

Synthesis of Bisazo Acid Dyes Based on 4-Hydroxy-1-Phenylquinoline-2(1H)-one System and Their Dyeing Performance on Various Fabrics

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Fifteen 3-(4'-R-azo-1'-phenylazo)-4-hydroxy-1-phenylquinolin-2(1H)-ones (IVa–o) were prepared by coupling diazotised 3-(4'-amino-1'-phenylazo)-4-hydroxy-1-phenylquinoline-2(1H)-one (III) with various coupling components (a–o). The compound (I) was coupled with diazotised 4-aminoacetanilide giving compound (II), which on hydrolysis gave compound (III). The compounds (IVa–o) were characterised by elemental and spectral analyses and their dyeing performance on silk, wool and nylon fabrics was assessed.

INTRODUCTION

The utility of quinoline derivatives¹ for the production of some commercial dyes and pigments has been reported. The quinoline based dyes using quinoline as coupling components^{2,3} and also diazo components⁴ have been reported. Hence, it was thought interesting to undertake the synthesis and study of dyeing properties of the bisazo acid dyes (IVa–o).

EXPERIMENTAL

4-Hydroxy-1-phenylquinoline-2 (1H)-one (I)

It was prepared by the method already reported².

3-[4'-Acetylamino-1'-phenylazo]-4-hydroxy-1-phenylquinoline-2(1H)-one (II)

A clear solution of I (2.37 g, 0.01 mole) in sodium hydroxide (30 mL, 10%) and acetone (60 mL) was cooled below 5°C and then the solution of diazotised 4-aminoacetanilide (1.5 g, 0.01 mole) was slowly added to it with stirring at 0–5°C during 1 h maintaining the pH between 7.5 to 8.0. The mixture was further stirred for 2 h at 0–5°C. The mixture was diluted with water and the pH was adjusted between 6.5 and 7.0 with acetic acid. The separated compound was filtered, washed with water and crystallised from alcohol to give II, yield 73%, m.p. 245°C. [Found: N, 13.86%; C₂₃H₁₈N₄O₃ requires N, 14.07%]. IR spectra (KBr): 3550–3100 (—OH stretching), 1570 (C=O stretching), 1460 (N=N stretching) and 1290 cm⁻¹ (C—N stretching). NMR spectra (DMSO-d₆ solvent): δ 2.39 (3H, —COCH₃), 2.06 (1H, —NH), 7.38 (1H, —OH quinoline ring) and 2.49 (5H, N—C₆H₅).

3-[4'-Amino-1'-phenylazo]-4-hydroxy-1-phenylquinoline-2 (1H)-one (III)

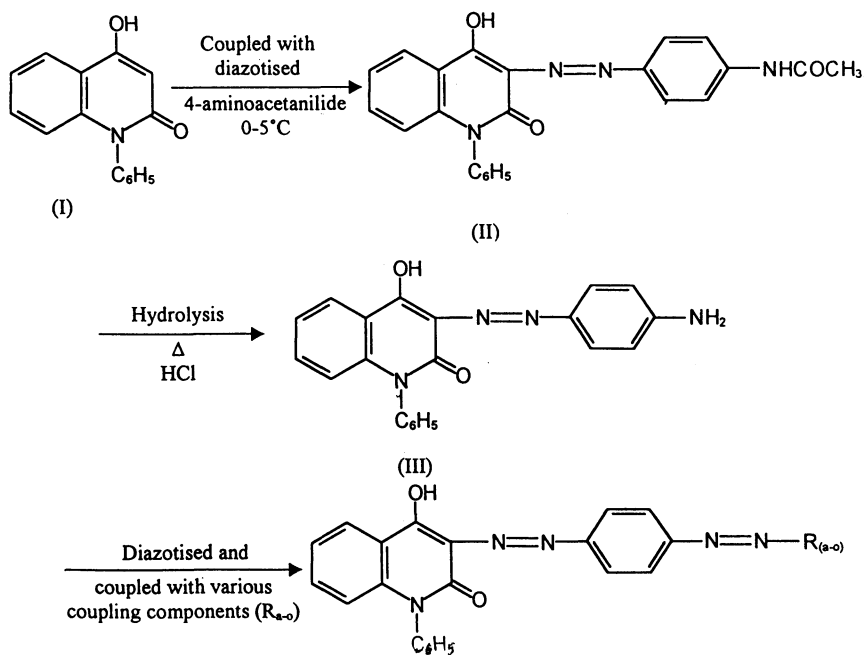
A mixture of **II** (10 g), concentrated hydrochloric acid (10 mL) and distilled water (25 mL) was first heated on water-bath for 30 min and then directly on wire gauze for 1 h. The reaction mixture was cooled and neutralised with sodium bicarbonate. The separated compound was filtered, washed with water and crystallised from alcohol to give **III**, yield 68%, m.p. 115–117°C. [Found: N, 15.60%; C₂₁H₁₆N₄O₂ requires N, 15.73%].

3-[4'-R-azo-1'-phenylazo]-4-hydroxy-1-phenylquinoline-2 (1H)-one (IV_{a-o})

H-acid (3.19 g, 0.01 mole) was suspended in water (20 mL) and dissolved to neutral pH with sodium carbonate solution (10% w/v) to obtain a clear solution. It was cooled below 5°C and then solution of diazotised compound **III** (3.56 g, 0.01 mole) was slowly added to it with stirring at 0–5°C. The pH was maintained between 7.5 and 8.0 by simultaneous addition of 10% sodium carbonate solution. The stirring was continued for 1 h. To this reaction mixture, sodium chloride was added until the coupling material was precipitated. After stirring for 1 h, the liquor was filtered and the product was washed with a small amount of sodium chloride solution (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 **IV_{b-o}** using various coupling components **R_{b-o}** (Scheme I). The characterisation data of compounds **IV_{a-o}** are given in Table-1.

TABLE-1
CHARACTERISATION DATA OF COMPOUNDS **IV_{a-o}**

Compound	Colour	Yield (%)	m.p. > (°C)	Nitrogen (%)	
				Found	Calcd.
IV_a	Violet	60	300	11.33	11.50
b	Violet	73	300	10.30	10.42
c	Red	71	300	13.22	13.37
d	Red	76	300	12.27	13.08
e	Raddish Brown	58	300	11.80	11.93
f	Brown	66	300	13.62	13.76
g	Brown	77	300	15.09	15.24
h	Red	55	300	9.62	9.79
i	Red	69	300	9.58	9.79
j	Violet blue	75	300	13.20	13.37
k	Red	62	300	11.24	11.41
l	Brown	68	300	11.32	11.50
m	Brown	65	300	12.98	13.13
n	Brown	80	300	11.39	11.50
o	Light Pink	65	300	12.04	12.13



For compounds IV_{a-o}

R_a H-acid	f 1-(2,5-dichloro-4-sulfophenyl)-3-methyl-5-pyrazolone	k Schaffer's acid
b N-Phenyl H-acid	g 1-(4-sulfophenyl)-3-methyl-5-pyrazolone	l Chicago acid
c J-acid	h R-acid	m Oxy-Tobias acid
d N-methyl J-acid	i G-acid	n K-acid
e N-phenyl J-acid	j γ acid	o Bon acid

Scheme I. Synthesis of 3-(4'-R-azo-1'-phenylazo)-4-hydroxy-1-phenylquinolino-2(1H)-ones (IV_{a-o}).

The structures of IV_{a-o} were confirmed by their elemental analysis, IR and NMR spectra. The IR spectrum of compound IV_a shows characteristic bands at 3550–3000 (—OH stretching), 1560 (C=O stretching), 1440 (N=N stretching), 1030, 1170 cm^{-1} (S=O stretching). The NMR spectrum of compound IV_a shows characteristic signals at δ 7.60 (1H, —OH quinoline ring), 6.75 (1H, —OH naphthol ring), 3.53 (5H, —N—C₆H₅), 3.77 (2H, —NH₂) and 7.80 (1H, —SO₃H).

RESULTS AND DISCUSSION

These compounds, when applied on silk, wool and nylon fabrics as 2% shade gave orange, brown, yellow, red, blue, violet and gray shades with poor to fairly good light fastness, fair to excellent wash fastness and poor to excellent exhaustion. Among these dyes the coupling components R_c, R_e, R_f, R_g, R_h, R_i, R_k and R_l give excellent deeper shades on silk, wool and nylon fabrics. The brilliancy and beauty of the shades and excellent wash fastness reveal that some of the acid dyes would prove to be useful dyes for dyeing silk, wool and nylon fabrics.

TABLE-2
λ_{max} EXHAUSTION AND FASTNESS PROPERTIES OF IVa-o

Com- pound	λ _{max} nm	Exhaustion (%)			Fastness properties					
		Silk	Wool	Nylon	Silk		Wool		Nylon	
					Light	Wash	Light	Wash	Light	Wash
IVa	316	74	71	62	3-4	2-3	3-4	3	2-3	2-3
b	314	71	73	64	2	3-4	3	4-5	2	4-5
c	315	72	77	59	2	2-3	2-3	3-4	2-3	4-5
d	315	71	74	59	2	2-3	2-3	2-3	2	4-5
e	315	73	85	57	3	3-4	3-4	4	2-3	3-4
f	315	69	80	72	2	2-3	2-3	4-5	4-5	4-5
g	315	71	67	68	2	4	2-3	4	3	4-5
h	317	69	71	61	2-3	3	2-3	4-5	3	4
i	314	73	83	69	2	2-3	3	4	3-4	4-5
j	316	66	74	62	2-3	3	2-3	3-4	2-3	4-5
k	316	69	73	59	2	3-4	2-3	4-5	2	4
l	315	71	72	64	2-3	3-4	2-3	4	2-3	4-5
m	314	71	78	69	2	3-4	2-3	4-5	3	4-5
n	314	71	73	72	2-3	4	2	3-4	2-3	4-5
o	315	69	70	70	2	4	2-3	4	2-3	4-5

ACKNOWLEDGEMENT

The authors are thankful to Principal, P.T. Sarvajani College of Science, Surat, for providing research facilities.

REFERENCES

1. E.N. Abrahat, *Dyes and Their Intermediates*, Edward Arnold Publishers, London, p. 170 (1976).
2. H.T. Mehta and A.G. Mehta, *Asian J. Chem.*, **11**, 515 (1999)
3. ———, *J. Inst. Chemists (India)*, **71**, 91 (1999).
4. Viral Desai and K.R. Desai, *J. Inst. Chemists (India)*, **67**, 150 (1995).

(Received: 14 April 2001; Accepted: 5 July 2001)

AJC-2385