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Synthesis of Quinoline Based Bisazo Disperse Dyes and Their Dyeing Performance on Polyester Fabric

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Eleven N-(3-hydroxy-4′-R-azobenzene-1′-sulfonamidoazobenzene)-8-quinoline sulfonamides (**IVa–k**) were prepared by coupling diazotized N-(3-hydroxy-4′-aminobenzene-sulfonamidoazobenzene)-8-quinoline sulfon amide (**III**) with various coupling components (**R_{a-k}**). The compound (**I**) was coupled with diazotized 4-amino-4′-acetylaminodiphenylsulfonamide giving compound (**II**) which on hydrolysis gave compound (**III**). The compounds (**IVa–k**) were characterized by elemental and spectral analyzes and their dyeing performance on polyester fabric was assessed.

Key Words: Quinoline based bisazo disperse dyes, Dyeing.

INTRODUCTION

Several diazo dyes have been proved useful for the colouration of cellulose acetate fibres. It comprises of almost 10% of total disperse dyes produced commercially. The quinoline based dyes using quinoline as coupling component[1,2] and as diazo component[3,4] have been reported. In view of this some new **(III)** based bisazo disperse dyes have been synthesized and their dyeing preformance on polyester fabric assessed.

EXPERIMENTAL

8-Quinoline sulfonyl chloride: It was prepared by the method already reported.[3]

N-(3-Hydroxyphenyl)-8-quinoline sulfonamide (I): It was prepared by the known method.[4]

N-(3-Hydroxy-4′-acetylaminobenzene-1′-sulfonamidoazobenzene)-8-quinoline sulfonamide (II)

A clear solution of **I** (3.0 g, 0.01 mole) in sodium hydroxide (30 mL, 10%) was cooled below 5°C. A clear solution of diazotized 4-amino-4′-acetylamino-diphenyl sulfonamide (3.05 g, 0.01 mole) was slowly added to the above cold solution with constant stirring maintaining the temperature 0–5°C and pH between 7.5–8.0. The coupled mass was stirred further at 0–5°C for 2 h. The reaction mixture was diluted with water and the pH was just adjusted between 6.5 and 7.0 with acetic acid. The brown dye thus separated was filtered, washed with water and crystallized from alcohol; yield 72%, m.p. 188°C (d). [Found: N, 13.48 %; $C_{29}H_{24}N_6O_6S_2$ required: N, 13.61 %]. IR spectra (KBr): 3427 ν(—OH and —NH stretching), 1676 ν(C=O stretching), 1240 ν(C—O stretching), 789 ν(C—S stretching), 1498 ν(—N=N— stretching) and 1159, 1375 cm^{-1} ν(S=O stretching). NMR spectra (DMSO-d_6 solvent): δ 7.74 (1H, —OH), δ 8.39 (1H, $—SO_2NH$), δ 2.02 (1H, $NHCOCH_3$) and δ 2.49 (3H, $—COCH_3$).

N-(3-Hydroxy-4′-aminobenzene-sulfonamidoazobenzene)-8-quinoline sulfonamide (III)

The mixture of **II** (10 g), sodium hydroxide (10 mL, 10%) and distilled water (25 mL) was first heated on water-bath for 30 min and then directly on wire gauze for 1 h. The clear solution was cooled and neutralized with hydrochloric acid. The product was filtered, washed with water and crystallized from DMF; yield 84%, m.p. 118°C (d). [Found: N, 14.46%; $C_{27}H_{22}N_6O_5S_2$ required: N, 14.63%]. IR spectra (KBr): 3383 ν(—OH stretching), 3250 ν(—NH stretching), 1238 ν(C—O stretching), 789 ν(C—S stretching), 1500 ν(—N=N— stretching), 1148, 1398 ν(S=O stretching) and 1319 cm^{-1} ν(C—N stretching). NMR spectra (DMSO-d_6 solvent): δ 7.32 (1H, —OH), δ 8.39 (1H, —SO_2NH), δ 3.34 (2H, NH_2).

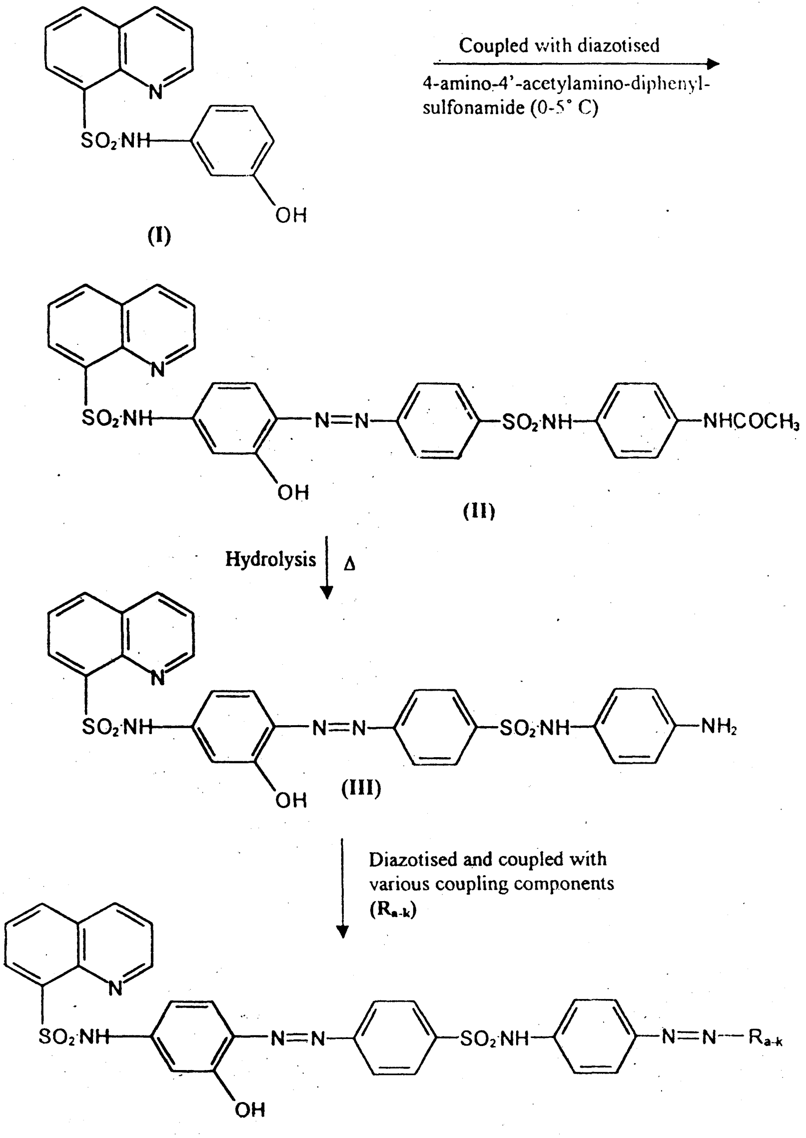
N-(3-Hydroxy-4′-R-azobenzene-1′-sulfonamidoazobenzene)-8-quinoline sulfonamide (IVa–k)

A clear solution of *bis* (acetoxyethyl-3-aminoacetanilide) (0.805 g, 0.0025 mole) in hydrochloric acid (2 mL, 50%) was cooled below 5°C. To this well-stirred solution the diazo solution of **III** (1.435 g, 0.0025 mole) was added over a period of 5 min, maintaining the temperature below 5°C. The stirring was continued for 2 h at 0–5°C. The reaction mixture was diluted with water and crystallized from DMF-methanol mixed solvent; yield 62%, m.p. 148°C. The same procedure was used to prepare **(IVb–k)** using various coupling components **Ra–k (Scheme-1)**. The characterization data of compounds **IVa–k** are given in Table-1.

TABLE-1
CHARACTERIZATION DATA OF COMPOUNDS **(IVa–k)**

Compd.	Colours	Yield (%)	λ_{max} (nm)	R_f Value	Nitrogen(%)	
					Found	Required
IVa	Dark brown	62	388	0.87	14.50	14.66
b	Light brown	63	388	0.90	13.29	13.44
c	Yellowish brown	60	399	0.85	13.81	13.97
d	Dark brown	62	392	0.80	15.69	15.86
e	Brown	61	385	0.75	15.24	15.42
f	Brown	59	386	0.79	16.06	16.25
g	Dark brown	63	330	0.73	16.35	16.60
h	Dark brown	70	383	0.70	15.64	15.86
i	Brown	69	395	0.78	17.05	17.23
j	Brownish black	64	384	0.80	13.00	13.17
k	Yellowish orange	71	410	0.87	15.18	15.35

The structures of **IVa–k** were confirmed by their elemental analysis, IR and NMR spectra. The IR spectrum of **IVk** shows characteristic bands at 3431 ν(—OH), 788 ν(C—S), 686 ν(C—Cl), 1240 cm^{-1} ν(C—O). The NMR spectrum of **IVk** shows characteristic signals at δ 7.49 (1H, —OH) and 8.37 (1H, —SO_2NH).



Scheme-1: Synthesis of N-(3-hydroxy-4'-R-azobenzene-1'-sulfonamidoazobenzene)-8-quinolinesulfonamide (**IVa–k**)

For compounds **IVa–k**

Ra	*Bis*(acetoxyethyl-3'-aminoacetanilide)	**g**	N-Cyanoethyl-N-ethylaniline
b	2'-Methoxy-5-acetylamino-N,N-*bis*(acetoxyethylaniline)	**h**	N-Ethylaniline
c	Dioxyethyl-3-chloroaniline	**i**	N-Cyanoethylaniline
d	3-N,N-diethylaminoacetanilide	**j**	Diacetoxy ethylaniline
e	N-cyanoethyl-N-2-acetoxyethylaniline	**k**	4-Chloro-2-aminophenol
f	N-Cyanoethyl-N-hydroxy ethylaniline		

TABLE-2
SHADES ON POLYESTER, % EXHAUSTION AND FASTNESS PROPERTIES OF **(IVa–k)**

Compd.	Colour on polyester	Exhaustion (%)	Light-fastness	Wash-fastness
IVa	Yellow	71	3–4	4
b	Yellow	71	3	4
c	Yellow	62	2–3	4–5
d	Light orange	78	2–3	5
e	Light brown	74	4	4–5
f	Dark brown	63	3–4	5
g	Yellow	71	2	4–5
h	Yellow	75	2–3	4–5
i	Yellow	69	3	4
j	Yellow	69	4–5	4
k	Light brown	81	3	5

RESULTS AND DISCUSSION

These compounds, when applied on polyester fabric as 2 per cent shade, gave brown, yellow and orange shades with poor to fairly good light-fastness, good to excellent wash-fastness and poor to excellent exhaustion.

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