml, 0.01 mol, 40% in sulphuric acid) was dropped slowly into the solution at 0–5 °C and the diazotisation was continued for 3 h. The compound **4** was sulphated and diazotised in a manner similar to compound **2**.

2.3.2. Coupling

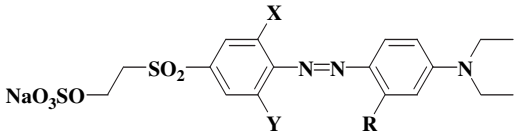
N,*N*-diethylaniline (1.49 g, 0.01 mol) or 3-(diethylamino)acetoanilide (2.06 g, 0.01 mol) was dissolved in water (60 ml) and 35% hydrochloric acid (10 ml) at room temperature. The prepared diazonium salt liquor and meshed ice (100 g) were added to the corresponding coupling component solution and the temperature was maintained between 0 and 5 °C. The solution was stirred for 2 h and allowed to reach room temperature. Sodium acetate solution (1 M) was added to adjust the pH between 4 and 5. After 30 g sodium chloride was added to the solution, the precipitate formed was filtered and dried in a vacuum oven at room temperature. A total of 3–5 g of dried dye was dissolved in DMF



8 ; X, Y = H, R = H
9 ; X, Y = H, R = $NHCOCH_3$
10 ; X = NO_2, Y = H, R = H
11 ; X = NO_2, Y = H, R = $NHCOCH_3$
12 ; X = NO_2, Y= Br, R = H
13 ; X = NO_2, Y= Br, R = $NHCOCH_3$

Scheme 3. Synthesis of temporarily solubilised azo disperse dyes.

Table 1
Yields, melting points, mass and elementary analysis data of dyes **8–13**



Dye	X	Y	R	Yield (%)	m.p. (°C)	mass (*m*/*z*)	Elemental analysis (%)			
							C	H	N	
8	H	H	H	51	204–206	464 ($[M + Na]^+$)	42.51	5.35	8.26	Calcd.
							42.60	4.74	8.82	Found
9	H	H	$NHCOCH_3$	77	208–210	521 ($[M + Na]^+$)	43.16	5.25	10.07	Calcd.
							43.19	5.02	10.03	Found
10	NO_2	H	H	60	190–192	509 ($[M + Na]^+$)	42.52	4.16	11.02	Calcd.
							42.30	4.51	11.40	Found
11	NO_2	H	$NHCOCH_3$	81	192–194	566 ($[M + Na]^+$)	42.47	4.28	12.38	Calcd.
							42.22	4.75	12.22	Found
12	NO_2	Br	H	63	201–203	587 ($[M + Na]^+$)	36.81	3.43	9.54	Calcd.
						589 ($[(M + 2) + Na]^+$)	37.02	3.89	9.39	Found
13	NO_2	Br	$NHCOCH_3$	87	203–205	644 ($[M + Na]^+$)	34.39	4.18	10.03	Calcd.
						646 ($[(M + 2) + Na]^+$)	34.61	3.56	10.02	Found

(20 ml) and the solution was poured into dichloromethane to give crytallized dyes **8–13**.

The yields, melting points, mass and elementary analysis data of the synthesised dyes **8–13** are given in Table 1.

2.4. Dispersant-free dyeing of polyester

Polyester fabric was dyed in an Ahiba dyeing machine at a liquor ratio of 20:1. The dyebaths were prepared with the synthesised dyes **8–13** without using any dispersants and buffered as follows: at pH 4 and 5 with sodium acetate (0.05 M)/acetic acid, at pH 6, 7 and 8 with sodium dihydrogen phosphate (0.05 M)/disodium hydrogen phosphate and at pH 10 with sodium dihydrogen phosphate (0.05 M)/trisodium phosphate. Dyeing was performed at 70 °C. The dyebath temperature was raised at a rate of 1 °C/min to 130 °C, maintained at this temperature for 60 min, and rapidly cooled to 60 °C. The dyeings were rinsed and then reduction cleared in an aqueous solution of 2 g/l sodium hydroxide and 2 g/l sodium hydrosulphite at 80 °C for 30 min.

The colour parameters of the dyed fabrics were determined on a Macbeth coloreye 3000 spectrophotometer, under illuminant D_{65} using the 10° standard observer with specular component excluded and UV component included.

The exhaustion of dye on the polyester fabric was measured by DMF extraction method (30 min at 150 °C). The absorbance of the solution extracted was determined using an HP 8452A UV/VIS spectrophotometer. The percentage exhaustion was calculated using Eq. 1:

$$\text{Exhaustion } (\%) = C_t/C_0 \times 100 \quad (1)$$

where C_t is the amount of dye extracted from a dyed fabric at time t and C_0 is the amount of dye in the initial dyebath.

Five locations on the dyed fabric were arbitrarily chosen and L^*, a^* and b^* values were measured by spectrophotometer. The CIEL$^*a^*b^*$ colour difference between any two points was calculated [6]. The leveling properties of the dyes on polyester fabric were assessed using the mean of 10 such colour difference results.

2.5. Fastness test

The dyed fabrics were subjected to wash (ISO 105-C06/C2S:1994), light (AATCC Test Method 16-1998), and rub (ISO 105-X12:2001) fastness tests after heat setting at 180 °C for 60 s. The shade change, together with staining of adjacent fabrics, was assessed according to appropriate SDC grey scale.

Table 2
The spectral data of synthesised dyes **8–13**

Dye	λ_{max} [a] (nm)	ϵ_{max} [a] ($l\,mol^{-1}\,cm^{-1}$)
8	464	30,000
9	490	33,200
10	502	35,200
11	520	40,600
12	508	30,800
13	528	36,800

[a] Measured in DMF.

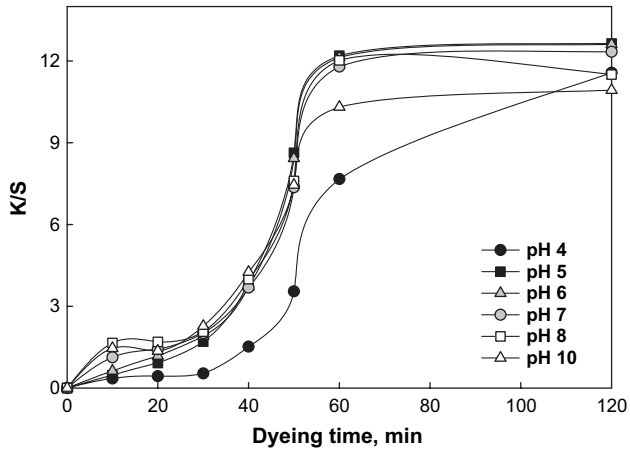


Fig. 1. Effect of pH on the colour yield of dye **12** on polyester fabric (1% owf, liquor ratio 20:1).

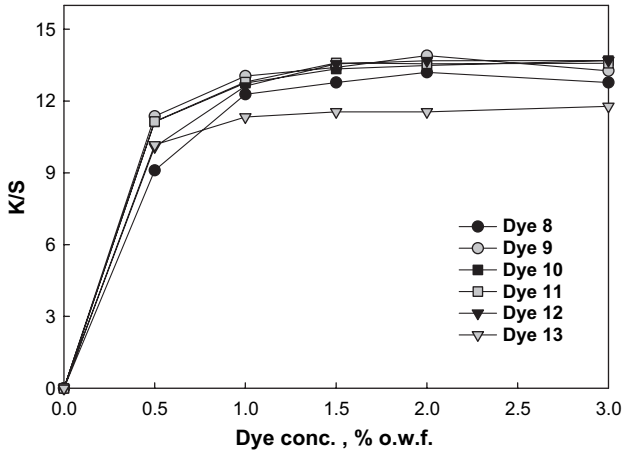


Fig. 3. Colour build-up of dyes **8**–**13** on polyester fabric (pH 5, liquor ratio 20:1).

3. Results and discussion

3.1. Synthesis of intermediates and dyes

The diazo components, 4-(2-hydroxyethylsulphonyl)-2-nitroaniline (**2**) and 2-bromo-4-(2-hydroxyethylsulphonyl)-6-nitroaniline (**4**) were synthesised as illustrated in Scheme 2. The structures of **2** and **4** were characterised by FTIR, ^{1}H NMR, elemental analysis, and mass spectrometry.

4-(2-Hydroxyethylsulphonyl)acetoanilide (**1**) was used as a starting material and nitrated using a mixture of concentrated nitric and sulphuric acids because the terminal amino group, if unprotected, would be attacked by active electrophilic nitronium ion, NO_2^+ during the nitration reaction. The electrophilic attack is expected to occur in the *ortho* to the activating acetamide group and *meta* to the deactivating hydroxyethylsulphonyl group. The nitrated compound was then deacetylated to give 4-(2-hydroxyethylsulphonyl)-2-nitroaniline (**2**).

The intermediate **2** was subjected to a bromination reaction with bromine in glacial acetic acid. The electrophilic attack is expected to occur in the *ortho* to the amino group and *meta* to the nitro and hydroxyethylsulphonyl group. Under the reaction condition of bromination, the terminal hydroxyl group was acetylated by acetic acid and we characterised the acetylated compound **3**. Therefore, the intermediate **4** could be obtained by subsequent deacetylation of compound **3**.

Sodium 4-(aminophenyl)sulphonylethylsulphate (**5**) was used as a diazo component for the preparation of dyes **8** and **9**. It was diazotised using hydrochloric acid and sodium nitrite. The synthesised intermediates **2** and **4** for dyes **10**–**13** were sulphated by dissolving them in sulphuric acid to give compounds **6** and **7**. Then these diazo components containing β-sulphatoethylsulphonyl group were diazotised using nitrosylsulphuric acid instead of hydrochloric acid and sodium nitrite because the electron-withdrawing nitro or bromo group made the diazo components weaker basic amines.

The six dyes (**8**–**13**) were prepared by coupling of the diazotised compounds of **5**, **6** and **7** with *N*,*N*-diethylaniline or 3-(diethylamino)acetoanilide as illustrated in Scheme 3. The structures of the synthesised

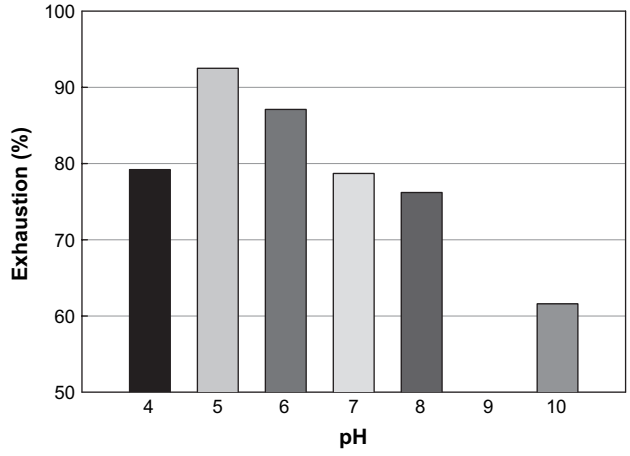


Fig. 2. The percentage exhaustion value of dye **12** on polyester fabric (1% owf, liquor ratio 20:1).

Table 3
The colour data of dyes **8**–**13** on polyester fabrics

Dye	CIEL*a*b* values			ISCC-NBS colour name
	*L**	*a**	*b**	
8	59.85	43.28	60.67	Vivid reddish orange
9	52.50	54.66	46.50	Vivid reddish orange
10	37.01	44.67	18.33	Moderate red
11	30.96	35.69	5.72	Dark purplish red
12	33.71	39.01	10/07	Dark red
13	28.95	28.85	−11.25	Dark reddish purple

Table 4
Colour differences between five points of polyester fabric

Dye	Colour differences										
	1	2	3	4	5	6	7	8	9	10	Avg.
8	0.36	0.54	0.42	0.39	0.60	0.13	0.33	0.54	0.29	0.29	0.39
9	0.21	0.05	0.26	0.42	0.23	0.09	0.27	0.26	0.44	0.33	0.26
10	0.45	0.55	0.25	0.12	0.25	0.26	0.46	0.30	0.49	0.21	0.34
11	0.27	0.22	0.39	0.42	0.23	0.26	0.35	0.46	0.53	0.13	0.32
12	0.42	0.42	0.72	0.67	0.22	0.45	0.37	0.31	0.26	0.12	0.40
13	0.48	0.45	0.28	0.15	0.14	0.26	0.46	0.17	0.38	0.21	0.30

dyes **8**–**13** were characterised by mass and elemental analysis as shown in Table 1. The melting points of the dyes were similar to each other and ranged between 190 and 210 °C.

3.2. Spectral properties of dyes

Table 2 shows the spectral data of synthesised dyes **8**–**13**. The absorption maxima of the dyes varied from 464 to 528, which was orange to red shade. Comparing the λ_{max} values and molar extinction coefficients of dyes **8**, **10** and **12** with those of **9**, **11** and **13**, a bathochromic (18–26 nm) and a hyperchromic effect (increase of ϵ_{max}) were observed. The bathochromic effect can be attributed to the introduction of electron-donating acetylamino group into the coupling component or push moiety in the push–pull chromophoric system. The higher tinctorial strength of dyes **9**, **11** and **13** than those of dyes **8**, **10** and **12** are probably due to the intramolecular hydrogen bond between the acetylamino group and the azo linkage [7].

The effect of the substituents in the diazo component ring on the λ_{max} and ϵ_{max} values was also outstanding. A large bathochromic (30–38 nm) and a hyperchromic effect were observed when comparing the λ_{max} values and molar extinction coefficients of dyes **10** and **11** into which nitro group was introduced, with those of dyes **8** and **9**. Introduction of electron-withdrawing nitro group into the diazo component or pull moiety caused the bathochromic effect. Additional introduction of bromo group at *ortho* to the azo linkage in dyes **12** and **13** resulted in a small bathochromic effect and a notable reduction in tinctorial strength. The decrease of strength can be explained by steric hindrance. Thus, with two *ortho*-substituents of nitro and bulky bromine group, the dye structures are hindered and planarity can be broken while mono *ortho*-substituted dyes (**10** or **11**) will not normally exert much of a steric effect.

3.3. Dyeing properties

As shown in a previous study, pH is a crucial factor in the application of temporarily solubilised disperse dyes [2]. Fig. 1 shows the colour yield of dye **12** on polyester fabric at various pH values. The results at pH 5, 6, 7 and 8 show a similar trend to that observed for conventional polyester dyeing and good colour yields were obtained. Although the colour yields at pH 7 and 8 were similar to those at pH 5 and 6, the dyeings showed some unlevelness. Therefore, the optimum pH condition for dyeing was concluded as pH 5 and 6. At pH 4, colour yield was low due to the low conversion rate of the soluble dye into the insoluble vinylsulphone form. However, the colour yield on the polyester fabric increased continuously throughout the whole dyeing procedure, implying that the conversion of dye occurred steadily. The low dye uptake and poor leveling at pH 10 can be attributed to the rapid conversion of dye causing a collapse in the dyebath dispersion stability. The percentage exhaustion values of dye **12** on the polyester fabric at various pH values are shown in Fig. 2. The exhaustion was highly dependent on the pH value with maximum exhaustion of 93% being obtained at pH 5.

Table 5
The wash fastness of dyes **8**–**13** (1% owf) on polyester fabric

Dye	Change	Staining					
		Acetate	Cotton	Nylon	Polyester	Acryl	Wool
8	4/5	4/5	5	4	5	5	4
9	4/5	4/5	5	4	5	5	4/5
10	4	4	5	4	5	5	4
11	4	4	5	3/4	4/5	5	4
12	4/5	4/5	5	4	5	5	4/5
13	4/5	4/5	5	4	5	5	4/5

Table 6
The light and rubbing fastness of dyes **8**–**13** (1% owf) on polyester fabric

Dye	Light	Rubbing	
		Staining	
		Dry	Wet
8	5	4/5	4/5
9	4/5	4/5	4/5
10	4	4	4/5
11	3/4	4	4
12	3	4/5	4/5
13	4	4	4/5

Fig. 3 shows the build-up of the dyes at pH 5. Dyes **8–13** exhibited good build-up on the polyester and the colour strength of these dyes reached saturation at ca. 2.0% owf. The dyes gave reddish orange, red, purplish red and reddish purple hues on polyester fabrics as shown in Table 3. The colour data were well consistent with the result of spectral data (Table 2). All the dyes showed good leveling on the polyester fabric. Table 4 gives the average of 10 results for the colour differences at five random locations on polyester fabric. All the dyes showed very small colour differences between locations, showing that leveling was good.

3.4. Fastness properties

All the dyeings for the fastness tests were carried out at pH 5, which had been demonstrated to be the optimum. The results of the wash fastness tests for dyes **8–13** on polyester are summarised in Table 5 and showed good to excellent wash fastness. Table 6 gives the results of light and rubbing fastness tests. The rubbing results were good and light fastness was moderate to good.

4. Conclusions

Temporarily solubilised azo disperse dyes of 4-diethylamino-4′-(2-sulphatoethylsulphonyl)azobenzene derivatives were synthesised and dispersant-free polyester dyeing was examined. Introduction of nitro group in the diazo component ring or acetylamino group in the coupling component ring resulted in the bathochromic effect and increase of extinction coefficient of the dye. Bromo group also increased the absorption maxima of the dyes but decreased the colour strength probably because of the steric hindrance. Polyester fabrics were successfully dyed with the synthesised dyes without using dispersing agent. The colour yield on polyester was dependent on the dyeing pH and the optimum result was obtained at pH 5. The dyes gave orange to purple hues on polyester fabrics and showed good build-up and leveling properties. The dyes exhibited good to excellent wash fastness while rubbing and light fastness results were good and moderate to good.

Acknowledgements

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