

Union dyeing of wool/polyester blend fabric using sulphatoethylsulphone dye derivative of C.I. Disperse Yellow 23

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Abstract: This paper aims to synthesise, characterise and find out the properties of a model dye for convenient union dyeing of wool, polyester and wool/polyester blend fabric compared with C. I. Disperse Yellow 23. The reactive disperse dye was prepared containing sulphatoethylsulphone (SES) as a reactive group. The dye was synthesised by diazotization and coupling reaction. Firstly, we synthesized azo dye intermediate **I** using 1-aminobenzene-4-sulphatoethylsulphone diazotized and then coupled it with aniline. The synthesized azo dye intermediate **I** was diazotized and coupled with phenol to give dye **2**. Different factors affecting the dyeability and fastness properties of SES dye **2** were thoroughly investigated on wool, polyester and wool/polyester blend fabrics in comparison with C. I. Disperse Yellow 23 dye **1**. Maximum exhaustion and total fixation yield using sulphatoethylsulphone (SES) dye **2** were achieved on wool fabric at neutral pH 7. The dye showed high dyeing performance due to its nonionic reactive VS derivative. The dyeing results indicate high quality dyeing properties.

Keywords: Synthesis, reactive disperse dyes, union dyeing, wool/polyester blend fabric, structure elucidation.

1. Introduction

It has been reported that C.I. Disperse Yellow 23 has been modified by either 2-bromoacrylic acid or 1,3,5-trichloro-2,4,6-triazine reactive groups for dyeing wool in non-aqueous (supercritical carbon dioxide) dyeing media (Schmidt et al., 2003a). Schmidt et al. (2003b) reported that C. I. Disperse Yellow 23 has been used for dyeing cotton modified by the 1,3,5-trichloro-2,4,6-triazine reactive group in non-aqueous (supercritical carbon dioxide) dyeing media. Sawada and Ueda (2007) reported that disperse dyes can be fixed on wool in non-aqueous (organic solvents) dyeing media using 4-(Dimethylamino)-pyridine and N,N'-dicyclohexylcarbodiimide as auxiliaries.

On the other hand, wool/polyester blends are exceptionally suitable for woven apparel goods (e.g. men's suits and knit dresses). Polyester is commercially dyed with disperse dyes. However, disperse dyes are not commercially used to dye wool since hydrophobic, sparingly water-soluble dyes display low substantivity towards wool and yield pale dyeings of low wet fastness properties (Bell & Lewis, 1978) Several works have been explored the possibility of modifying wool so as to increase its substantivity towards disperse dyes. Lewis and Pailthorpe (1983 & 1984) showed that chemically modified wool with various hydrophobic compounds possesses high substantivity towards disperse dyes and such treatments improve both wash and light fastness properties of dyes on the substrate. Burkinshaw and Lu (1994) reported that wool dyeing with disperse dyes in

the presence of ammonium persulphate and thiourea dioxide as radical initiators exhibit lower fixation efficiency and the resultant dyeings exhibit lower wash fastness than using conventional anionic reactive dyes.

In our previous studies (Mousa *et al.*, 2011; Renfrew & Smith, 1979), we reported that wool and wool/polyester blend fabrics have been successfully dyed at neutral conditions in aqueous dyeing media using sulphatoethylsulphone (SES) reactive disperse dyes. To the best of our knowledge, the modification of C.I. Disperse Yellow 23 with SES reactive groups for textile dyeing has not been previously reported. As part of our ongoing interest of process optimization in textile coloration, a SES derivative of C. I. Disperse Yellow 23 was initially synthesized and investigated for dyeing wool/polyester blend fabric in aqueous dyeing media. Different factors affecting the dyeability and fastness properties of SES dye **2** were thoroughly investigated on wool, polyester and wool/polyester blend fabrics in comparison with C. I. Disperse Yellow 23 dye **1**.

2. Experimental

2.1. Materials

Fabrics

Wool fabric was 100% twill weave (2/2) of equal warp and weft (26x24 threads/cm, yarn count Nm 44/2). Polyester fabric was 100% plain weave of warp and weft (72x32 thread/cm, yarn count (tex 150x1) of continuous filaments. Wool/polyester blend fabric (55/45) was plain weave of warp and weft (21x19 thread/cm, yarn count (Nm 42x2). All these fabrics were supplied by Misr El-Mahala Co. Egypt and scoured in an aqueous solution containing 2 g/L Hostapal CV for 1 hour at 80°C at a 50:1 liquor ratio (LR), and then rinsed thoroughly in water and dried at room temperature.

Chemicals

1-aminobenzene- 4- β -sulphato-ethylsulphone was obtained from Amar Impex, India. Aniline and phenol were obtained from Fluka Chemie AG. All the chemicals used in the study were of the laboratory reagent grade.

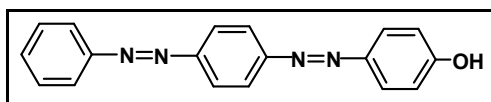
2.2. General procedure

Elemental analyses of C, H and N were established by a Vario EL III analyzer. Infrared (IR) spectra were recorded on a Nexus 670 FTIR Spectrometer (KBr; Thermo Nicolet). ¹H-NMR spectra were recorded on a JEOL JNM-EX 270 MHz spectrometer using tetramethylsilane (TMS) as an internal standard. The maximum absorption wavelength (λ_{max}) and extinction coefficient (ϵ_{max}) were measured on a Shimadzu UV-2401PC UV/Vis spectrophotometer. Dyes were evaluated for their color yield using an UltraScan PRO spectrophotometer (Hunter Lab) with a D65 illuminant and 10° standard observer.

2.3. Synthesis of dyes

2.3.1. C.I. Disperse dye 23 (Dye 1)

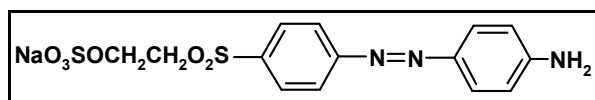
C. I. Disperse Yellow 23 (**1**) was synthesized by the previously described method (Renfrew & Smith, 1979) structure **1**.



C.I. Disperse Yellow 23 (**1**) structure **1**.

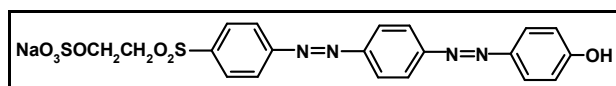
2.3.2. Sulphatoethylsulphone derivative of C. I. Disperse Yellow 23 (Dye 2)

1-aminobenzene-4- β -sulphatoethylsulphone (22.2 g, 0.075 mol) was diazotized by the previously described method (Lewis *et al.*, 2000) and then coupled with aniline (7 g, 0.075 mol) under weakly acidic conditions. The reaction mixture was stirred at 0–5°C and at pH 4-5 for 2 hours. The resulting mono azo dye intermediate **I** was filtered and dried in a vacuum oven at 40°C (yield 86 %) structure **2**.



Intermediate I structure 2.

The synthesized azo dye intermediate **I** (20.35g, 0.05 mol) was diazotized in aqueous hydrochloric acid (35%, 13 ml, 0.12 mol) and sodium nitrite (3.5g, 0.051 mol) dissolved in 5 ml water. The reaction mixture of the diazonium salt was stirred at 0-5°C for 1 hour. The diazotized intermediate **I** was added slowly to a weakly alkaline solution of phenol (4.7g; 0.05 moles) and water (50 ml). The coupling reaction was completed under such conditions within 2 hours at 0-5°C and later neutralized to pH 6.5. The precipitated disazo dye **2** (yield 81.0 %) was filtered and dried in a vacuum oven at 40°C structure **3**.



SES dye (2) structure 3.

Dye **1**: brown, m.p. >300°C, yield 80%, λ_{\max} (H₂O) 389 nm, ϵ . 31730 l mol⁻¹ cm⁻¹.

C₁₈H₁₄N₄O (302) calcd.: C, 71.52; H, 4.63; N, 18.54, found: C, 71.50; H, 4.31; N, 18.14. IR (v/cm⁻¹): 3460 (phenyl-OH), 1560 (-N=N-). ¹H-NMR: δ H (ppm) in [DMSO-²H₆] DMSO: 7.17-7.26 (3H, m, phenyl Hc*/Ha), 7.50-7.58 (6H, m, phenyl Hb/Ha'/Hb'), 7.65-7.67 (2H, d, J = 7.6 Hz phenyl Hb*), 8.00-8.02 (2H, d, J = 7.6 Hz phenyl Ha*)

Dye **2**: dark brown, m.p. >300°C, yield 85%, λ_{\max} (H₂O) 395 nm, ϵ . 26410 l mol⁻¹ cm⁻¹.

C₂₀H₁₇N₄O₇S₂ Na (512), calcd: C, 46.87; H, 3.32; N, 10.93; S, 12.50%, found: C, 46.80; H, 3.21; N, 10.80; S, 12.21 %. IR (v/cm⁻¹): 3460 (phenyl-OH), 1560 (-N=N-), 1138 (-SO₂-), 1293, 1251,

1048, and 1007 cm⁻¹ (-OSO₃H). ¹H-NMR: δ H (ppm) in [DMSO-²H₆]DMSO: 3.54-3.57(2H, t, J = 6.8 Hz, α -CH₂), 3.91-3.94 (2H, t, J = 6.8 Hz, β -CH₂), 7.24-7.26 (2H, d, J = 9.2 Hz phenyl Ha), 7.50-7.58 (6H, m, phenyl Hb/Ha'/Hb'), 7.84-7.85 (2H, d, J = 7.6 Hz phenyl Hb*), 8.00-8.02 (2H, d, J = 7.6 Hz phenyl Ha*).

2.4. Dye purification

For convenient dyeing and chemical investigation, it was necessary to purify the synthesised dyes **1, 2** (Guo et al., 1993). The purification was done by dissolving the dyes in dimethylsulphoxide at 45°C. Inorganic impurities were eliminated by filtration and the dye solution was re-precipitated in n-butanol at room temperature. The dyes were filtered, washed with acetone and dried in a vacuum oven at 40°C.

2.5. Dyeing procedures

Wool, polyester and wool/polyester blend fabrics were dyed using the SES dye derivative of C. I. Disperse Yellow 23 (**2**) at 1-5% owf in an Ahiba Polymat dyeing machine, with 2-g samples at a 50:1 LR. The dyebath was prepared at pH 3, 4, 5, 6 and 7 in 0.2 M disodium hydrogen phosphate/0.1 M citric acid buffer solutions in the presence of a 1 g/L nonionic dispersing agent (Sera Gal P-LP, DyStar) and a 1g/L leveling agent (Lyogen CN liquid, Clariant). In the case of polyester and wool/polyester blend fabrics, a 2 g/L low odor carrier (Sera Gal P-EW, DyStar) was added. Dyeing started at 40°C and the temperature raised to 100°C over 60 minutes. Dyeing continued at the desired temperature for another 90 minutes (150 minutes in total) for studying the effect of pH and dye concentration. To study the effect of dyeing time for wool, dyebath samples were examined at intervals up to the total dyeing time. After dyeing, all the dyed samples were rinsed with water and dried. Dye exhaustion and total dye fixation on wool fabric were evaluated spectrophotometrically. For comparison, C. I. Disperse Yellow 23 (**1**) was applied to all the fabrics at 2% owf dye concentration, pH 5.5 (0.2 M disodium hydrogen phosphate/0.1 M citric acid buffer solutions) and 100°C in the presence of the same dye bath auxiliaries mentioned above.

2.6. Measurements and Testing

2.6.1. Dye Exhaustion

The dye uptakes of dyes **1** and **2** for wool, polyester and wool/polyester blend fabrics were measured by sampling the dyebath before and after dyeing. The dye concentration (g/l) of the dyebath, diluted 20-fold with 50% aqueous DMF, was measured on a Shimadzu UV-2401PC UV/Vis spectrophotometer at the λ_{\max} value using a calibration curve for each dye. The dye concentration range (0.005 g/l to 0.03 g/l $\cong 9.76 \times 10^{-6}$ mol l⁻¹ to 9.93×10^{-5} mol l⁻¹) in 50% aqueous DMF was chosen to obey the Beer-Lambert law. The percentage of dyebath exhaustion (%E) was calculated using Eq. 1.

$$\%E = \left[1 - \left(\frac{C_2}{C_1} \right) \right] \times 100 \quad (1)$$

where C_1 and C_2 are the dye concentrations of the dyebath before and after dyeing respectively.

2.6.2. Dye Fixation

The dye fixation (%F), the percentage of exhausted dye chemically bound on wool fabric, was measured by refluxing the wool dyed samples in 50% aqueous DMF (20:1 LR) for 15 minutes to extract the unfixed dye (Burkinshaw *et al.*, 2001). This procedure was repeated until the extract was colorless. The concentration of the extract was then measured spectrophotometrically at the λ_{\max} value of each dye and the dye fixation ratio was calculated using Eq. 2.

$$\%F = \frac{(C_1 - C_2 - C_3)}{(C_1 - C_2)} \times 100 \quad (2)$$

where C_3 is the concentration of the extracted dye.

From the dyebath exhaustion (%E) and dye fixation (%F), the total dye fixation (%T), the percentage of chemically bound dye relative to the total amount of dye used, was calculated for wool dyeings using Eq. 3.

$$\%T = \frac{(\%E \times \%F)}{100} \quad (3)$$

2.6.3. Colour measurements

The colour parameters of the un-dyed and dyed wool, polyester and wool/polyester blend fabrics were determined using an UltraScan PRO spectrophotometer (Hunter Lab) with a D65 illuminant and 10° standard observer (Hu *et al.*, 1987; Savarino *et al.*, 1989).

2.6.4. Fastness Testing

The dyed wool, polyester and wool/polyester blend samples, after washing-off using a 2 g/L nonionic detergent at 80°C for 15 minutes, were tested by standard ISO methods. Washfastness (ISO 105-C02 (1989), crockfastness (ISO 105-X12 (1987)), and fastness to perspiration (ISO 105-E04 (1989)) were evaluated using the visual ISO Gray Scale for both color change (AATCC Evaluation Procedure (EP) 1—similar to ISO 105-A02) and color staining (AATCC EP 2—same as ISO 105-A03). Lightfastness (carbon arc) was evaluated using ISO 105-B02.

3. Results and Discussion

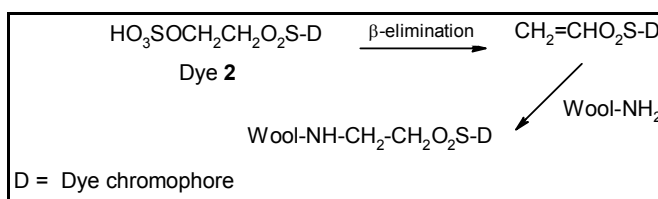
3.1. Dye Characterization

The expected tendency of the synthesized dye **2** towards a lower ϵ_{\max} value (26410 l mol⁻¹ cm⁻¹) relative to C. I. Disperse Yellow 23, dye **1** (31730 l mol⁻¹ cm⁻¹) was possibly caused by the electronic effect of the additional temporarily anionic SES group in the dye molecule. The IR spectra of dye **2** showed

characteristic absorption bands of the $-\text{OSO}_3\text{H}$ group. The IR spectra also showed absorption bands at 1138 cm^{-1} for the $-\text{SO}_2-$ group. The $^1\text{H-NMR}$ spectrum of dye **2** was characterized by two triplet peaks at 3.54-3.57ppm (2H) and 3.91-3.94 ppm (2H), assignable to the α - and β -methylene protons of the SES respectively.

3.2. Effect of dyebath pH

The dyeing properties of dye **2** were initially investigated on wool fabric by varying the pH from 3 to 7. The extent of dye exhaustion and total fixation yield showed maximum values at neutral pH 7 (Fig. 1), which was mainly attributed to the higher β -elimination reaction of the temporarily anionic SES group in the dye structure which generates a nonionic reactive vinylsulphone (VS) derivative (Lee & Kim, 1999). At a low pH, the exhaustion of dye **2** onto wool was favoured by electrostatic interaction between the unconverted temporarily anionic SES group of the dye and the protonated amino groups on wool fabric. However, a higher pH resulted in the conversion of the SES group into reactive VS species as shown in scheme 1, favouring better total dye fixation. It is also clear that the dyeing results were more satisfactory at the neutral pH as the generated nonionic VS derivative would account for higher fixation values relative to acidic dyeing.



Scheme 1

Conversely, conventional anionic reactive dyes were applied to wool fabric under weakly acidic condition, but their use under neutral conditions was not favorable because of low dye substantivity (Cho & Lewis, 2002). The temporarily anionic dye **2** operated in a different manner at neutral dyeing conditions, which confirms the suitability of applying such a dye to wool dyeing owing to its high substantivity and total fixation yield.

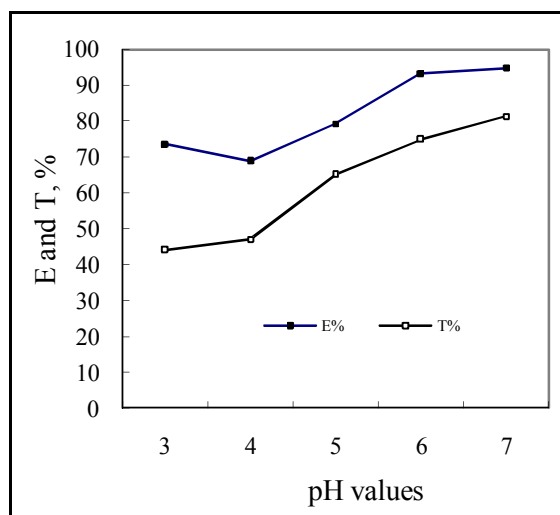


Fig. 1. Exhaustion (E) and total fixation (T) values of dye **2** (2% owf, 100°C for 90 minutes and various pH values) on wool fabric

The results of dyeing polyester and wool/polyester blend fabrics using dye **2** at different pH values are shown in Fig. 2. Dye **2** exhibits lower exhaustion values at $\text{pH} \leq 4$ on polyester. This was probably caused by the relatively hydrophilic character of the unconverted temporarily anionic SES group in dye **2**, which suppressed the adsorption of dye molecules into hydrophobic polyester fiber. Fig. 2 shows that the exhaustion values on polyester increases when the pH is increasing. In this case, exhaustion was favoured by a higher pH, because, when increasing the pH, the anionic form of the SES dye was progressively converted into a nonionic vinyl sulphone form, which had higher substantivity for polyester. However, on both wool and wool/polyester, exhaustion decreased between pH 3 and pH 4, and increased above pH 4 probably because the exhaustion onto wool decreased as the pH increased from pH 3 to pH 4. The decrease could not be compensated by the increase

in substantivity of the nonionic reactive form of the dye, because at a low pH the nonionic reactive form is not present in a sufficiently high concentration. However, above pH 4 the concentration of the reactive species progressively increased, which explained the increased exhaustion with the further increase in pH. As a consequence, the blend dyeings using dye 2 exhibited better results of exhaustion than those of dye 1. The results shown in Table 1 indicate that the dye uptake of dye 2 at the neutral pH is likely to behave similarly to the disperse dye 1 on polyester at pH 5.5, exhibiting approximately the same K/S values. Interestingly, a better colour yield on wool/polyester fabrics was detected at the neutral pH for dye 2 and showed comparable K/S values in the case of wool dyeing (Table 1), which suggests the success of using the SES dye derivative of C. I. Disperse Yellow 23 for a neutral dyeing of wool and wool/polyester blend fabrics.

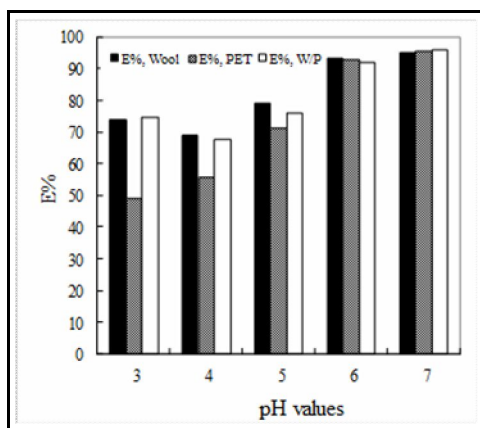


Fig. 2. Effect of pH on exhaustion values of dye 2 (2% owf, 100°C for 90 minutes and various pH values) on wool, polyester and wool/polyester blend fabrics

3.3. Effect of Dyeing Time

Having established that the optimum dyeing pH was 7 for dye 2, the extent of exhaustion and total fixation yield was investigated on wool at different dyeing times using 2% owf dye concentration. From the results in Fig. 3, the dye fiber reaction is characterized by fast initial rate followed by slower rate within the last 30 minutes of the dyeing process. The high rate of dye fixation could be attributed to the structure-reactivity of the nonionic VS reactive system which not only imparted high substantivity but also exhibited high fixation efficiency.

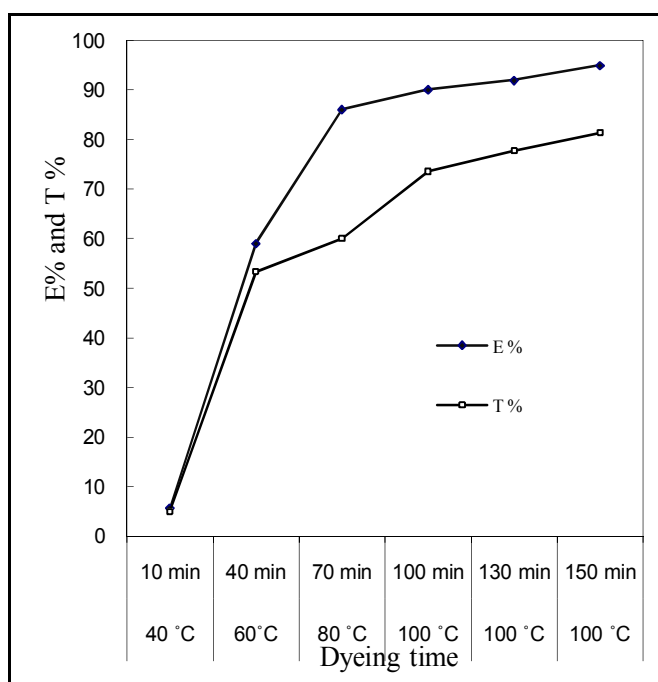


Fig. 3. Effect of dyeing time on exhaustion (E) and total fixation (T) values of dye 2 (2% owf) on wool fabric at pH 7

3.4. Effect of dye concentration

The exhaustion and total fixation yield of dye 2 on wool fabric were examined using different depths of shades (1-5% owf) at pH 7 and at 100°C. The results are shown in Fig. 4. The high fixation efficiency of the VS derivative of dye 2 might offer further dye uptake, even at higher dye concentrations. The effect of dye concentration on dye exhaustion on polyester and wool/polyester blend fabrics is shown in Fig. 5. Increasing dye concentration reduces exhaustion on polyester and blend fabrics and exhibits approximately the same manner in wool dyeing (Fig. 4). This observation shows that the SES dye derivative of C. I. Disperse Yellow 23 can be applied to polyester/wool blend fabric, even at a high dye concentration.

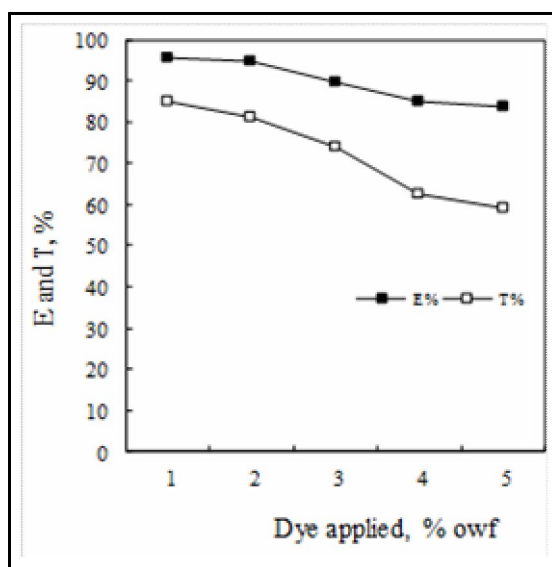


Fig. 4. Exhaustion (E) and total fixation (T) values at different concentrations of dye 2 (100°C for 90 minutes and pH 7) on wool fabric.

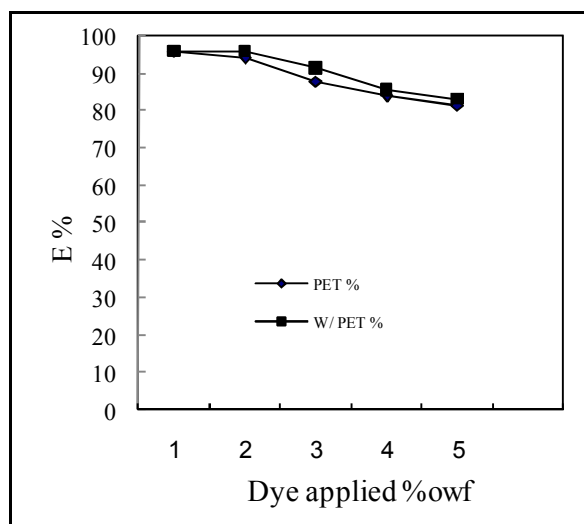


Fig. 5. Exhaustion (E) values at different concentrations of dye 2 (100°C for 90 minutes and pH 7) on polyester and wool/polyester blend fabrics

3.5. Colorimetric and Fastness properties

The results shown in Table 1 indicate that the dye uptakes of disperse dye 1 and reactive disperse dye 2 are affected by the introduction of the SES group in the dye structure, enhancing the K/S values of the dyed wool fabrics as confirmed by the K/S values of dye 2 (27.40) relative to dye 1 (5.69). While dye 2 could produce high K/S values for all fibers and dye 1 for polyester fiber, their abilities can be attributed to their highly conjugated system, planarity of dye molecules and relatively high extinction coefficients. Also, the

relatively high L^* values of dye **2** might be caused by the presence of an additional electron-acceptor sulphonyl group along with an electron-donor hydroxyl group through a highly conjugated dye chromophore.

The colourimetric CIE $L^*a^*b^* C^*h^\circ$ data on un-dyed (blank) and dyed wool, polyester and wool/polyester blend fabrics using both dyes **1** and **2** are shown in Table I. It can be seen that the chroma (C^*) and hue (h°) values of the dyed wool, polyester and wool/polyester blend fabrics using the SES dye derivative of C. I. Disperse Yellow 23 (**2**) are approximately similar when taking into account those values of the blank fabrics used. Therefore, a successful union dyeing of the wool/polyester blend fabric could possibly be achieved. Additionally, the high molar extinction coefficient of synthesized dyes can impart high K/S values of dyed fabrics.

Table I. Colorimetric data of un-dyed and dyed wool, polyester and wool/polyester blend fabrics using dyes 1 and 2 (2% owf) at 100°C for 90 minutes (pH 7 for modified dye 2 and pH 5.5 for dye 1).

Dye	Fabric	Colour on fibers	K/S	L^*	a^*	b^*	C^*	h°
Blank	W	--	--	86.84	-1.23	12.13	12.20	95.80
	PET	--	--	91.18	-0.32	1.40	1.43	102.91
	W/PET	--	--	90.56	-0.77	7.77	7.80	95.63
1	W	yellow	5.69	71.86	6.47	48.06	48.49	82.33
	PET	yellow	23.10	53.13	16.48	62.64	64.77	75.26
	W/PET	yellow	6.51	69.75	10.76	51.53	52.65	78.20
2	W	yellowish-orange	27.40	50.46	42.03	71.13	82.62	59.42
	PET	yellow	22.38	53.56	26.44	72.21	76.90	69.89
	W/PET	yellowish-orange	25.86	50.32	43.19	69.37	81.72	58.69

The colour yield and fastness properties of dyes **1** and **2** on wool, polyester and wool/polyester blend fabrics were evaluated. The results in Table II show that the colour fastness properties of rubbing, washing and perspiration of the aforementioned dyes are good to excellent and are approximately the same, depending on the proportion of the dye fixed.

Table II. Fastness properties of modified dye 2 and commercial dye 1 on wool, polyester and wool/polyester blend fabrics (100°C and 2% owf of both dyes, pH 7 for dye 2 and pH 5.5 for dye 1)

Dye	Fabric	Fastness to rubbing		Wash fastness			Fastness to Perspiration						Light
		Dry	Wet	Alt	SC	SW	Alkaline			Acidic			
							Alt	SC	SW	Alt	SC	SW	
1	W	1	1-2	1	4	1-2	1	4-5	1-2	1	4	1-2	5-6
	PET	5	5	5	5	5	5	5	5	5	5	5	5-6
	W/ PET	2	2-3	2	4-5	2	2	4	2-3	2	4-5	2-3	5-6
2	W	5	5	4-5	4-5	4-5	4-5	5	4-5	5	4-5	4-5	5-6
	PET	5	5	5	5	5	5	5	5	5	5	5	5-6
	W/PET	5	5	4-5	4-5	4-5	4-5	5	4-5	5	4-5	4-5	5-6

Alt = Alteration of dyed sample; SC = staining on cotton; SW = staining on wool.

4. Conclusion

A model of the sulphatoethylsulphone (SES) dye derivative of C.I. Disperse Yellow 23 was synthesized and applied to wool, polyester and wool/polyester fabrics. Dye **2** exhibited lower exhaustion values at $pH \leq 4$ on polyester. This was probably caused by the relatively hydrophilic character of the unconverted temporarily anionic SES group in dye **2**, which suppressed the adsorption of dye molecules into hydrophobic polyester fiber. The maximum exhaustion and total fixation yield using the sulphatoethylsulphone (SES) dye **2** were

achieved on wool fabric at neutral pH 7. The dye showed high dyeing performance due to its nonionic reactive VS derivative. The temporarily anionic SES disperse dye system could replace the conventional anionic/disperse dye system in wool/polyester blend dyeing with an improved process.

As shown in Table II, the fastness properties of washing, rubbing and perspiration of all the samples dyed with dye 2 were very good to excellent irrespective of the fabric used. Dye 2, having high fixation on wool, gave better fastness results than conventional disperse dye especially on wool and wool/polyester blend fabrics. Also, for each dyed fabric, both dyes 1 and 2 showed the same light fastness properties. This seems reasonable as the investigated dyes had approximately the same chromogen.

Different factors affecting the dyeability and fastness properties of sulphatoethylsulphone (SES) dye 2 were thoroughly investigated on wool, polyester and wool/polyester blend fabrics in comparison with C. I. Disperse Yellow 23 (dye 1). Very good to excellent wet fastness properties on all the dyed fabrics were achieved using the sulphatoethylsulphone (SES) dye derivative if compared with C. I. Disperse Yellow 23

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