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Studies on Synthesis and Dyeing Preformance of Acid Dyes Based on 4,7-Dihydroxy-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-aminotoluene-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-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

The newest additions of existing acid dyes are the centre of attraction in dyestuff research¹. Various dyes based on quinazoline, quinoline and 4-oxo-quinazoline ring system have been reported to be useful on natural and man-made fibres, showed high affinity and good dyeing performance²⁻⁴. Some of them also showed potential biological activity. Therefore it was thought interesting to study the synthesis, characterization and dyeing performance of various acid dyes based on 4,7-dihydroxy-1,10-phenanthroline-2,9-dione heterocyclic ring 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 1×10^{-3} M aqueous solution. TLC was carried out on Silica gel-G as absorbent using reported procedure⁵.

Synthesis of 4,7-dihydroxy-1,10-phenanthroline-2,9-dione (A)

A solution of *o*-phenylenediamine (0.1 mole, 10.80 g) in diethyl malonate (0.22 mole, 13.930 mL) was added portion wise to poly phosphoric acid (prepared from 38.6 g P₂O₅ and 21.4 mL of orthophosphoric acid). The deep green pasty mass obtained was heated at 170°C for 30 minutes. The reaction mixture was left to cool and poured into cold water. The solution obtained was neutralized with 5% NaOH solution. The product obtained was filtered, dried and crystallized. m.p. 315°C, yield 75%.

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 (0.01 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 hours at 0-5°C. The reaction mixture was heated to 60°C after 2h 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.

Reaction Scheme*Dyeing of fibres*

All the dyes D₁ to D₁₅ were applied on viscose rayon, wool and cotton fibres in 2% shade by using the reported procedure⁶.

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 D₁ to D₁₅ 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 (-SO₃H).

All the dyes D₁ to D₁₅ were applied to viscose rayon, wool and cotton fibres gave violet, blue, green, yellow, orange brown and maroon shades. D₇, D₈, D₁₁, D₁₄, D₁₅ gives deeper shades on all three fibres. Most of the dyes give green shades. The % exhaustion and

fastness data of dyes D₁ to D₁₅ are given in Table 2 indicates good affinity for all the fibres 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 63.75% to 74.50%.

Table 1. Physical data for acid dyes

Dye No.	Diazo component (R)	Molecular formula	% N found	m.p. °C	% yield	$\lambda_{\max}$	R _f
D ₁	Anthranilic acid	C ₁₉ H ₁₂ N ₄ O ₆	15.60	170	79	226	0.91
D ₂	Sulphanilic acid	C ₁₈ H ₁₁ N ₄ O ₇ SNa	12.50	155	72	301	0.89
D ₃	Laurent acid	C ₂₂ H ₁₁ N ₄ O ₇ SNa	11.24	165	80	306	0.83
D ₄	Peri acid	C ₂₂ H ₁₁ N ₄ O ₇ SNa	11.25	190	77	389	0.79
D ₅	Tobias acid	C ₂₂ H ₁₁ N ₄ O ₇ SNa	11.28	180	75	319	0.81
D ₆	H-acid	C ₂₂ H ₁₂ N ₄ O ₁₁ S ₂ Na ₂	9.10	120	65	560	0.83
D ₇	J-acid	C ₂₂ H ₁₃ N ₄ O ₈ SNa	10.89	160	69	410	0.78
D ₈	Gamma acid	C ₂₂ H ₁₃ N ₄ O ₈ SNa	10.88	190	71	252	0.91
D ₉	Sulphotobias acid	C ₂₂ H ₁₂ N ₄ O ₁₀ S ₂ Na ₂	9.34	170	74	398	0.91
D ₁₀	4-aminotoluene-3-sulphonic acid	C ₁₉ H ₁₃ N ₄ O ₇ SNa	12.10	199	78	398	0.89
D ₁₁	5-sulpho anthranilic acid	C ₁₉ H ₁₁ N ₄ O ₉ SNa	11.25	135	72	240	0.91
D ₁₂	2-naphthylamine-3,6,8-trisulphonic acid	C ₂₂ H ₁₁ N ₄ O ₁₃ S ₃ Na ₃	7.99	230	80	330	0.88
D ₁₃	Bronner acid	C ₂₂ H ₁₃ N ₄ O ₇ SNa	11.28	160	79	325	0.84
D ₁₄	Metanilic acid	C ₁₈ H ₁₁ N ₄ O ₇ SNa	12.50	170	75	320	0.81
D ₁₅	Cleve acid	C ₂₂ H ₁₃ N ₄ O ₇ SNa	11.25	120	81	410	0.79

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

Dye No.	Viscos rayon			Wool			Cotton		
	%Ex.	L.F.	W.F.	%Ex.	L.F.	W.F.	%Ex.	L.F.	W.F.
D ₁	63.75	4-5	2-3	66.25	4-5	3	66.25	4	4-5
D ₂	66.25	4	3-4	67.50	3-4	3	63.75	3-4	4
D ₃	65.00	3-4	2-3	70.00	2-3	4-5	54.45	2-3	3-4
D ₄	72.00	3-4	3	73.75	3	3-4	71.25	4-5	3
D ₅	68.00	2-3	3	70.50	4-5	2-3	71.75	4-5	4-5
D ₆	70.00	3	3	67.50	4	3	74.50	4	2-3
D ₇	65.50	3	3-4	70.00	2-3	4-5	73.00	3-4	3
D ₈	67.00	3	3-4	66.75	3	4	70.00	2-3	4
D ₉	68.75	3-4	2-3	63.75	4	4	68.75	3	3-4
D ₁₀	70.50	2-3	3	64.45	3-4	3-4	67.50	4	4
D ₁₁	73.75	3-4	3	71.25	3	2-3	67.00	4-5	2-3
D ₁₂	74.50	2-3	3	73.75	3-4	2-3	65.00	3-4	3
D ₁₃	71.25	3	3-4	74.50	4-5	3	68.87	3	4
D ₁₄	70.00	3	2-3	73.00	4	2-3	67.50	4	4
D ₁₅	65.00	2-3	3	70.00	2-3	3	65.50	4-5	2-3

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

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