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Heterocyclic Monoazo Dyes Derived from 2-Amino Benzoxazole and Their Application on Polyester Fibre

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Nine azo disperse dyes of the type 2-arylazo benzoxazole have been synthsised by using 2-hydroxy phenylthiourea, lead oxide, then after diazotisation and coupling with various substituted amino benzene derivatives. Dyeing properties of these dyes on polyester fibre were assessed. These dyes were characterised by elemental analysis and IR spectra.

INTRODUCTION

Disperse dyes have been applied to a large number and variety of fibres, such as cellulose acetate and triacetate, polyamide and polyester fibres. Disperse dyes have therefore achieved an importance which is probably greater than the weights used suggest. They were the first type to be applied directly to a fibre from an insoluble suspension. Their preparation includes a process of particle-size reduction and often drying in the presence of a dispersing agent.

Benzoxazoles are of interest due to use as intermediates for the synthesis of the bacteriological and pharmacological studies,[1–4] photographic color images[5] and in copper electroplating.[6] They are also very useful as fluorescent whitening agents[7] and in application in dyes. In the present study, we report the synthesis of some mono azo dyes derived from 2-amino benzoxazole derivatives, spectral characteristics and dyeing properties of the prepared dyes also discussed.

EXPERIMENTAL

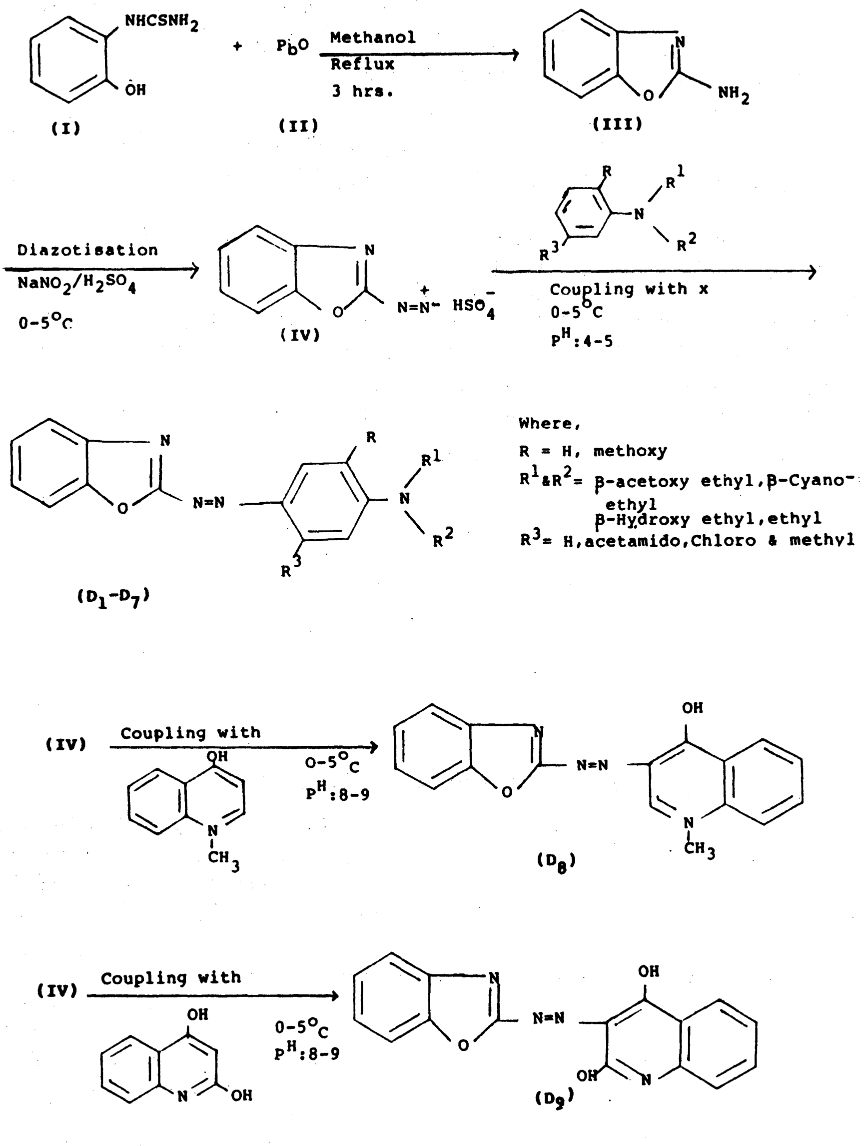
All the melting points were determined in open capillaries and are uncorrected. IR spectra (in KBr disc) were recorded on a Perkin-Elmer 377 Spectrophotometer; elemental analysis was done on Carlo Erba 1108 analyser; λ_{max} was determined in DMF at 28°C using Bausch and Lomb spectronic-20 at 2×10^{-3} M dye conc. R_f values are determined on DMF-chloroform (2 : 5) solvent system. Fastness tests were assessed by the standard method of testing (BS:1006–1978 and IS: 765–1979).

Preparation of 2-hydroxy Phenylthiourea(I)

The title compound was prepared by the reported method[8–10].

Preparation of 2-amino benzoxazole (III)

A mixture of 2-hydroxy phenylthiourea(I) (1.68 gm, 0.01 mole) and yellow lead oxide (2.23 g., 0.01 mole) is refluxed in 100 mL of methanol with stirring for 3 h The PbS formed is filtered off together with excess of lead oxide and washed with methanol and the filtrate is distilled *in vacuo*. Recrystallization of the residue from benzene gave white crystals of (III) yield 68%; m.p. 132°C.



Scheme-I

Diazotisation of 2-amino benzoxazole (IV)

2-Amino benzoxazole(III) (1.34 g, 0.01 mole) was diazotised in the usual manner. The resulting diazo solution was used for subsequent coupling reaction.

Preparation of 2-Substituted 4″-N,N-dialkylamino phenyl azo benzoxazole (D_1—D_7)

m-Chloro-N,N-bis-β-hydroxy ethyl aniline (X) (2.2 g; 0.01 mole) was dissolved in hydrochloric acid (1.8 ml, 0.05 mole).

solution, the above mentioned diazo solution was added maintaining the pH at 5–6 by the addition of sodium carbonate solution (10% w/v). The stirring was continued for 3 h at the same temperature and precipitated solid was then filtered, washed with water, dried and recrystallised from DMF-chloroform to give dye D_1; yield 72%; m.p. 216°C.

The same procedure was used to prepare dye D_2 to D_7.

TABLE-1
YIELD, MELTING POINTS AND ELEMENTAL ANALYSIS OF MONOAZO DISPERSE DYES.

Dye No.	Coupling component (X)				Yield (%)	m.p. (°C)	Nitrogen (%)	
	R	R^1	R^2	R^3			Found	Calcd.
D_1	H	CH_2CH_2OH	CH_2CH_2OH	Cl	72	216	15.54	15.51
D_2	H	$CH_2CH_2OCOCH_3$	$CH_2CH_2OCOCH_3$	Cl	58	222	12.56	12.58
D_3	H	CH_2CH_2OH	CH_2CH_2OH	CH_3	59	254	16.39	16.42
D_4	H	CH_2CH_3	CH_2CH_3	$NHCOCH_2CH_3$	76	228	19.90	19.94
D_5	H	CH_2CH_2CN	$CH_2CH_2OCOCH_3$	H	80	204	18.60	18.56
D_6	OCH_3	$CH_2CH_2OCOCH_3$	$CH_2CH_2OCOCH_3$	$NHCOCH_3$	84	300	14.27	14.25
D_7	H	CH_2CH_2CN	CH_2CH_3	H	75	212	21.90	21.94
D_8	1-Methyl-4-hydroxy quinoline				76	236	22.91	22.95
D_9	2,4-Dihydroxy quinoline				62	282	22.89	22.87

Preparation of 2-(1′-methyl-4′-hydroxy quinoline-3′-yl)azo benzoxazole

The diazo solution was added to a clear solution of 1-methyl-4-hydroxy quinoline (1.60 g; 0.01 mole) in dilute sodium carbonate solution (10 mL) and the temperature was maintained between 0–5°C. The pH was maintained between 8–9 by the addition of sodium carbonate solution (10% w/v). The mixture was stirred for 3 h at the same temperature. The yellowish-orange dye (D_8) so obtained was filtered, washed with water, dried and purified from DMF-chloroform solution. Yield 76%, m.p. 258°C.

The same procedure was followed for the preparation of the dye (D_9), 2-(2′,4′-dihydroxy quinoline-3′-yl) azo benzoxazole.

Dyeing procedure

The dyeing of the polyester fabric samples were carried out by HTHP dyeing method.[11]

RESULTS AND DISCUSSION

Structures of the new synthetic azo disperse dyes were characterised by means of elemental analysis and IR spectra. Compound shows C═N stretching vibration near 1495–1485 cm^{-1}; C—O—C stretching at 1300–1285 cm^{-1}; —N═N— strectching 1610–1590 cm^{-1}; C—Cl strecthing vitration at 770 cm^{-1}; O—H stretching at 3640–3300 cm^{-1}. All the dyes have good substantivity for polyester fibre. Table-2 shows moderate to fairly good light-fastness. However, compounds D_5 and D_7 shows somewhat better light fastness. This may be due to the presence of an additional acceptor substituent such as cyano group, which results into increasing electron mobility in these compounds and thus into good light-fastness. The wash-fastness of all the compounds, as may be anticipated on the basis of their increased molecular weight and polarity to less substituted analogues, was also of an acceptably very good order. No notable change is observed in the percentage exhaustion by introducing β-hydroxyethyl group at the terminal amino group for better dispersibility. Overall, the prepared dyes gave generally satisfactory dyeing on polyester fibre.

TABLE-2

λ_{max}, % EXHAUSTION, SHADE AND FASTNESS PROPERTIES OF THE PREPARED COMPOUNDS

Dye No.	Shade on polyester	λ_{max} (mm)	R_f (value)	% Exhaustion	Fastness to		ubbing	
					Light	Washing	Dry	Wet
D_1	Pale cream	440	0.83	48	4	4–5	5	5
D_2	Pink	520	0.75	54	4	4	5	5
D_3	Orange	492	0.72	50	3	4	5	5
D_4	Orange	495	0.87	50	3	4	4	4
D_5	Golden yellow	464	0.85	52	5	4	4	4
D_6	Pastel red	462	0.72	46	3	4	4	4
D_7	Pale yellow	425	0.80	53	5	3	5	5
D_8	Lemon yellow	470	0.82	46	4	4–5	5	5
D_9	Lemon yellow	472	0.76	48	4	4	5	5

Light-fastness: 1—minimum, 2—Poor, 3—moderate, 4—fairly good, 5—good, 6—very good, 7—Excellent, and 8—maximum.

Washing and rubbing fastness: 1—poor, 2—fair, 3—good, 4—very good, and 5—excellent.

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