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Characterization, application and microbial study of imidazole base acid anthraquinone dyes

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ABSTRACT

A new series of Imidazole based anthraquinone acid dyes were synthesized by condensation of bromanie acid with imidazole to produce 1-amino-4-(imidazolyl)anthraquinone-2-sulfonic acid was diazotized and coupled with various naphthalene based acid coupling components to produce the novel series of acid anthraquinone dyes. All the synthesized dyes were characterized by elemental analysis spectral studies and the dyeing performance on wool, silk and nylon fibres has been assessed. All the synthesized dyes give good to very good light fastness on each fibre. The percentage dye-bath exhaustion on different fibres was good and aceptable. The dyed fibres showed moderate to very good fastness to light, washing and rubbing. All the synthesized dyes have been screened for their antimicrobial activity.

Key words: Anthraquinone, Acid dyes, Dyeing, Wool, Silk and Nylon.

INTRODUCTION

Anthraquinone acid dyes are one of the most important class of acid dyes being principally used for green, blue or violet shades having excellent light and wet fastness. One important group of such dyes is those obtained by the condensation of bromamine acid with imidazole by an Ullmann reaction catalyzed by copper salt. Acid dyes have found wide application in dyeing wool, polyamide fibres and blends of both fibres[1,2] but they meet very high requirements as regards their application and fastness. The characteristics chromophore of the anthraquinone series consists of one or more carbonyl group in association with a conjugated system. Anthraquinone dyes and colorants make important are acid, direct, disperse, modertant, vat, solvent are reactive dyes even as pigments[3-8] as a class anthraquinone acid dyes are known for their specific colour and high light fastness characteristic. Anthraquinone acid dyes provide a number of bright fast no light blue and green neither available among the azo dyes nor in fact equaled by any other class. The colour for anthraquinone dyes depends upon substitution among definite lines[9-10].

EXPERIMENTAL

The purity of all the dyes has been by TLC[11], IR spectra were recorded in KBr on a perkin Elmer Model 881 spectrophotometer and ^{1}H-NMR spectra on Brucker AVANCE II 400 NMR spectrometer (SAIF, chandigarh, Panjab university) using TMS as internal standard and DMSO as solvent. Absorption spectra were recorded on a Shimadzu UV-1700 spectrophotometer. Elemental analysis of C, H and N was carried out on Carlo Erba 1108 instruments. The light fastness was assessed in accordance with BS: 1006-1978 [12], the rubbing fastness test was carried out with corkmeter (atlas) in accordance with AATCC-1961 [13] and the wash fastness test is accordance with IS: 765-1979 [14].

1-Amino-4-imidazolylanthraquinone-2-sulfonicacid B

Bromamine acid A (0.1 mol) was dissolved in 400ml hot water (70-80°C), imidazole (0.1mol), acid binding agent sodium bicarbonate (0.2mol), copper sulfate (0.5g) and ferrous sulfate (0.5g) catalysts were then added to it. The reaction mixture was stirred and heated to 90°C. Maintained temperature 90°C for 6 h under stirring. Charcoal (1g) was added and stirred for 15 min and the solution was filtered by slowly adding diluted hydrochloride acid (1:1) under stirring. The product was salted out by adding sodium chloride, cooled to room temperature, stirred for 30-min at room temperature, filtered and washed with 10% w/v brine solution. Dried it. (82% yield).

IR (KBr) cm^{-1} : 3550, 3420 (-NH_2), 1280 (C-N), 1680 (C=O), 1150, 1050 (-SO_3H)

Diazotization of 1-amino-4-imidazolylan thraquinone-2-sulfonic acid C

The solution of 1-amino-4-imidazolylan thraquinone-2-sulfonic acid B (0.01 mol) in water. Hydrochloric acid (0.015mol) was added to this and well stirred. The solution was cooled at 0-5°C in are ice bath. A solution of sodium nitrite ($NaNO_2$ 0.015 mol) in water (8 ml) previously cooled to 0-5°C, was then added over a period of five minutes with stirring. The stirring was continued for an h, maintained the same temperature with positive test for nitrous acid on starch iodide paper. After stirring one h, just destroyed excess of nitrous acid with require amount of a solution of sulfonic acid. The resulting diazo solution was obtained at 0-5°C was used for subsequent coupling reaction.

Coupling of diazo solution with J-acid D

J-acid (2.39 gm, 0.01mol) was suspended in water (20ml) and dissolved at neutral pH with sodium carbonate (10% w/v) to obtain a clear solution. The solution was cooled to below 0-5°C in an ice bath. To this well stirred solution, diazo chloride solution C was added drop wise over a period of 10-15 min maintaining the pH 7.5-8.0 by simultaneous addition of sodium carbonate solution (10% w.v). The stirring was continued for three h keeping the temperature at 0-5°C. The temperature of reaction mixture was then raised to 60 °C and sodium chloride added to precipitate the coloring material. The stirring was continued for 1h. Filter and washed with a small amount of Sodium chloride solution (5% w/v). The solid was dried at 80-90°C and extracted with DMF. Coupling the DMF extract with excess of chloroform. The dyes thus obtained was filtered, washed with chloroform and dried at 60°C (81% yield).

Same procedure was adapted to synthesized using various naphthalene base acid coupling components such as gamma acid, m-amino benzoyl K-acid, N-methyl, J-acid, sulfogamma acid, chromotropic acid, violet acid, N-phenyl J-acid, G-acid, R-acid, N-benzoyl J-acid, epsilon acid, Schaeffer acid, acetyl H-acid, N-(3-sulfophenyl)-gamma acid, acetyl gamma acid, m-aimobenzoyl H-acid, acetyl J-acid, H-acid and N-benzoyl H- acid. Characterization data IR, spectral data and ^1H NMR data of all the dyes are given in Table 1,2 and 3 respectively.

Dey - Bath materials

Materials	for wool	for silk	for nylon
Fibre (g)	2.0g	2.0g	2.0g
Amount of dye (mg)	40.0mg	40.0mg	40.0mg
Glauber salt (20%)	1.5ml	1.0ml	1.0ml
Formic acid soln. (10%)	1.5ml	1.0ml	1.5ml
pH	3.0	3.0	3.0
MLR	1.40	1.40	1.40
Dyeing time (min)	60min	40min	90min
Dyeing temp (°C)	100°C	85°C	100°C
Total volume	80ml	80ml	80ml

Table 1: Characterization data of D_1 - D_{20}

Dye No.	Coupling component	Molecular formula	Yield (%)	Found (Calcd %) C	H	N	Rf Value
D_1	J-acid	$C_{27}H_{15}O_9N_5S_2Na_2$	80	48.82 48.87	2.20 2.26	10.50 10.56	0.36
D_2	Gamma acid	$C_{27}H_{15}O_9N_5S_2Na_2$	74	48.81 48.87	2.21 2.26	1049 10.56	0.46
D_3	M-Amino benzoyl K-acid	$C_{34}H_{19}O_{13}N_6S_3Na_3$	81	46.09 46.15	2.08 2.15	9.42 9.50	0.42
D_4	N-methyl J-acid	$C_{28}H_{17}O_9N_5S_2Na_2$	74	49.58 49.63	2.46 2.51	10.26 10.34	0.38
D_5	Sulfogamma acid	$C_{27}H_{14}O_{12}N_5S_3Na_3$	80	42.29 42.35	1.77 1.83	9.08 9.15	0.40
D_6	Chromotropic acid	$C_{27}H_{13}O_{13}N_4S_3Na_3$	79	42.22 42.30	1.64 1.70	7.23 7.31	0.43
D_7	violet acid	$C_{27}H_{13}O_{13}N_4S_3Na_3$	81	43.24 43.20	1.66 1.73	7.40 7.47	0.44
D_8	N-phenyl J-acid	$C_{33}H_{19}O_9N_5S_2Na_2$	73	53.51 53.58	2.51 2.57	9.41 9.47	0.42
D_9	G-acid	$C_{27}H_{13}O_{12}N_3S_3Na_3$	79	43.14 43.20	1.68 1.73	7.42 7.47	0.41
D_{10}	R-acid	$C_{27}H_{13}O_{12}N_3S_3Na_3$	74	43.15 43.20	1.67 1.73	7.41 7.47	0.39
D_{11}	N-benzoyl J-acid	$C_{34}H_{19}O_{10}N_5S_2Na_2$	80	53.11 53.19	2.41 2.48	9.09 9.13	0.45
D_{12}	Epsilon acid	$C_{27}H_{13}O_{13}N_4S_3Na_3$	78	43.12 43.20	1.68 1.73	7.47 7.47	0.37
D_{13}	Schaffer's acid	$C_{27}H_{14}O_9N_4S_2Na_2$	76	49.96 50.00	2.08 2.16	8.59 8.64	0.46
D_{14}	Acetyl H-acid	$C_{29}H_{16}O_{13}N_5S_3Na_3$	73	43.06 43.12	1.93 1.98	8.61 8.67	0.36
D_{15}	N-(3-sulfophenyl)-Gamma acid	$C_{33}H_{18}O_{12}N_5S_3Na_3$	81	47.01 47.08	2.08 2.14	8.25 8.32	0.40
D_{16}	Acetyl gamma acid	$C_{29}H_{17}O_{10}N_5S_2Na_2$	72	49.29 49.36	2.34 2.41	9.86 9.93	0.42
D_{17}	m-amino benzoyl H-acid	$C_{34}H_{19}O_{13}N_6S_3Na_3$	86	46.08 46.15	2.09 2.15	9.43 9.50	0.43
D_{18}	Acetyl J-acid	$C_{29}H_{17}O_{10}N_5S_2Na_2$	85	49.28 49.36	2.35 2.41	9.85 9.93	0.45
D_{19}	H-acid	$C_{27}H_{14}O_{12}N_5S_3Na_3$	81	42.27 42.35	1.78 1.83	9.08 9.15	0.42
D_{20}	N-Benzoyl H-acid	$C_{34}H_{18}O_{13}N_5S_3Na_3$	78	46.88 46.95	2.00 2.07	7.09 8.05	0.37

Dyeing of fibres

All the dyes D_1 to D_{20} were applied on wool, silk and nylon by using different procedure having dye bath material as given below.

RESULTS AND DISCUSSION

The new series of acid anthraquinone dyes contain imidazole were synthesized by condensation of bromamine acid with imidazole, this condense product is diazotized and couple with various naphthalene based acid coupling components to produce the novel series of acid anthraquinone dyes. The series of dyes have found wide application in dyeing wool, polyamide fibre and blend. In this series chromophoric group such as carbonyl group is condense with conjugated system like anthraquinone to produce violet colour shades and high light fat ness characteristics.

Exhaustion and fixation study

The percentage exhaustion of 2% dyeing on wool fabric shows exhaustion 67.00% to 78.00%, on silk fabric shows exhaustion 65.10% or 79.40%

Table 2: IR Spectral data of D_1 to D_{20}

Comp.	IR Spectra
D_1	$-NH_2$ asym & sym (3538, 3430), -OH (3386), -C=O str. (1692), -N=N- (1453), -C-N (1271), S=O asym & sym (1200, 1034)
D_2	$-NH_2$ asym & sym (3542, 3424), -OH (3380), -C=O str. (1682), -N=N- (1467), -C-N (1264), S=O asym & sym (1207, 1053)
D_3	$-NH_2$ asym & sym (3530, 3425), -OH (3400), -C=O str. (1692), -CONH- (1660),-N=N (1454) -C-N (1278), S=O asym & sym (1185, 1049)
D_4	$-NH_2$ asym & sym (3590) -OH (3372), -C=O str. (1674), -N=N- (1462), -C-N (1278), S=O asym & sym (1183,1052)
D_5	$-NH_2$ asym & sym (3531,3422) -OH (3382), -C=O str. (1691), -N=N- (14625, -C-N (1271), S=O asym & sym (1193,1041)
D_6	-OH (3385), -C=O str (1675)-N=N-(1455)-S=O asym & sym (1202, 1057)
D_7	-OH (3388), -C=O str (1683)-N=N-(1440)-S=O asym & sym (1209, 1060)
D_8	-NH- (3507)-OH (3381), -C=O str (1683)-N=N-(1472)-S=O asym & sym (1183, 1057)
D_9	-OH (3394), -C=O str (1695)-N=N-(1457)-C-N (1289),-S=O asym & sym (1199, 1065)
D_{10}	-OH (3412), -C=O str (1663)-N=N-(1460)-S=O asym & sym (1187, 1068)
D_{11}	-OH (3394), -C=O str (1676)-CONH-(1660), N=N-(1475)-S=O asym & sym (1203, 1064)
D_{12}	-OH (3378), -C=O str (1689)-N=N-(1454)-S=O asym & sym (1192, 1052)
D_{13}	-OH (3376), -C=O str (1668)-N=N-(1453)-S=O asym & sym (1212, 1044)
D_{14}	-OH (3374), -C=O str (1675)-CONH-(1656), N=N-(1459)-S=O asym & sym (1200, 1041)
D_{15}	-NH-(3392), -OH(3385), -C=O str (1672)-N=N-(1455)-S=O asym & sym (1196, 1047)
D_{16}	-OH (3368), -C=O str (1673)-N=N-(1464)-S=O asym & sym (1205, 1055)
D_{17}	$-NH_2$ asym & sym (3532, 3439), -OH (3360), -C=O str. (1671),- CONH-(1645)-N=N- (1453), -C-N (1292), S=O asym & sym (1198, 1050)
D_{18}	-OH (3369), -C=O str (1684)-CONH-(1655), N=N-(1460)-S=O asym & sym (1210, 1051)
D_{19}	$-NH_2$ asym & sym (3545, 3440), -OH (3382), -C=O str. (1670), -N=N- (1454), -C-N (1292), S=O asym & sym (1200, 1053)
D_{20}	-OH (3372), -C=O str (1673)-CONH-(1652), N=N-(1467)-S=O asym & sym (1202, 1054)

Table 3: ^{1}H-NMR Spectral data of D_1 to D_{20}

Comp.	^{1}H-NMR
D_1	-CH_2 - (2H,t, 2.40), -NH_2 (2H, s, 3.82)- OH (1H, s, 5.28), Ar-H (9H, m, 7.22-8.43)
D_2	-CH_2 - (2H,t, 2.48), -NH_2 (2H, s, 3.74)- OH (1H, s, 5.27), Ar-H (9H, m, 7.22-8.39)
D_3	-CH_2 - (2H,t, 2.50), -NH_2 (2H, s, 3.72)- OH (1H, s, 5.14), Ar-H (12H, m, 7.17-8.34), -NHCO-(1G, s, 9.62)
D_4	-NH-(1H, t, 1.74), -CH_2 (2H, t, 2.41)- OH (1H, s, 5.16), Ar-H (9H, m, 7.22-8.45)
D_5	-CH_2 - (2H,t, 2.46), -NH_2 (2H, s, 3.77)- OH (1H, s, 5.20), Ar-H (8H, m, 7.18-8.32)
D_6	-CH_2 - (2H,t, 2.35), -OH (1H, s, 5.18), Ar-H (9H, m, 7.32-8.40)
D_7	-CH_2 - (2H,t, 2.35), -OH (1H, s, 5.18), Ar-H (9H, m, 7.10-8.22)
D_8	-NH-(1H, t, 1.74), -CH_2 (2H, t, 2.43)- OH (1H, s, 5.16), Ar-H (13H, m, 7.31-8.45)
D_9	-CH_2 - (2H, t, 2.47), -OH (1H, s, 5.08), Ar-H (9H, m, 7.24-8.41)
D_{10}	-CH_2 - (2H, t, 2.44), -OH (1H, s, 5.16), Ar-H (9H, m, 7.20-8.43)
D_{11}	-CH_2 - (2H, t, 2.50), -NH_2 (2H, s, 5.22), Ar-H (13H, m, 7.31-8.46), -NHCO-(1G, s, 9.76)
D_{12}	-CH_2 - (2H, t, 2.42), -OH (1H, s, 5.19), Ar-H (9H, m, 7.25-8.36)
D_{13}	-CH_2 - (2H, t, 2.47), -OH (1H, s, 5.24), Ar-H (10H, m, 7.27-8.38)
D_{14}	-CH_2 - (2H, t, 2.50), -OH (1H, s, 5.20), Ar-H (8H, m, 7.24-8.35)
D_{15}	-NH_2 - (1H, t, 1.78), -CH_2 (2H, t, 2.43), -OH (1H, s, 5.17), Ar-H (13H, m, 7.12-8.36)
D_{16}	-CH_2 - (2H, t, 2.53), -OH-(1H, s, 5.15) Ar-H (7.15-8.33), -NHCO-(1H, s, 9.75)
D_{17}	-CH_2 - (2H, t, 2.46), -OH-(1H, s, 5.20) Ar-H (12H, m, 7.28-8.46), -NHCO-(1H, s, 9.60)
D_{18}	-CH_2 - (2H, t, 2.41), -OH-(1H, s, 5.19) Ar-H (9H, m, 7.25-8.36), -NHCO-(1H, s, 9.69)
D_{19}	-CH_2 - (2H, t, 2.48), -NH_2 (2H, s, 3.71) -OH-(1H, s, 5.12) Ar-H (8H, m, 7.25-8.38), -NHCO-(1H, s, 9.66)
D_{20}	-CH_2 - (2H, t, 2.54), -OH-(1H, s, 5.14) Ar-H (13H, m, 7.26-8.45), -NHCO-(1H, s, 9.72)

Table 4: Results of Exhaustion and Fixation study of D_1 - D_{20} on wool, silk and nylon

Dye	Wool		Silk		Nylon	
No.	% Exhaustion	%Fixation	% Exhaustion	%Fixation	%Exhaustion	%Fixation
D_1	70.15	89.90	75.18	83.80	67.00	85.82
D_2	74.00	87.83	74.25	85.52	68.80	85.75
D_3	70.10	86.30	76.80	86.58	63.93	86.04
D_4	74.28	85.49	77.83	84.81	66.12	85.44
D_5	70.55	88.58	71.85	86.29	62.00	85.48
D_6	75.20	90.36	68.22	87.94	68.10	87.37
D_7	78.00	89.10	79.40	90.05	69.05	87.62
D_8	71.80	91.23	69.82	84.02	76.25	85.90
D_9	77.15	84.25	70.25	86.83	68.22	85.74
D_{10}	72.00	87.50	66.45	91.05	70.80	86.86
D_{11}	67.65	89.40	73.95	83.16	74.15	83.61
D_{12}	75.85	86.35	73.20	84.69	70.85	81.86
D_{13}	67.00	91.79	71.80	87.05	76.10	84.76
D_{14}	73.25	86.88	79.30	80.01	76.62	85.48
D_{15}	72.00	88.88	75.85	80.35	74.55	83.16
D_{16}	67.50	87.40	77.85	85.42	71.17	89.92
D_{17}	68.90	88.53	71.85	80.29	72.60	87.47
D_{18}	72.82	91.31	73.98	87.86	75.67	83.25
D_{19}	70.15	89.90	65.10	90.62	69.00	83.33
D_{20}	74.10	78.27	73.93	87.92	64.50	84.49

Table 5: Fastness properties of acid dyes on wool, silk and nylon

Dye	Wool				Silk				Nylon			
No.	Light	Wash	Rubbing		Light	Wash	Rubbing		Light	Wash	Rubbing	
			Dry	wet			Dry	wet			Dry	wet
D_1	4	3	5	4	3-4	3-4	3	3-4	4	3	4	5
D_2	4	3-4	4	3	3	3-4	3	3	4	4	4	4
D_3	3	2	3-4	3	5-6	3	3	3-4	3-4	4-5	4-5	4
D_4	3-4	3-4	3	3-4	3-4	3	3-4	4	4	5	4	3
D_5	3	4	4	3	5	4-5	3-4	3	3-4	3-4	3	3-4
D_6	3-4	3-4	3	3	4-5	4	3-4	4-5	4	4	3-4	3
D_7	3	4	3-4	4	3-4	3-4	4	3	4-5	3	3	3
D_8	4	3	3	3-4	4	3	3-4	4	3-4	4	3-4	3-4
D_9	3-4	3	4	3-4	4	3	4	4	3	3	3-4	4
D_{10}	4	4	3	3	3-4	3	3-4	3-4	4	4	3	3
D_{11}	5-6	3	3-4	3-4	5	3-4	4	3-4	5	4	3-4	3-4
D_{12}	3	3-4	3-4	5	4-5	4-5	3	4	4	4-5	4	4
D_{13}	4	3	4	3	3	4	3-4	3	4-5	4	3-4	4
D_{14}	4	4	3-4	5	3	3-4	4	3-4	3-4	3-4	3	4
D_{15}	4-5	5	3	2-3	4-5	3-4	3-4	4	4	3	4	3
D_{16}	4	3-4	3-4	3-4	4	3	4	3-4	5	5	4	4
D_{17}	3	3-4	3-4	3	4	3	3-4	3	3-4	4	3	3-4
D_{18}	4	4	4	3-4	5	4	4	3-4	4	3	3-4	3
D_{19}	4	3-4	4	3-4	4-5	4	4	3-4	5-6	4	4	3-4
D_{20}	3	3-4	3-4	3-4	4	3	4	3-4	3	4-5	3	4

Light: Poor - 1, Slight - 2, Moderate - 3, Fair -4, Good - 5, Very good - 6 and Excellent - 7

Wash and Rubbing: Poor - 1, Slight - 2, Moderate - 3, Fair -4, Good - 5, Very good - 6 and Excellent - 7

Table 6: Calibration data for exhaustion study of acid dyes

Subtrate for dyeing : Wool (2.0g), Silk (2.0g) and Nylon (2.0g)
Medium of spectral study ; Aqueous

Dye No.	wave lengh (mm)	Absorbance of dye solution at specified wavelentgh Conc. × 10^{-3} mg.ml^{-1}				slop of linear
		4.0	8.0	12.0	16.0	plot K*
D_2	470	0.060	0.120	0.180	0.240	15.0
D_{12}	489	0.090	0.180	0.271	0.360	22.5
D_{20}	435	0.110	0.220	0.329	0.44	27.5

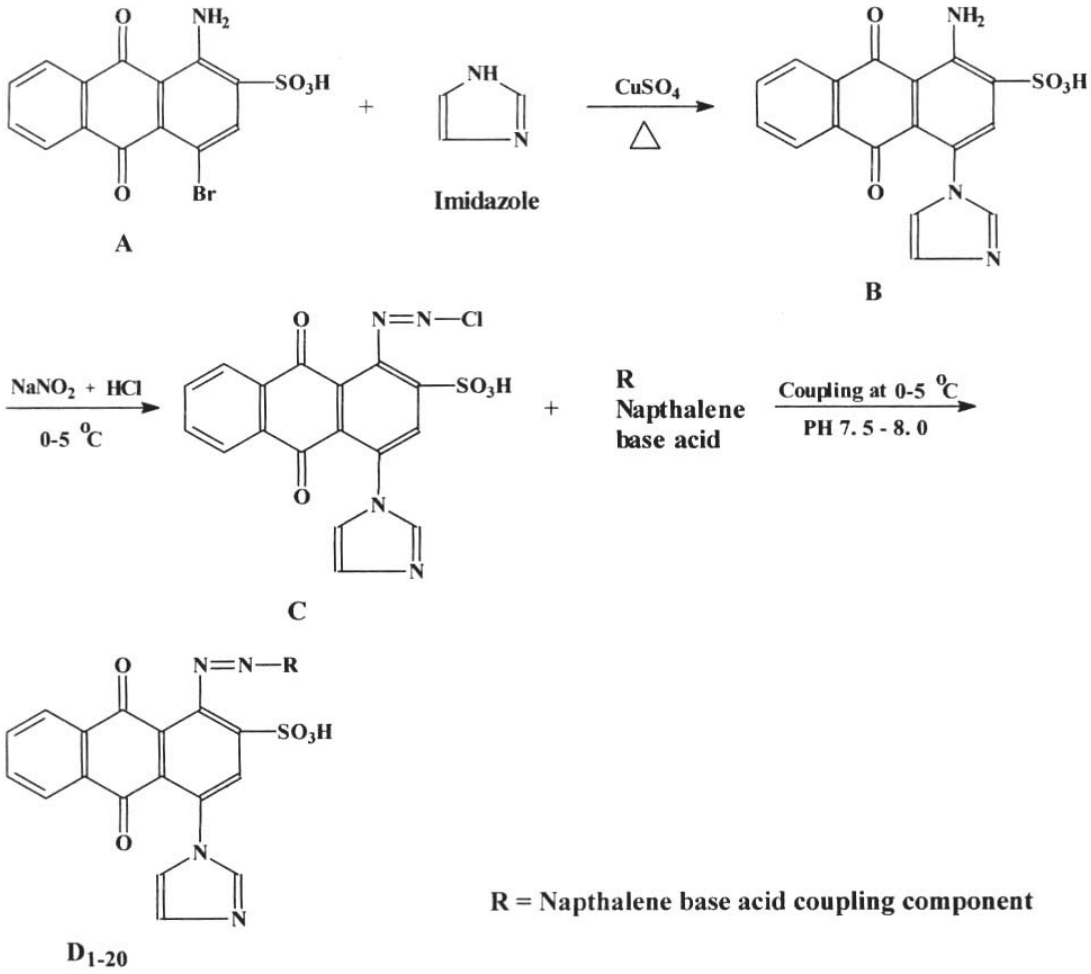
Absorbance = K* (Conc. × 10^{-3} mg.ml^{-1})

Table 7: Calibration data for fixation study of acid dyes

Subtrate for dyeing : Wool (2.0g), Silk (2.0g) and Nylon (2.0g)
Medium of spectral study ; Conc. sulfuric acid

Dye No.	wave lengh (mm)	Absorbance of dye solution at specified wavelentgh Conc. × 10^{-3} mg.ml^{-1}				slop of linear
		4.0	8.0	12.0	16.0	plot K*
D_6	478	0.048	0.096	0.143	0.192	12.00
D_{13}	489	0.090	0.180	0.270	0.360	22.5
D_{18}	480	0.066	0.132	0.197	0.264	16.5

Absorbance = K* (Conc. × 10^{-3} mg.ml^{-1})



Scheme 1

and on nylon fabric shows exhaustin 62.00% to 76.62%. The percentage fixation of 2% dyeing on wool fabric shows fixation 78.27% or 91.79%, for silk fabric shows fixation 80.01% to 91.05% and for nylon fabric shows fixation 81.86% to 89.92%. Exhaustion and Fixation data of D_1-D_{20} on wool, silk and nylon are given in Table 4.

Fastness properties

All the dyes give fair to very good light fastness on wool, silk and nylon. All the dyes give good to excellent fastness to washing and rubbing on each fiber. Fastness properties data of D_1-D_{20} are given in Table 5.

Microbial studies

All the acid anthraquinone dyes are inactive against both Gram Positive (*Pseudomonas* Sp. *& B. subtilis*) and Gram Negative (*Ceretium* & *E. coli*) bacteria at 100µg/ml, and 200µg concentration compared to Penicillin, Ampicillin and Amoxicillin.

All the acid anthraquinone dyes are inactive against *C. albicans* at 100µg/ml and 200µg concentration compared to Amphotericine-B.

CONCLUSIONS

The difference in the colour of newly synthesized dyes depends upon the position of the substituents present and/or position of the substituents on the naphthalene ring. The dyes D_{11} and D_{15} gave fair to very good light fastness on wool, while D_5 and D_{12} gave fair to very good wash and rubbing fastness on silk. The dyes D_7, D_{12} and D_{19} gave fair to very good rubbing fastness on nylon.

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