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Studies on Synthesis and Dyeing Preformance of Acid Dyes Based on 4,7-Dihydroxy-3,8-di-α-naphthyl-azo-1,10-phenanthroline-2,9-dione

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Abstract: Some new azo acid dyes were prepared by coupling various diazotized acid components such as anthranilic acid, sulphanilic acid, laurent acid, peri acid, tobias acid, H-acid, J-acid, gamma acid, sulphotobias acid, 4-aminotoluiene-3-sulphonic acid, 5-sulpho- anthranilic acid, 2-naphthylamine-3,6,8-trisulphonic acid, bronner acid, metanilic acid and cleve acid with 4,7-dihydroxy-3,8-di-α-naphthylazo-1,10-phenanthroline-2,9-dione. The dyes were characterized by elemental, IR and TLC analyses. Their dyeing performance as acid dyes has been assessed on viscose rayon, wool and cotton fibres.

Keywords: Acid azo dyes, Dyeing performance, IR.

Introduction

Some of the dyes based on 4-oxo-quinazoline ring system have been reported to be useful on natural and man-made fibres[1,2]. Acid dyes, the newest addition of existing dyes are the centre of attraction in dyestuff research[3]. The acid dyes based on 4,7-dihydroxy-1,10-phenanthroline-2,9-dione (A), a heterocyclic ring system, were reported earlier[4] showed good affinity, moderate to very good light and wash fastness and good to very good exhaustion.

In continuation of our earlier work on acid dyes based on heterocyclic ring system it was thought interesting to study the synthesis, characterization and dyeing performance of various acid dyes based on 4,7-dihydroxy-3,8-di-α-naphthylazo-1,10-phenanthroline-2,9-dione system.

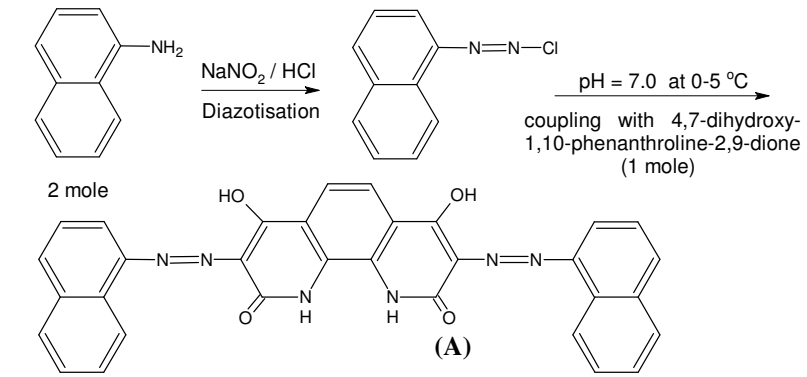
Experimental

Melting points were determined in open capillaries and are uncorrected. The IR spectra (KBr) were recorded on a Perkin-Elmer spectrophotometer (Model 377). UV-Visible spectra were recorded on Backman DB-GT grating spectrophotometer using $1x10^{-3}$ M aqueous solution. TLC was carried out on Silica gel-G as absorbent using reported procedure[5].

Synthesis of 4,7-dihydroxy-3,8-di-α-naphthylazo-1,10-phenanthroline-2,9-dione (A)

2-Naphthylamine (0.02 mole, 2.860 g) was diazotized at 0-5 °C was then coupled with 4,7-dihydroxy-1,10-phenanthroline-2,9-dione (0.01 mole, 3.169 g) at 0-5 °C maintaining the pH 7.0. The reaction mixture was stirred for 2 h. The compound formed was filtered and dried at 80 °C in an oven, m.p. 145 °C, was then purified by DMF-acetone and used as coupling component in the synthesis of tetra azo acid dyes.

Reaction scheme



Coupling of diazotized acid component with (A)

The coupling component (0.01 mole, 5.32 g) was suspended in NaOH (10 %, 15 mL) and the pH of the solution is maintained neutral with sodium carbonate solution (10 % w/v) to obtain a clear solution. The solution was cooled to 0-5 °C and the cooled diazo solution of acid component (0.02 mole) was added drop wise with stirring, maintaining the pH to 8.0 by simultaneous addition of sodium carbonate (10 % w/v). Stirring was continued for 2 h at 0-5 °C. The reaction mixture was heated to 60 °C after 2 h and sodium chloride was added till the dyestuff is precipitated out. After stirring for an hour the liquor was filtered and

the product was washed with a small amount of 5 % sodium chloride solution. The dye was dried at 90°C. It was then purified by extraction with DMF and precipitation by diluting the DMF extract with acetone.

Dyeing of fibres

All the dyes D_1 to D_{15} were applied on viscose rayon, wool and cotton fibres in 2 % shade by using the reported procedure[6].

Fastness test

Fastness to the light was assessed in accordance with BS:1006-1978 and the wash fastness was assessed in accordance with IS:765-1979.

Results and Discussion

The melting point, % yield, % N, R_f values and λ_{max} values of the dyes synthesized are given in Table 1. All the dyes were obtained in excellent yield (68-81 %). TLC of the samples indicates single spot purity for all the dyes. The IR spectra of the dyes D_1 to D_{15} showed characteristic band at 3000-3235 cm^{-1} (-OH), 3330-3455 cm^{-1} (-NH), 1590-1640 cm^{-1} (-C=O), 1380-1450 and 170 cm^{-1}(-N=N-), 1050-1250 cm^{-1} ($-SO_3H$).

Table 1. Physical data for acid dyes

Dye No.	Diazo component (R)	Molecular formula	% N found	m.p. °C	Yield %	λ_{max}	R_f
D_1	Anthranilic acid	$C_{16}H_{28}N_{10}O_8$	16.54	160	80	300	0.88
D_2	Sulphanilic acid	$C_{44}H_{26}N_{10}O_{10}S_2Na_2$	14.56	185	75	320	0.79
D_3	Laurent acid	$C_{52}H_{30}N_{10}O_{10}S_2Na_2$	13.21	130	71	370	0.78
D_4	Peri acid	$C_{52}H_{30}N_{10}O_{10}S_2Na_2$	13.20	150	81	240	0.81
D_5	Tobias acid	$C_{52}H_{30}N_{10}O_{10}S_2Na_2$	13.23	190	72	280	0.83
D_6	H-acid	$C_{52}H_{28}N_{10}O_{18}S_4Na_4$	10.80	170	69	330	0.80
D_7	J-acid	$C_{52}H_{30}N_{10}O_{12}S_2Na_2$	12.82	155	70	310	0.82
D_8	Gamma acid	$C_{52}H_{30}N_{10}O_{12}S_2Na_2$	12.80	190	74	330	0.85
D_9	Sulphotobias acid	$C_{52}H_{28}N_{10}O_{16}S_4Na_4$	11.10	210	72	280	0.88
D_{10}	4-aminotoluene-3-sulphonic acid	$C_{46}H_{30}N_{10}O_{10}S_2Na_2$	14.15	170	68	225	0.74
D_{11}	5-sulpho anthranilic acid	$C_{46}H_{26}N_{10}O_{14}S_2Na_2$	13.38	120	74	310	0.91
D_{12}	2-naphthylamine-3,6,8-trisulphonic acid	$C_{52}H_{30}N_{10}O_{10}S_2Na_2$	9.60	160	77	398	0.92
D_{13}	Bronner acid	$C_{52}H_{30}N_{10}O_{10}S_2Na_2$	13.20	120	71	295	0.84
D_{14}	Metanilic acid	$C_{44}H_{26}N_{10}O_{10}S_2Na_2$	14.60	170	79	320	0.74
D_{15}	Cleve acid	$C_{52}H_{30}N_{10}O_{10}S_2Na_2$	13.20	150	73	340	0.82

All the dyes D_1 to D_{15} were applied to viscose rayon, wool and cotton fibers gave violet, blue, green, yellow, orange brown and maroon shades. D_7, D_8, D_{11}, D_{14}, D_{15} gives deeper shades on all three fibers. Most of the dyes give green shades. The % exhaustion and fastness data of dyes D_1 to D_{15} are given in Table-2 indicates good affinity for all the fibers used in study. The pick-up values of these dyes varied from 2 to 4. The light fastness varied

from poor to fairly good whereas fastness to washing varied from fair to excellent. Exhaustion ranges from 62.00 % to 72.50 %.

Table 2. % Exhaustion, Light fastness and Wash fastness data

Dye	Viscos rayon			Wool			Cotton		
No.	%Ex.	L.F.	W.F.	%Ex.	L.F.	W.F.	%Ex.	L.F.	W.F.
D_1	62.50	3-4	3	65.00	3-4	3-4	70.00	3	4
D_2	63.75	2-3	4-5	70.00	4	4-5	71.25	2-3	2-3
D_3	65.00	3	3	67.00	3-4	2-3	74.25	3	3-4
D_4	70.00	3-4	4-5	72.00	3-4	3	73.75	4-5	2-3
D_5	68.87	2-3	3	66.25	3	3	70.00	2-3	3-4
D_6	66.25	3-4	3	70.50	2-3	3	68.75	3	4
D_7	67.00	4-5	3-4	67.50	3-4	4	67.00	3-4	2-3
D_8	63.00	2-3	4	65.00	3	2-3	65.50	4-5	4
D_9	70.50	3-4	2-3	68.87	3	3-4	70.00	2-3	3
D_{10}	67.50	3	3-4	73.75	3	3-4	68.00	3-4	4
D_{11}	64.45	3	3	73.00	3-4	2-3	72.00	3	2-3
D_{12}	68.87	3-4	3	70.00	4-5	3-4	65.00	3-4	3
D_{13}	66.25	3-4	3	63.75	4-5	3-4	66.25	4-5	4
D_{14}	65.00	2-3	2-3	74.50	2-3	3	63.75	2-3	3
D_{15}	67.50	4	4-5	67.00	3	2-3	74.50	3-4	4-5

%Ex. = % Exhaustion; L.F. = Light fastness; W.F. = Wash fastness

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