Synthesis and Dyeing Performance of Disperse Dyes Based on 4-Hydroxy-1-Methyl-Quinolin-2(1H)-One System

H.T. MEHTA and A.G. MEHTA*
Department of Chemistry, P.T. Sarvajanik College of Science
Surat-395 001, India

4-Hydroxy-1-methyl-quinolin-2(1H)-one (III) used as a coupling component, was prepared by the methylation of anthranilic acid giving N-methylanthranilic acid (I) followed by acetylation giving N-methyl-N-acetyl anthranilic acid (II) and ring closure. Twenty 3-arylazo-4-hydroxy-1-methyl-quinolin-2(1H)-ones disperse dyes (V_{a-t}) were prepared by coupling(III) with various diazo components (IV_{a-t}). The compounds V_{a-t} are characterized by elemental and spectral analyses and their dyeing performance on polyester fibre was assessed. These compounds gave yellow, orange, brown and red shades with poor to good light-fastness, very good to excellent wash fastness and poor to excellent exhaustion.

INTRODUCTION

The utility of quinoline derivatives[1] for the production of some commercial dyes and pigments has been reported. The quinoline based dyes using quinoline as coupling component[2, 3] and as diazo component[4] have been reported. In view of this encouraging report, some new (III) based azo disperse dyes have been synthesized and their dyeing performance on polyester fibre assessed.

EXPERIMENTAL

N-Methylanthranilic acid (I): Prepared by the known method[5].

N-Methyl-N-acetylantharanilic acid (II): A mixture of N-methylanthranilic acid [15.1 g, 0.1 mol] was refluxed for 2 h. The reaction mixture was poured over ice and allowed to stand at room temperature for 24 h. The product was crystallized from ethanol, (72%), m.p. 268°C. [Found: N, 7.14%; $C_{10}H_{11}O_3N$ requires N, 7.25%]. IR (KBr): 3550–3350 ν(—OH), 1630 ν(C=O), 1240 ν(C—O), 1410 ν(C—N of N—CH_3), 2740 ν(C—H, N—CH_3) cm^{-1}.

4-Hydroxy-1-methyl-quinolin-2(1H)-one (III)

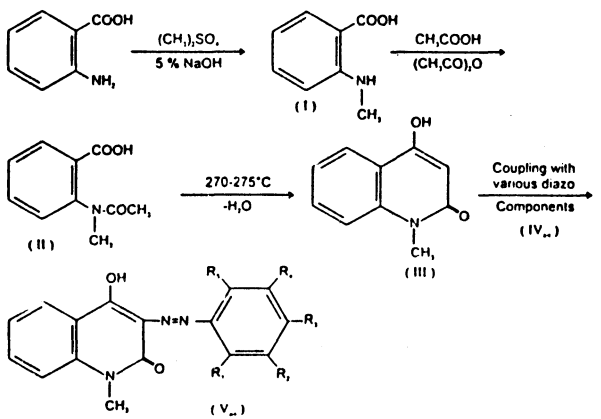
N-Methyl-N-acetyl anthranilic acid [9.65 g; 0.05 mol] was heated in an oil-bath at 270–275°C for 1.5 h. The brown residue was collected and crystallized from DMF (80%), m.p. 252–255°C. [Found: N, 7.82%; $C_{10}H_9O_2N$ requires: N, 8.0%]. IR (KBr): 3560–3240 ν(—OH), 1635 ν(C=O), 1220 ν(C—O), 2740 ν(C—H, N—CH_3), 1415 ν(C—N, N—CH_3) cm^{-1}; NMR (DMSO)d_6 : 3.55 (3H, N—CH_3), 7.32 (1H, —OH); quinoline ring.

3-Arylazo-4-hydroxy-1-methyl quinolin-2(1H)-ones (V_{a-t})

A clear solution of III (1.75 g, 0.01 mol) in sodium hydroxide (30 mL, 10%) and acetone (60 mL) was cooled below 5°C. A clear solution of diazotized aniline (I_{Va}, 0.01 mol) 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. The mixture was diluted

with water and the pH was adjusted between 6.5 and 7.0 with acetic acid. The separated compound Va was filtered, washed with water and crystallized from alcohol. The same procedure was used to prepare V_{b-t} (Table-2). The characterization data of compounds V_{a-t} are given in Table-1.

For compounds IV_{a-t} and V_{a-t}:



Scheme: Synthesis of 3-arylazo-4-hydroxy-1-methyl-quinolin-2(1H)-ones [V_{a-t}].

	R_1	R_2	R_3	R_4	R_5
a	H	H	H	H	H
b	CH_3	H	H	H	H
c	H	CH_3	H	H	H
d	CH_3	H	H	NO_2	H
e	CH_3	H	CH_3	H	H
f	Cl	H	H	H	H
g	H	Cl	H	H	H
h	NO_2	H	H	H	H
i	H	NO_2	H	H	H
j	OCH_3	H	H	H	H
k	H	OCH_3	H	H	H
l	OCH_3	H	NO_2	H	H
m	OC_2H_5	H	H	H	H
n	H	OC_2H_5	H	H	H
o	H	Cl	F	H	H
p	H	CF_3	H	H	H
q	OH	H	H	H	H
r	H	OH	H	H	H
s	H	Cl	H	H	OH
t	H	H	$NHCOCH_3$	H	H

The absorption spectra of compounds were recorded on a Hitachi spectrophotometer (Model V-320) in DMF solution, infrared on a Perkin-Elmer spectrophotometer (Model-377) using KBr pellets, and NMR spectra (DMSO-d_6) on a Varian EM 360L (60 MHz) spectrophotometer using TMS as internal standard. The purity of compounds was checked by TLC using DMF : chlorofrom [3 : 1] as solvent and silica gel G as adsorbent. All compounds were obained in good yield. The structures of V_{a-t} are confirmed by their elemental analyses, infrared and NMR spectra. The IR spectra of these compounds showed characteristic bands at 3560–3100 ν(—OH), 1640–1590 ν(C=O), 1490–1450 ν(N=N), 2740 ν(C—H, N—CH_3) 1415 ν(C—N, N—CH_3) cm^{-1}. The NMR spectra of compound V_t showed characteristic signals at δ 2.47 (3H, —$COCH_3$), 2.05 (1H, —NH), 3.55 (3H, N—CH_3), 7.32 (1H, —OH) quinoline ring.

TABLE-1
CHARACTERIZATION DATA OF V_{a-t}

Compd.[a]	Colour	Yield (%)	m.p.[b] (°C)	λ_{max} (nm)	R_f value	Nitrogen %	
						Found	Required
Va	Light brown	45	195	329	0.85	14.87	15.05
b	Light brown	53	185	324	0.75	14.10	14.33
c	Brown	50	115	320	0.76	14.12	14.33
d	Light brown	67	182	398	0.78	14.38	14.56
e	Light yellow	43	190	324	0.83	13.54	13.68
f	Light brown	48	150	402	0.77	13.16	13.39
g	Light orange	43	155	316	0.79	13.25	13.39
h	Orange	45	230	423	0.80	17.08	17.28
i	Brown	57	135	396	0.82	17.15	17.28
j	Light orange	53	140	324	0.78	13.45	13.59
k	Light brown	41	185	325	0.77	13.48	13.59
l	Brown	64	235	448	0.80	15.67	15.82
m	Light brown	54	194	317	0.84	12.87	13.00
n	Light brown	58	204	318	0.81	12.66	13.00
o	Light brown	67	160	330	0.82	12.41	12.66
p	Light brown	46	130	323	0.68	11.94	12.10
q	Brown	73	250	321	0.69	14.03	14.23
r	Light brown	75	> 300	325	0.72	14.11	14.23
s	Brown	76	209	324	0.81	12.58	12.74
t	Brown	66	207	322	0.87	16.48	16.60

[a]All compounds gave satisfactory elemental analysis for C and H.
[b]Melting points are uncorrected.
λ_{max} was determined at 28°C and 2×10^{-3} M conc.

TABLE-2
SHADES ON POLYESTER, % EXHAUSTION AND FASTNESS PROPERTIES OF V_{a-t}

Compd.	Colour on polyester	Exhaustion (%)	Light fastness[a]	Wash fastness[b]
Va	Yellow	70	3	4–5
b	Light orange	68	3–4	4–5
c	Light orange	61	3	4–5
d	Yellow	65	2–3	4–5
e	Orange	68	2	4
f	Yellow	71	2–3	4–5
g	Yellow	69	2–3	5
h	Yellow	66	2	4–5
i	Yellow	53	4–5	4–5
j	Yellow	69	4	4–5
k	Cherry red	46	2–3	4
l	Light orange	69	2	4
m	Golden yellow	65	2–3	5
n	Orange	42	3–4	4–5
o	Cream	71	3	4–5
p	Yellow	60	2	4–5
q	Yellow	62	2	4–5
r	Cream	64	3–4	4–5
s	Orange	47	2–3	4–5
t	Brown	69	3–4	4–5

[a]Light–fastness: 1–minimum, 2–poor, 3–moderate, 4–fairly good, 5–good, 6–very good, 7–excellent and 8–maximum

[b]Wash fastness: 1–poor, 2–fair, 3–good, 4–very good and 5–excellent

Application

For dyeing polyster in 2% shade, a laboratory model glycerine-bath high temperature beaker dyeing machine was used. A paste of 40 mg dye was prepared with dispersing agent Dadamol (40 mg), wetting agent Tween-80 (5 mg) and water (1 mL) in a ball-mill. To this paste, water (99 mL) was added with stirring and the pH was adjusted to 4.0 using acetic acid. The above dye solution (100 mL) was added to a beaker provided with a lid screw cap. Before closing the lid and tightening the metal cap over the beaker a wetted pattern of polyester was rolled into the beaker. The beaker was then placed vertically on a rotary carrier inside the tank and the clamp was firmly tightened. The rotary carrier was then allowed to rotate in the glycerine-bath, the temperature of which was raised to 130°C at the rate of 2°C/min. The dyeing was continued for 1 h at 130°C under pressure. After cooling for 1 h, the beaker was removed from the bath and the dyed pattern was washed several times with cold water, treated with a mixture of

detergent (0.2 g) and sodium carbonate (0.1 g) in water (100 mL) for 30 min at 80°C, again washed thoroughly with water and dried at room temperature.

The characterization data and fastness properties of compounds V_{a-t} are given in Tables 1 and 2 respectively. These compounds, when applied on polyester fibre as 2% shade, gave yellow, orange, brown and red shades with poor to good light fastness, very good to excellent wash-fastness and poor to excllent exhaustion (Table-2).

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REFERENCES

1. E.N. Abrahart, Dyes and Their Intermediates, Edward Arnold Publishers Ltd., London, p. 170 (1976).
2. U.V. Malankar and K.R. Desai, *Oriental J. Chem.*, **10**, 171 (1994).
3. H.T. Mehta and A.G. Mehta, *J. Inst. Chemists (India)* (in press).
4. Viral Desai and K.R. Desai, *J. Inst. Chemists (India)*, **67**, 150 (1995).
5. F.G. Mann and B.C. Saunder, Practical Organic Chemistry, 3rd Edition, p. 215.

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