

**UNION DYEING OF WOOL/POLYESTER BLEND FABRIC USING A SULPHATOETHYLSULPHONE DYE ANALOGUE OF C.I. DISPERSE YELLOW 16**

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**Abstract**

Dyeing of wool, polyester and wool/polyester blend fabrics with a sulphatoethylsulfone dye analogue of the commercial C. I. Disperse Yellow 16 were investigated. Maximum exhaustion values and colour yield were observed using the modified disperse dye at pH 7. The result showed that the modified disperse dye was more convenient for union dyeing of wool/polyester blend fabric compared with the commercial disperse dye. Excellent to very good wet fastness properties on all dyed fabrics were achieved using the modified disperse dye if compared with the commercial disperse dye.

**Keywords:** Pyrazolone dyes; Reactive disperse dyes; Wool/polyester blend fabric; Neutral dyeing.

**Introduction**

Wool/polyester blends are exceptionally suitable for woven apparel goods e.g. men's suiting fabrics or knit dress goods because they have a level of abrasion resistance, crease resistance and strength which is not matched by wool. Polyester fabric is commercially dyed with disperse dyes. However disperse dyes are not commercially used to dye wool since the hydrophobic, sparingly water-soluble dyes display low substantivity towards wool and yield pale dyeings of low wet fastness properties[1-4].

Several workers have explored the possibility of modifying wool so as to increase its substantivity towards disperse dyes. Asquith *et al* [2] showed that the substantivity of disperse dyes towards the wool fibre could be increased by increasing the hydrophobicity of the fibre by either chemically modifying or physically blocking the hydrophilic groups of the fibre. However, such treatments resulted in wool dyeings of low wet fastness. In contrast, Lewis *et al* [5, 6] showed that the chemically modified wool with various hydrophobic compounds possessed high substantivity towards disperse dyes and such treatments improved both the wash fastness and light fastness of the dyes on the substrate. Burkinshaw *et al* [7] reported that dyeing of wool with disperse dyes in the presence of ammonium persulphate or thiourea dioxide radical initiator exhibited lower fixation efficiency

and, thus, the resultant dyeings exhibited also lower wash fastness than that typically obtained using conventional anionic reactive dyes. The dye-fibre covalent attachment and/or disperse dye polymer formation within the wool substrate may also contribute to the shade changes of the dyed wool fibre. Recently, Sawada *et al* [8] reported that disperse dyes can be fixed on wool fibre in the presence of 4-(Dimethylamino)-pyridine and N,N'-dicyclohexyl-carbodiimide as auxiliaries in non-aqueous dyeing media. However, application of organic solvents as non-aqueous dyeing media would not be industrially acceptable. All the above researchers have been interested in the modification of wool fabric to increase its substantivity towards commercial disperse dyes.

As a part of our ongoing interest of process optimization in textile coloration, we present here a new approach of dyeing wool/polyester blend fabric. A sulphatoethylsulphone analogue of the commercial C. I. Disperse Yellow 16 was initially synthesized and investigated for dyeing wool/polyester blend fabric. Different factors affecting the dyeability and fastness properties of the modified dye structure (dye 2) were thoroughly investigated on wool, polyester and wool/polyester blend fabrics in comparison with C. I. Disperse Yellow 16 (dye 1).

## **Experimental**

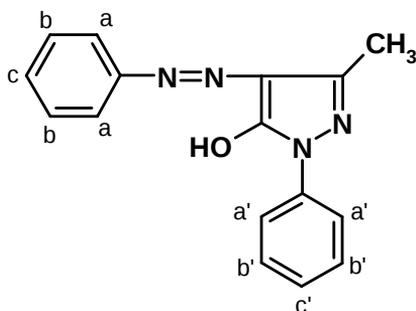
### **Materials**

Wool fabric, 100% was twill weave fabric (2/2) of equal warp and weft (26x24 threads/cm, yarn count Nm 44/2). Polyester fabric, 100% was plain weave fabric of warp and weft (72x32 thread/cm, yarn count (tex 150x1) continuous filament, yarn count. Wool/polyester blend fabric (55/45) was plain weave fabric of warp and weft (21x19 thread/cm, yarn count (Nm 42x2). All these fabrics, supplied from Misr El-Mahala Co. Egypt, were treated in an aqueous solution containing 2 g/L Hostapal CV for 1 h at 80°C and a 50:1 liquor ratio (LR), then washed thoroughly in water and air dried at room temperature. 1-aminobenzene- 4- $\beta$ -sulphato-ethylsulphone was obtained from Amar Impex, India. All chemicals used in the study were of laboratory reagent grade.

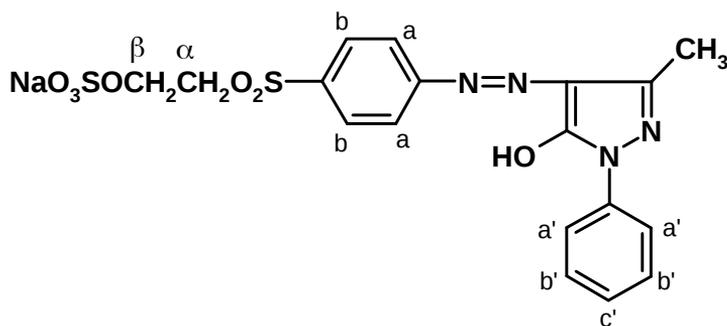
### **Synthesis of dyes**

Typically, the synthesis of C. I. Disperse Yellow 16 (1) and its modified structure 2 was carried out by initial preparation of 1-phenyl-3-methyl-5-pyrazolone coupling component 3 following a previously reported method (mp. 129-130°C) [9]. The

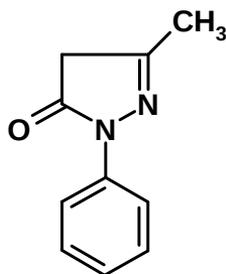
pyrazolone product was then coupled with the diazo salt solutions of aniline [10] and *p*-aminophenyl sulphatoethylsulphone [11]. The coupling reactions were completed within 2 h at pH 6.5 with the formation of the desired C. I. Disperse Yellow 16 **1** or its modified dye structure **2**. The precipitated dyes **1** and **2** were filtered off and dried in a vacuum oven at 40°C. Dyes **1** and **2** were characterized by maximum absorbance wavelength ( $\lambda_{\max}$  in 50% aqueous DMF) 389 nm.



C.I. Disperse Yellow 16 (Dye 1)



Modified disperse dye (Dye 2)

1-phenyl-3-methyl-5-pyrazolone (**3**)

Dye **1** structure was confirmed by elemental analysis. Calculated for  $C_{16}H_{14}N_4O$ : C 69.06; H 5.04; N 20.14. Found: C 69.21; H 5.07; N 20.03. IR ( $\nu/cm^{-1}$ ): 3431 (pyrazolone-OH), 1556 (-N=N-).  $^1H$ -NMR:  $\delta H$  (ppm) in [ $^2H_6$ ] DMSO: 2.27 (3H, s, -CH<sub>3</sub>), 7.17-7.21 (2H, m, phenyl Hc/Hc'), 7.41-7.44 (4H, t, J = 7.6 Hz, phenyl Hb/Hb'), 7.59 (2H, d, J = 8.4 Hz, phenyl Ha'), and 7.86 (2H, d, J = 6.4 Hz, phenyl Ha).

Dye **2** structure was confirmed by elemental analysis. Calculated for  $C_{18}H_{17}N_4O_7S_2Na$ : C, 44.26; H, 3.51; N, 11.47; S, 13.13. Found: C, 44.40; H, 3.55; N, 11.31; S, 13.04. IR ( $\nu/cm^{-1}$ ): 3437 (pyrazolone-OH), 1559 (-N=N-), 1142 (-SO<sub>2</sub>-), 1249, 1139, 1055, and 999  $cm^{-1}$  (-OSO<sub>3</sub>H).  $^1H$ -NMR:  $\delta H$  (ppm) in [ $^2H_6$ ]DMSO: 2.29 (3H, s, -CH<sub>3</sub>), 3.58-3.61(2H, t, J = 6.8 Hz,  $\alpha$ -CH<sub>2</sub>), 3.89-3.92 (2H, t, J = 6.8 Hz,  $\beta$ -CH<sub>2</sub>), 7.18-7.22 (1H, t, J = 7.6 Hz, phenyl Hc'), 7.42-7.45 (2H, t, J = 6.9 Hz, phenyl Hb'), 7.80 (2H, d, J = 9.1 Hz, phenyl Ha'), and 7.87-7.90 (4H, m, phenyl Ha/Hb).

### Dyeing procedures

Wool, polyester and wool/polyester blend fabrics were dyed using dye **2** at 1- 5% (owf) in an Ahiba 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 1 g/L nonionic dispersing agent (Sera Gal P-LP, DyStar) and 1 g/L leveling agent (Lyogen CN liquid, Clariant). In case of polyester and wool/polyester blend fabrics, 2 g/L low odor carrier (Sera Gal -P-EW, DyStar) was added. Dyeing was started at 40°C and then the temperature raised to 100°C over 45 min. Dyeing was continued at the desired temperature for a further 45 min (90 min total dyeing time) for studying the effect of pH and dye concentration. To study the effect of dyeing time, dyed samples were examined at intervals up to total dyeing time of 90 min. After dyeing, all dyed samples were rinsed with water and air dried. Dye exhaustion and total dye fixation on wool fabric were evaluated spectrophotometrically. For comparison, dye **1** was applied to all 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 presence of the same dye bath auxiliaries mentioned above.

### Measurements and Testing

#### Dye Exhaustion

Dye uptake of dyes **1** and **2** for wool, polyester and wool/polyester blend fabrics was 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  $\lambda_{\max}$  value using a calibration curve previously obtained using known dye concentrations (g/L) in 50% aqueous DMF. 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.

### Dye Fixation

The dye fixation (%*F*), the percentage of the exhausted dye chemically bound on the wool fabric, was measured by refluxing the wool dyed samples in 50% aqueous DMF (20:1 LR) for 15 min to extract the unfixed dye [12]. This procedure was repeated until the extract was clear. The concentration of the extract was then measured spectrophotometrically at the  $\lambda_{\max}$  value of each dye and the dye fixation ratio 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 extracted dye

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

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

### 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 [13, 14].

### **Fastness Testing**

Dyed wool, polyester and wool/polyester blend samples, after washing-off using 2 g/L nonionic detergent at 80°C for 15 min, 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.

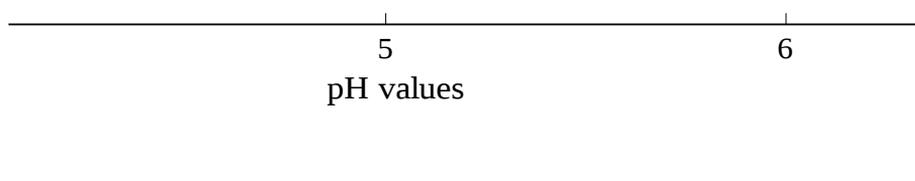
### **Results and Discussion**

#### **Dye characterization**

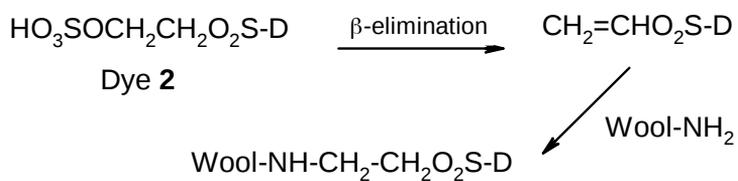
The IR spectra of the synthesized dye **2** showed characteristic absorption bands of the OSO<sub>3</sub>H group. The IR spectra also showed absorption bands at 1142 cm<sup>-1</sup> for the -SO<sub>2</sub>- group. The <sup>1</sup>H-NMR spectrum of the dye was characterized by two triplet peaks at 3.58-3.61ppm (2H) and 3.89-3.92 ppm (2H), assignable to the α- and β-methylene protons of the sulphatoethylsulphone (SES), respectively.

#### **Effect of dyebath pH**

The dyeing properties of dye **2** was initially investigated on wool fabric by varying the pH from 3 to 7. The extent of dye exhaustion and total fixation yield (Figure 1) showed maximum values at neutral pH 7. This is mainly attributed to higher β-elimination reaction of the temporarily anionic SES group in the dye structure which generates a nonionic reactive vinylsulphone (VS) derivative. The formation of the nonionic VS derivative (Scheme 1) increases the substantivity of the modified dye towards wool fabric and, therefore, enhanced the extent of reaction with the nucleophilic sites in wool fabric (Scheme 1). Contrary to wool/dye **2** system, the commercial disperse dye **1** showed very low dye build up on wool fabric at the conventional pH 5.5 (E%; 15.3). A further advantage, the nonionic character of the VS reactive system of dye **2** seems to assist further build-up on wool fabric under neutral conditions as there is no repulsive effect between the nonionic fixed dye in the fabric and the nonionic dye in solution.



**Figure 1 Exhaustion (E) and total fixation (T) values of the modified dye 2 on wool (2% owf, 100°C and various pH values).**

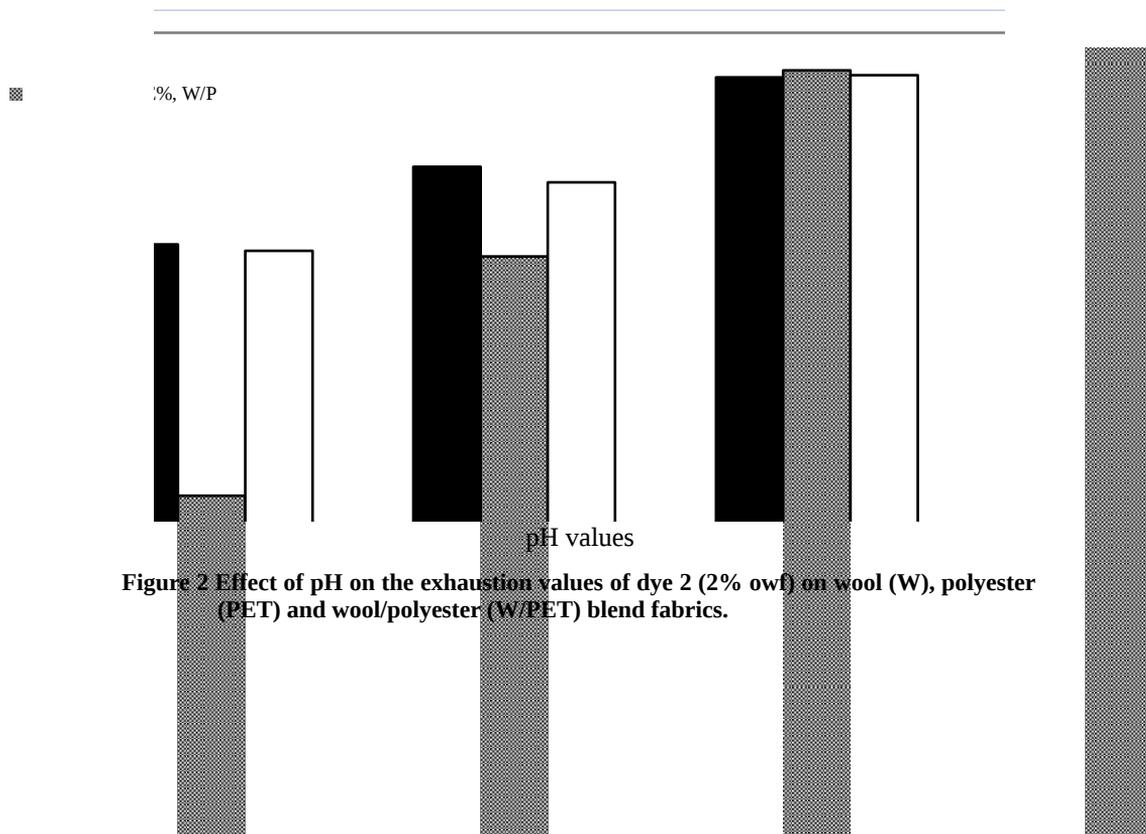


D = Dye chromophore

**Scheme 1**  $\beta$ -elimination reaction of the SES reactive group containing modified dye 2 and their chemical reaction with wool fabric.

Also, it can be recognized that the extent of dye fixation of the reactive VS derivative increases at the dye bath pHs higher than 5. This fact suggests that the  $\beta$ -elimination reaction is promoted as pH increases, resulting in a pronounced increase in the substantivity of the modified dye 2 on wool fabric if compared with the traditional disperse dye structure 1.

Dye 2 is also applied to polyester and wool/polyester blend fabrics and its dyeing exhaustion values at different pH values are shown in Figure 2. The results indicate that dye 2 exhibit lower exhaustion values at  $\text{pH} \leq 4$ . This is probably caused by the relatively hydrophilic character of the unconverted temporarily anionic SES group in dye 2, which can create a difficulty in diffusion of dye molecules into hydrophobic polyester fiber. As the pH increases, the extent of dye exhaustion on polyester and wool/polyester blend fabrics significantly increases (Figure 2). Thus, a better colour yield on polyester and wool/polyester fabrics is detected at neutral pH. As a consequence, the blend dyeings using dye 2 exhibit better results of exhaustion than those of dye 1 (table 1). This reviles the success of using the modified disperse dye 2 for a neutral dyeing of wool and wool/polyester blend fabrics.



**Figure 2** Effect of pH on the exhaustion values of dye 2 (2% owf) on wool (W), polyester (PET) and wool/polyester (W/PET) blend fabrics.

**Effect of dye concentration**

The exhaustion and total fixation yield of dye 2 on wool fabric were examined using different depth of shades (1-5% owf) at pH 7 and 100 °C. The results are given in Figure 3. The higher fixation efficiency of the VS derivative of dye 2 may offer further dye uptake, even at higher dye concentrations. The effect of dye concentration on the extent of dye exhaustion on polyester and wool/polyester blend fabrics is shown in Figure 4. Increasing the dye concentration reduces the exhaustion on polyester and blend fabrics with similar rates on wool dyeing (Figure 3). This observation results in potentially a good dyeing performance of the modified dye 2 on the blend fabric, even at higher dye concentration.

**Figure 3 Exhaustion and total fixation values at different concentrations of dye 2 on wool fabric.**

**Figure 4 Exhaustion values at different concentrations of dye 2 on polyester and wool/polyester blend fabrics.**

#### **Colorimetric and Fastness properties**

The colourimetric CIE L\*a\*b\* C\*h° data of the un-dyed (Blank) and dyed wool, polyester and wool/polyester blend fabrics using both dyes 1 and 2 are shown in Table (1). It can be seen that the chroma (C\*) and hue (h°) values of the dyed wool, polyester and wool/polyester blend fabrics using dye 2 are approximately similar. This indicates that a properly union dyeing of the wool/polyester blend fabric is successfully achieved. Additionally, the exhaustion and K/S values obtained for all dyeings are better to justify the performance of dye 2.

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

<b>Dye</b>	<b>Fabric</b>	<b>%E</b>	<b>K/S</b>	<b>*L</b>	<b>*a</b>	<b>*b</b>	<b>*C</b>	<b>°h</b>
<b>Blank</b>	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	15.3	3.23	77.66	-0.38	37.48	37.48	90.57
	PET	99	27.41	70.83	8.31	78.18	78.62	83.93
	W/PET	26.6	7.46	76.91	1.89	55.19	55.22	88.04
2	W	95.72	30.94	70.06	14.62	77.32	78.69	79.30
	PET	98.18	26.34	70.94	9.02	69.75	70.34	82.63
	W/PET	96.93	27.5	71.40	13.26	74.31	75.48	79.89

As shown in Table 2, the fastness to washing, rubbing and perspiration of all samples dyed with dye 2 were excellent to very good irrespective to the fabric used. The modified dye 2, having high fixation on wool part, showed better fastness results than the commercial dye 1 especially on wool and wool/polyester blend fabrics. Also, for each dyed fabric the light fastness using both dyes 1 and 2 was the same. This seems reasonable as the dyes under investigation have approximately the same chromophoric system.

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

Dye	Fabric	Fastness to rubbing		washfastness			Fastness to Perspiration						Light
							Alkaline			Acidic			
		Dry	Wet	Alt	SC	SW	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
	PET	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
	PET	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

Where: Alt. colour change of dyed sample; SC, staining on cotton; SW staining on wool.

### Conclusion

A SES dye analogue of C.I. Disperse Yellow 16 was synthesized and applied to wool, polyester and wool/polyester fabrics. Maximum exhaustion as well as total fixation efficiency using the SES-based dye on wool fabric was achieved at neutral pH 7. The dye showed better dyeing performance due to its nonionic reactive VS derivative. This feature of the dye structure significantly improve the union dyeing of wool/polyester blend fabrics with very good build up and fastness properties in comparison with C. I. Disperse Yellow 16.

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