

Synthesis and Dyeing Performance of Acid Dyes Based on N-[3-Hydroxyphenyl]-8-Quinoline Sulphonamide System on Various Fabrics

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N-(3-Hydroxyphenyl)-8-quinoline sulphonamide (II), used as a coupling component, was prepared by the chlorosulfonation of quinoline giving 8-quinoline sulfonylchloride (I) followed by condensation with 3-aminophenol. Fifteen N-(3-hydroxyphenyl-4-R-azo)-8-quinoline sulphonamide acid dyes (IVa-o) were prepared by coupling (II) with various diazo components (IIIa-o). The compounds (IVa-o) were characterized by elemental and spectral analysis and their dyeing performance on silk, wool and nylon fabrics was assessed.

Key Words: Synthesis, Dyeing, Acid dyes, N-[3-Hydroxyphenyl]-8-quinoline sulphonamide.

INTRODUCTION

The heterocyclic dyes using quinoline¹⁻³ and quinolinoquinazoline⁴ moieties have been very recently reported. In view of this, it was thought interesting to undertake the synthesis and study of dyeing properties of the monoazo acid dyes (IVa-o).

EXPERIMENTAL

8-Quinoline sulfonylchloride (I): Freshly distilled quinoline (12.9 g, 0.01 mole) was added over a period of 1 h, to previously cooled chlorosulfonic acid (58.25 g, 0.05 mole); during the addition of chlorosulfonic acid the temperature was maintained at 0-5°C. The resultant mixture was then heated to 140°C slowly and maintained at 140 ± 2°C for 3-5 h. The mixture was cooled to room temperature, poured over ice, neutralized with sodium carbonate and finally extracted with chloroform. The chloroform extract was evaporated to dryness and the residue dried further over P₂O₅. The product (I), yield 49.2%, m.p. 124-127°C, was immediately used up in the next stage.⁵ [Found: N, 6.02 %; C₉H₆NO₂SCI required N, 6.15%]. IR (KBr): 683 v(C-S stretching), 1375, 1192 v(S=O stretching), 3009 cm⁻¹ v(C-H stretching, aromatic).

N-(3-Hydroxyphenyl)-8-quinoline sulphonamide (II): The product (I) was then immediately added to previously cooled (25°C) acetone and condensed with 3-aminophenol (5.42 g, 0.05 mole) in presence of trace of pyridine as a catalyst. Due to exothermic nature of the reaction the temperature was found to rise up to

38–40°C. The reaction mixture was further stirred at 38–40°C for 2 h for the completion of the reaction, checked by TLC. A dark bluish green solution was poured in ice-cold water at 10–15°C. A light grey product thus obtained was filtered, washed with water and treated with hydrochloric acid (10%). The product was filtered, washed with water and crystallized from methanol; yield 45 %, m.p. 178°C. [Found: N, 9.14%; C₁₅H₁₂N₂O₃S required N, 9.33%]. IR (KBr): 3468, 3238 ν (—OH, —NH stretching), 1217 ν (C—O stretching), 695 ν (C—S stretching), 2926 (C—H stretching, aromatic), 1315, 1165 ν (S=O stretching), 1566 cm^{-1} ν (N—H bending). NMR spectra (DMSO-d₆ solvent): δ 7.694 (1H, —OH), 8.479 (1H, —SO₂NH).

N-(3-Hydroxyphenyl-4-R-azo)-8-quinoline sulfonamide (IVa–o): A clear solution of II (1.5 g, 0.005 mole) in sodium hydroxide (15 mL, 10%) was cooled below 5°C. A clear solution of diazotized IV_a (0.005 mole) was slowly added to the above solution with stirring at 0–5°C during 1 h, maintaining the pH between 7.5 and 8.0. To this reaction mixture 10% sodium chloride solution was added until the coupled material was precipitated. After stirring for 1 h, the liquor was filtered and the product was washed with a small amount of sodium chloride (5% w/v). The product was dried at 80–90°C and extracted with DMF and precipitated by diluting the DMF extract with excess of acetone. The same procedure was used to prepare IVb–o using various diazo components IIIb–o (Scheme I). The characterization data of compound IVa–o are given in Table-1.

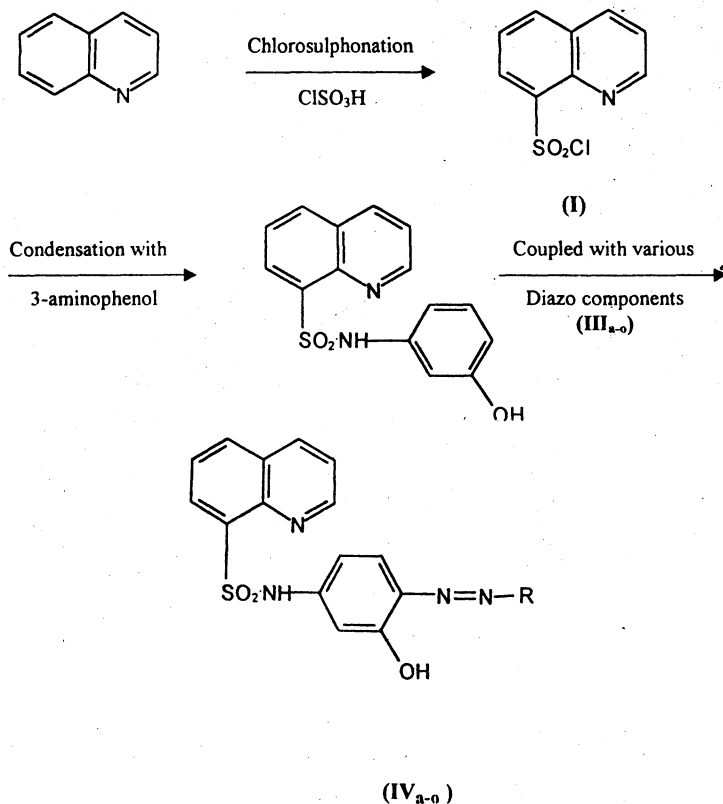
TABLE-1:
CHARACTERIZATION DATA OF COMPOUNDS (IVa–o)

Compd. ^a	Colours	Yield (%)	m.p. ^b (°C)	R _f Value	λ_{max} (nm)	Nitrogen (%)	
						Found	Calculated
IVa	Brown	84	> 300	0.85	490	8.38	8.58
b	Brownish yellow	88	> 300	0.85	450	10.83	11.06
c	Dark brown	82	> 300	0.76	400	12.24	12.43
d	Dark brown	69	> 300	0.78	470	12.53	12.70
e	Dark brown	65	> 300	0.68	470	9.48	9.54
f	Dark brown	68	> 300	0.69	450	12.95	13.08
g	Yellowish brown	73	> 300	0.76	480	10.20	10.40
h	Light brown	79	> 300	0.90	400	9.90	10.07
i	Orange brown	72	> 300	0.83	450	8.44	8.58
j	Reddish brown	71	> 300	0.85	400	7.71	7.82
k	Brown	78	> 300	0.89	450	9.62	9.78
l	Dark brown	70	> 300	0.79	500	7.65	7.82
m	Brown	83	> 300	0.88	470	10.90	11.06
n	Brown	79	> 300	0.79	500	8.39	8.58
o	Brown	80	> 300	0.82	400	9.67	9.79

^aAll compounds gave satisfactory elemental analysis for C and H.

^bMelting points are uncorrected.

λ_{max} was determined at 20°C and 2×10^{-3} M concentration.



Scheme: Synthesis of N-(3-hydroxyphenyl-4-R-azo) 8-quinoline sulphonamides (IV_{a-o})
For compounds IV_{a-o}

- | | |
|---|--------------------|
| a: H-acid | i: Sulfo-J acid |
| b: Metanilic acid | j: Sulfo-C acid |
| c: 4-Aminoacetanilide-4-sulfonic acid | k: S-acid |
| d: 4-Nitroaniline-3-sulfonic acid | l: K-acid |
| e: 4,4'-Nitroaminostilbene-2,2'-disulfonic acid | m: Sulfanilic acid |
| f: Nitro F. C. acid | n: Chicago acid |
| g: 5-Sulfoanthranilic acid | o: γ -acid |
| h: 6-Chloro-2-aminophenol-4-sulfonic acid | |

The structures of IV_{a-o} were confirmed by their elemental analysis, IR and NMR spectra. The IR spectrum of IV_a shows characteristic bands at 3440 ν (—OH stretching), 1220 ν (C—O stretching), 780 ν (C—S stretching), 1490 ν (—N=N— stretching), 3020 ν (C—H stretching, aromatic), 1340, 1180 cm^{-1} ν (S=O stretching). The NMR spectrum of compound IV_a shows characteristic signals δ 7.48 (1H, —OH), 8.40 (1H, —SO₂NH), 7.80 (1H, —SO₃H).

RESULTS AND DISCUSSION

These compounds when applied on silk, wool and nylon fabrics as 2% shade gave yellow, brown, maroon and coffee shades with poor to good light-fastness,

fair to excellent wash-fastness and poor to excellent exhaustion. Among these dyes the diazo components R_d, R_f, R_e and R_o give deeper shades while the whole group of dyes imparts shine on wool. The brilliance and beauty of the shades and the excellent wash-fastness reveal that some of the acid dyes would prove to be useful dyes for dyeing wool, silk and nylon fabrics.

TABLE-2
SHADES ON POLYESTER, % EXHAUSTION AND FASTNESS PROPERTIES OF IVa-o

Compd.	Exhaustion (%)			Fastness properties					
	Silk	Wool	Nylon	Silk		Wool		Nylon	
				Light ^a	Wash ^b	Light ^a	Wash ^b	Light ^a	Wash ^b
IVa	82	83	82	2	4	2-3	4	2-3	3-4
b	82	85	81	3	3	3	3-4	2-3	4
c	86	87	85	2	3-4	2-3	3-4	2-3	4
d	88	83	81	2	2-3	3	3-4	3	4-5
e	83	80	86	2-3	3-4	2-3	4	2	4-5
f	81	83	82	2	3	3-4	3-4	2	4
g	82	82	81	3	2-3	3	4-5	3-4	4-5
h	84	85	85	2	4	4	4	4	4
i	87	87	81	2-3	3	4-5	4	3-4	4
j	84	82	84	3-4	3-4	4	3-4	4	4
k	81	87	86	2-3	2-3	3	3	4	3-4
l	87	85	85	2	4	2-3	3-4	3	4
m	80	87	86	3-4	4	2-3	4-5	3	4-5
n	82	80	87	3	2-3	2	4-5	2	4-5
o	84	88	84	3-4	3-4	2	3	4	3-4

^aLight-fastness: 1-minimum, 2-poor, 3-moderate, 4-fairly good, 5-good, 6-very good, 7-excellent and 8-maximum.

^bWash-fastness: 1-poor, 2-fair, 3-good, 4-very good and 5-excellent.

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