

## Synthesis of 1-(2',4'-Dichloro-5'-Fluorophenyl)-3-(3'-Hydroxy-4'-Methoxy-5'-aryl azo)-2-Propene-1-one Disperse Azo Dyes Based on Chalcone and Their Application on Polyester Fibre

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Some new azo disperse dyes 1-(2',4'-dichloro-5'-fluoro phenyl)-3-(3'-hydroxy-4'-methoxy-5'-aryl azo)-2-propene-1-one were synthesized and their dyeing performance on polyester fibre was assessed. These dyes were characterized by elemental analysis, IR spectra and NMR spectra. The fastness properties of these dyes were evaluated by applying them on polyester fibre. The values of  $\lambda_{\text{max}}$ ,  $R_f$ , % exhaustion and dyeing behaviour, such as wash, light and rubbing fastness of prepared compounds, have been studied.

**Key Words:** Disperse azo dyes, Chalcone, Application, Polyester fibre.

### INTRODUCTION

The notable dyeing performance and characteristics of azo disperse dyes<sup>1,2</sup> previously reported<sup>3,4</sup>, dyes based on chalcone<sup>5-9</sup>, have been reported as being useful on polyester fibre<sup>10</sup>. These dyes have excellent dyeing properties<sup>11-13</sup> including low sublimation and high thermal stability. All the dyes possess brilliance of shade and good overall dyeing properties on polyester fibre. Encouraged by these properties, we report here the synthesis of azo disperse dyes based on chalcone.

### EXPERIMENTAL

Melting points were taken in open capillaries and are uncorrected. IR spectra were recorded on Perkin-Elmer-377 spectrometer and NMR spectra were recorded on Bruker Avance DPX 200 MHz spectrometer. Elemental analyzes were recorded on Carlo-Erba-1108 analyzer. The absorption spectra were recorded on Hitachi (model U-320) spectrometer in DMF solution at 28°C using  $2 \times 10^{-3}$  m dye concentration. The physical and analytical data are given in Table-1.  $R_f$  values were determined by benzene : propenol (9 : 1) as solvent. A glycerine bath and laboratory high temperature beaker dyeing machine were used for dyeing on polyester fibre.

#### **Preparation of 1-(2',4'-dichloro,-5'-fluoro phenyl)-3-(3'-hydroxy-4'-methoxy phenyl)-2-propene-1-one (chalcone)**

2,4-Dichloro acetophenone (0.01 mole) and 3-hydroxy-4-methoxy benzalde-

hyde (0.01 mole) in ethanolic solution of KOH were stirred for 3 h, then kept at room temperature for 24 h. The reaction mixture was diluted with ice-cold water, acidified with dilute HCl and extracted with diethyl ether. The ethereal layer was washed with water, dried over anhydrous  $\text{Na}_2\text{SO}_4$  and evaporated to dryness. The crude product was recrystallized in alcohol, m.p.  $120^\circ\text{C}$ . The purity of the compound was checked on TLC by using benzene : acetone (9 : 1) as mobile phase.

$\nu_{\text{max}}$  ( $\text{cm}^{-1}$ ) (KBr): 1150  $\nu(\text{C}-\text{F})$ , 1055  $\nu(\text{O}-\text{CH}_3)$ , 940  $\nu(-\text{CH}=\text{CH}-)$ , 730  $\nu(\text{C}-\text{Cl})$ .

TABLE-1

Dye No.	R	m.p. ( $^\circ\text{C}$ )	Yield (%)	m.f.	$\lambda_{\text{max}}$ (nm)	Elemental analysis %		
						Found (Calcd.)		
						C	H	N
1.	Phenyl	95	70	$\text{C}_{22}\text{H}_{15}\text{N}_2\text{O}_3\text{Cl}_2\text{F}$	520	59.30 (59.32)	3.37 (3.37)	6.26 (6.29)
2.	4-Nitro phenyl	> 300	72	$\text{C}_{22}\text{H}_{14}\text{N}_3\text{O}_5\text{Cl}_2\text{F}$	530	53.85 (53.87)	2.83 (2.85)	8.55 (8.57)
3.	4- $\text{SO}_3\text{H}$ phenyl	> 300	75	$\text{C}_{22}\text{H}_{15}\text{N}_2\text{O}_6\text{SCl}_2\text{F}$	525	50.29 (50.28)	2.83 (2.85)	5.32 (5.33)
4.	4-Methoxy phenyl	99	74	$\text{C}_{28}\text{H}_{19}\text{N}_2\text{O}_4\text{Cl}_2\text{F}$	520	62.56 (62.56)	3.52 (3.53)	5.20 (5.21)
5.	3,4-Dimethyl phenyl	105	73	$\text{C}_{24}\text{H}_{19}\text{N}_2\text{O}_3\text{Cl}_2\text{F}$	525	60.85 (60.88)	4.00 (4.01)	5.90 (5.91)
6.	3-Nitro phenyl	> 300	71	$\text{C}_{22}\text{H}_{14}\text{N}_3\text{O}_5\text{Cl}_2\text{F}$	520	53.86 (53.87)	2.85 (2.85)	8.56 (8.57)
7.	4-Methyl phenyl	105	70	$\text{C}_{23}\text{H}_{17}\text{N}_2\text{O}_3\text{Cl}_2\text{F}$	515	60.11 (60.13)	3.70 (3.70)	6.09 (6.10)
8.	4-Ethyl phenyl	115	72	$\text{C}_{24}\text{H}_{19}\text{N}_2\text{O}_3\text{Cl}_2\text{F}$	520	60.85 (60.88)	4.00 (4.01)	5.90 (5.91)
9.	4-Nitro-2-methyl phenyl	109	74	$\text{C}_{23}\text{H}_{16}\text{N}_3\text{O}_5\text{Cl}_2\text{F}$	550	54.75 (54.76)	3.15 (3.17)	8.33 (8.33)
10.	2-Nitro-4-methyl phenyl	110	73	$\text{C}_{23}\text{H}_{16}\text{N}_3\text{O}_5\text{Cl}_2\text{F}$	545	54.74 (54.76)	3.17 (3.17)	8.30 (8.33)
11.	4-Chloro-2-nitro phenyl	112	70	$\text{C}_{22}\text{H}_{13}\text{N}_3\text{O}_5\text{Cl}_3\text{F}$	540	50.32 (50.33)	2.45 (2.47)	8.03 (8.00)
12.	6-Nitro-3,4-dichloro phenyl	> 300	72	$\text{C}_{22}\text{H}_{12}\text{N}_3\text{O}_5\text{Cl}_4\text{F}$	545	47.20 (47.22)	2.11 (2.14)	7.50 (7.51)
13.	6-Chloro-2,4-dinitro phenyl	119	74	$\text{C}_{22}\text{H}_{12}\text{N}_4\text{O}_7\text{Cl}_3\text{F}$	550	46.32 (46.35)	2.09 (2.10)	9.83 (9.83)
14.	6-Bromo-2-cyano-4-nitro phenyl	> 300	70	$\text{C}_{23}\text{H}_{11}\text{N}_4\text{O}_5\text{Cl}_2\text{BrF}$	545	47.34 (47.34)	2.05 (2.05)	9.60 (9.60)
15.	6-Bromo-2,5-dichloro-4-nitro phenyl	> 300	73	$\text{C}_{22}\text{H}_{11}\text{N}_3\text{O}_5\text{Cl}_4\text{BrF}$	550	42.09 (42.10)	1.73 (1.75)	6.68 (6.69)

### General procedure of diazotization of different aryl amines

Different aryl amines (0.01 mole) were diazotized in the usual manner<sup>14, 15</sup> by using HCl/H<sub>2</sub>SO<sub>4</sub> and NaNO<sub>2</sub>. These diazo components were used in the following reaction:

#### Preparation of 1-(2',4'-dichloro-5'-fluoro-phenyl)-3-(3'-hydroxy-4'-methoxy-5'-aryl azo)-2-propene-1-one

Various aryl azo amines (0.01 mole) were coupled<sup>16</sup> separately with 1-(2',4'-dichloro-5'-fluoro phenyl)-3-(3'-hydroxy-4'-methoxy phenyl)-2-propene-1-one (0.01 mole) at 0–5°C and pH 8 to obtain 10 dyes. All the dyes were recrystallized from suitable solvent.

$\nu_{\max}$  (cm<sup>-1</sup>) (KBr): 3500  $\nu$ (—OH); 1600  $\nu$ (—N=N); 1150  $\nu$ (C—F); 1060  $\nu$ (O—CH<sub>3</sub> stretching in ether); 940  $\nu$ (—CH=CH); 750–700  $\nu$ (C—Cl). NMR (CDCl<sub>3</sub>): 1.3 (s, 3H, —CH<sub>3</sub>); 3.89 (s, 3H, —OCH<sub>3</sub>); 6.4–7.7 (m, 8H, Ar—H); 8.05 (d, 2H, olefinic proton).

#### Dyeing and fastness properties of dyes

These types of dyes gave generally shades ranging from yellow to orange with good levelness, brightness and depth on polyester fibre. It was observed that the dyes containing electron donating groups like —OH increase the light-fastness.

The wash-fastness and rubbing fastness also appeared to be good to excellent. The percentage exhaustion of the dyes shows that the good fastness properties were carried out by the known method. R<sub>f</sub> value and fastness properties are given in Table-2.

TABLE-2

Dye No.	R <sub>f</sub> value	Exhaustion (%)	Fastness properties			
			Light	Washing	Rubbing	
					Dry	Wet
1.	0.78	69	4	4	4	4
2.	0.80	71	5	4	5	5
3.	0.79	70	4	4	4	4
4.	0.83	72	5	5	5	5
5.	0.81	68	6	5	6	6
6.	0.82	73	5	4	5	5
7.	0.82	72	5	5	4	4
8.	0.83	71	4	4	5	5
9.	0.84	74	5	5	5	5
10.	0.84	73	6	5	5	5
11.	0.82	72	5	4	5	5
12.	0.80	68	4	5	4	4
13.	0.79	69	5	5	5	5
14.	0.78	70	5	4	4	4
15.	0.79	68	5	5	5	5

### Application of dyes on polyester fabrics in 2% shade

A glycerine bath and laboratory high temperature beaker dyeing machine were used.

#### Preparation of dye bath

A paste of weighed quantity of dye (40 mg) was prepared with dispersing agent Dadamol (40 mg), wetting agent Tween-80 (5 mg) and water (1 mL). To it water (99 mL) was added with stirring and pH adjusted to 4 by acetic acid.

#### Dyeing process

The dye solution (10 mL) was added to a beaker provided with lid and screw cap. Before closing the lid and tightening the metal cap over the beaker, wetted pattern of polyester was rolled properly and dropped into the beaker. The beaker was then placed vertically on the rotatory carrier inside the tank and clamp plate was firmly tightened by the nut of the stud. The rotatory carrier was allowed to rotate in the glycerine bath at a temperature of 130°C and continued for 60 min under pressure. After cooling over a period of 1 h, the beaker was removed from the bath and washed with water. The pattern was washed several times with cold water.

The above dyed pattern was further treated with a solution of the detergent (0.2 g) and Na<sub>2</sub>CO<sub>3</sub> (0.1 g) in 100 mL water at 800°C for 30 min. After washing the pattern thoroughly with water, it was dried.

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