

NOTE

Disperse Dyes: Synthesis of 2,4'-Dichloro Diphenyl Ether 4-(Aryl Azo) Dyes and Their Application to Polyester Fabric

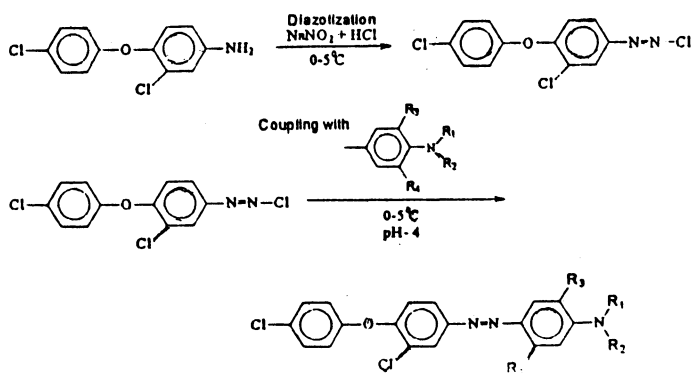
K.R. DESAI* and C.K. DESAI

Department of Chemistry, South Gujarat University
Surat-395 007, India

Attempts have been made to prepare disperse dyes based on aryl ether. 4-Amino-2,4'-dichloro diphenyl ether was diazotized and coupled with various diazo components. These compounds were evaluated for the dyeing properties on polyester fibre. The structures of these compounds have been confirmed by elemental analysis and IR spectral studies.

The literature survey of the disperse dyes based on diphenyl amino ether reveals that these dyes possess good dyeing and fastness property. Hence, it was thought interesting to undertake the synthesis of disperse dyes based on 4-amino-2,4-dichloro diphenyl ether.

All the melting points were determined by open capillary method and are not corrected. IR spectra were recorded in KBr pellets on FTIR-8101 spectrophotometer. The purity of the compound in addition to elemental analysis was checked by TLC.



Preparation of 4-amino-2,4'-dichloro diphenyl ether

4-Amino-2,4'-dichlorodiphenyl ether was prepared by reported procedure^{1,2}

Diazotization of 4-amino-2,4'-dichloro diphenyl ether

4-Amino dichloro diphenyl ether (2.54 g, 0.01 mole) was dissolved in hydrochloric acid (36%, 20 mL) with stirring and the solution was cooled to 0–5°C in an ice-bath. A solution of sodium nitrite (0.8 g, 0.012 mole) in 5 mL water to 0°C was then added and the reaction mixture was stirred until positive test for nitrous acid on starch iodide paper (*i.e.*, blue colour on S.I. paper), and the mixture was filtered to get clear diazonium salt for subsequent coupling reaction.

Synthesis of 2,4'-dichloro diphenyl ether 4-(aryl azo) dyes

The various coupling components, as for example N-N-bis-hydroxyethyl-m-chloroaniline [2.15 g, 0.01 mole] were dissolved in 15 mL hydrochloric acid and the solution was cooled to 0–5°C. To this wellstirred solution, the above diazonium solution was added slowly so that temperature did not rise above 5°C, while maintaining the pH at 4–5 by the addition of sodium acetate solution [10% w/v]. The mixture was then stirred for 3 h at 0–5°C. After completion of the reaction, the solid material was filtered, washed, dried and crystallized from DMF to get red crystals.

The yield, melting point and elemental analysis of disperse dyes are reported in Table-1. The dyes varied in hue from golden orange to reddish orange to lemon yellow colour depending on the nature of the diazo component used and their absorption maxima are reported in Table-1. The dyes were applied to polyester fabric. The dyes gave generally satisfactory dyeing on polyester fibres. Fastness to washing varied from moderate to good and the lightfastness was better on polyester fibres.

TABLE-1
PHYSICAL DATA OF DISPERSE DYES

R-Component				m.p. (°C)	λ_{\max}	R _f	Colour on polyester fabric
—R ₁	—R ₂	—R ₃	—R ₄				
CH ₂ CH ₃	CH ₂ CH ₃	H	NHCOCH ₃	111	478	0.37	Lemon yellow
CH ₂ CH ₂ OH	CH ₂ CH ₂ OH	CH ₃	H	110	477	0.61	Deep orange
CH ₂ CH ₃	CH ₂ CH ₃	OCH ₃	NHCOCH ₃	120	476	0.47	Lemon yellow
CH ₂ CH ₂ CN	CH ₂ CH ₃	H	H	125	478	0.65	Golden orange
CH ₂ CH ₂ OCOCH ₃	CH ₂ CH ₂ OCOCH ₃	Cl	H	260	476	0.30	Golden orange
CH ₂ CH ₂ OH	CH ₂ CH ₂ OH	Cl	H	200	423	0.33	Golden yellow
CH ₂ CH ₂ OCOCH ₃	CH ₂ CH ₂ OCOCH ₃	H	NHCOCH ₃	201	448	0.45	Light orange
CH ₂ CH ₂ CN	CH ₂ CH ₂ CN	H	H	150	406	0.50	Deep orange
CH ₂ CH ₂ OCOCH ₃	CH ₂ CH ₂ OCOCH ₃	OCH ₃	NHCOCH ₃	160	443	0.65	Reddish yellow
CH ₂ CH ₂ OCOCH ₃	CH ₂ CH ₂ OCOCH ₃	CH ₃	H	155	477	0.80	Golden yellow
CH ₂ CH ₂ OH	CH ₂ CH ₂ OH	H	NHCOCH ₃	170	470	0.81	Reddish orange
CH ₂ CH ₂ CN	CH ₂ CH ₂ OH	H	H	180	469	0.82	Lemon yellow

IR spectroscopy: IR spectra of 2,4'-dichloro diphenyl ether 4-(aryl azo) dyes were recorded. 2-4'-dichloro diphenyl ether 4-(aryl azo) dyes were separated on TLC using chloroform-methanol (95 : 5) solvent system and using silica gel G as absorbent. IR (KBr): 1230–1000 cm^{-1} (ethereal linkage), 1630–1575 cm^{-1} $\nu(\text{N}=\text{N})$, 732–725 cm^{-1} $\nu(\text{C}-\text{Cl})$, 3420–3280 cm^{-1} $\nu(\text{OH})$.

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