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Research Article

SYNTHESIS OF HOT BRAND REACTIVE DYES AND THEIR DYEING PERFORMANCE ON VARIOUS FIBRES

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ABSTRACT

Some hot brand reactive dyes have been synthesized by coupling of tetrazotized 4,4'-methylene bis(2-nitro aniline) and coupled with two moles of various 2-((4-amino-3-methoxyphenyl) sulphonyl)ethyl hydrogen sulphato cyanurated coupling components such as H-acid, Gamma acid, S-acid, Peri acid, J-acid, N-Methyl J-acid, N-Phenyl J-acid, N-Benzoyl J-acid, K-acid, Koch acid, Bronner acid, Laurent acid, Cleve acid and Tobias acid. They were characterized by nitrogen elemental analysis, IR and ¹H NMR spectra. The dyeing performance of all these dyes on wool, silk and cotton fibres gave fair to good light fastness, good to excellent wash fastness and rubbing fastness. Spectral properties and colorimetric data (L*, a*, b*, C*, H* and K/S) have also been investigated.

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INTRODUCTION

Reactive dyes constitute a very important class for dyeing cellulosic fibre and a high rate of growth is predicted in future. These dyes are coloured compounds which contain one or two groups capable of forming covalent bonds between a carbon or phosphorous atom of the dye ion or molecule and an oxygen, nitrogen or sulphur atom of a hydroxyl, an amino or a mercapto group, respectively, of the substrate. Such covalent bonds are formed with the amino, hydroxyl groups of cellulosic fibres, with the amino, hydroxyl and mercapto groups of protein fibres and with the amino groups of poly-amides (Zollinger H., 1991). In general, reactive dyes are the only textile colourants designed to bond covalently with the substrate on application. They are used for the dyeing and printing of cellulose and to a lesser extent polyamides fibres.

The first commercial reactive dyes for cellulose were synthesized by Rattee and Stephen and Marketed by ICI in 1956 under the trade name Procion M. (Lewis D. M., 1974; Stead C.V., 1969; Rouette et al., 1971; Lewis et al., 2000; Rattee et al., 1960; Dolby P. J., 1976). Several new reactive system have been introduced from time to time, which covers the subject of innumerable patents and publications (JP, 1981; Seigel E., 1972; BP, 1967; EGP, 1968). Over the last

decade a number of major changes have been taken place, primary aimed at minimizing the major weakness of reactive dyes for facilitating new application condition. In this period, cold brand reactive dyes (Patel et al., 1996; Patel et al., 2002; Mehta et al., 2008) and hot brand reactive dyes (Patel et al., 2014; Patel et al., 2015; Patel et al., 2015; Patel et al., 2015) have been established as a major group for fixation to cellulose.

MATERIALS AND METHODS

The melting points of the compounds were determined using open capillary and are uncorrected. The purity of dyes was determined by thin-layer chromatography using silica gel-G coated Al plates. IR spectra were recorded on SHIMADZU, Model: FTIR 8400S using KBr pellets. ¹H NMR spectra were obtained on Bruker Avance-II 400 NMR spectrometer using D₂O solvent and TMS as internal reference (chemical shifts in , ppm). Colorimetric data (L*, a*, b*, C*, H* and K/S) were recorded on Reflectance spectrophotometer Gregtag Macbeth Color-Eye 7000A.

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Synthesis of 4,4'-methylene bis(2-nitro aniline)(I) (Patel et al., 2010)

2-Nitro aniline (13.8 g, 0.1mol) was dissolved in water (125 ml) and 36.5% hydrochloric acid (25 ml) at 50°C. The reaction mixture was then treated with 3% aqueous formaldehyde solution (35ml) at 60°C with stirring for an hour and neutralized with 10% sodium hydroxide to give yellow precipitates of 4,4'-methylene bis(2-nitro aniline). It was filtered, washed with hot water, dried and recrystallized from ethanol. Yield: 86 %, m.p. 247°C.

Tetrazotization of 4,4'-methylene bis(2-nitro aniline)(II)

4,4'-Methylene bis(2-nitro aniline) (2.88 g, 0.01 mol) was suspended in H₂O (60 ml). Conc. hydrochloric acid (10 ml) was added dropwise to this well stirred suspension. The mixture was gradually heated up to 70°C, till clear solution was obtained. The solution was cooled to 0-5°C in an ice bath. A solution of NaNO₂ (1.38 g, 0.02mol) in water (8 ml) was added to the above solution at 0°C temperature over a period of five minutes with continuous stirring. The stirring was continued for an hour, maintaining the same temperature, with positive test for nitrous acid on starch iodide paper. The excess of nitrous acid was destroyed with required amount of sulphamic acid solution in water. The clear tetrazotized solution at 0-5°C was obtained and used for subsequent coupling reaction.

Synthesis of 2-((4-amino-3-methoxyphenyl)sulphonyl)ethyl hydrogen sulphato cyanurated H-acid(III)

Cyanuration of H-acid

Cyanuric chloride (3.69 g, 0.02mol) was stirred in acetone (50 ml) at a temperature below 5 °C for a period of an hour. A neutral solution of H-acid (6.38 g, 0.02mol) in aqueous sodium carbonate solution (10% w/v) was then added in small lots in about an hour. The pH was maintained neutral by simultaneous addition of sodium carbonate solution (1% w/v). The reaction mass was then stirred at 0-5 °C for further 4 hours. The solution was used for subsequent coupling reaction.

Condensation with 2-((4-amino-3-methoxyphenyl) sulphonyl) ethyl hydrogen sulphate

The temperature of ice-cooled well stirred solution of cyanurated H-acid was gradually raised to 45°C. 2-((4-Amino-3-methoxyphenyl)sulphonyl)ethyl hydrogen sulphate (6.22 g, 0.02 mol) was added slowly to the cyanurated H-acid solution at same temperature during a period of 30 minutes, maintaining the pH neutral by simultaneous addition of sodium carbonate solution (1% w/v). After the addition was completed, stirring was continued for further 3 hours. The 2-((4-amino-3-methoxyphenyl) sulphonyl)ethyl hydrogen sulphato cyanurated H-acid solution thus obtained was subsequently used for further coupling reaction.

Coupling of tetrazotized solution with 2-((4-amino-3-methoxyphenyl) sulphonyl) ethyl hydrogen sulphato cyanurated H-acid coupling component:

Synthesis of dyes (D₁ to D₁₄)

The above mentioned freshly prepared tetrazotized solution was added to an ice cooled and well stirred solution of 2-((4-amino-3-methoxyphenyl)sulphonyl)ethyl hydrogen sulphato

cyanurated H-acid over a period of 10-15 minutes. The pH was maintained at 8.0 to 8.5 by simultaneous addition of sodium carbonate solution (10% w/v) and stirring was continued for 4 hours. Sodium chloride (15 g) was then added and the mixture was stirred for an hour. The solid dye separated out was filtered, washed with minimum amount of acetone and dried at room temperature. Following the above procedure, other reactive dyes **D₂** to **D₁₄** were synthesized by using 2-((4-amino-3-methoxyphenyl) sulphonyl)ethyl hydrogen sulphato cyanurated coupling components such as Gamma acid, S-acid, Peri acid, J-acid, N-Methyl J-acid, N-Phenyl J-acid, N-Benzoyl J-acid, K-acid, Koch acid, Bronner acid, Laurent acid, Cleve acid and Tobias acid.

RESULT AND DISCUSSION

Characterization

All the synthesized dyes have been characterized by Visible absorption spectra, some representative IR spectra and PMR spectra.

Visible Absorption Spectra

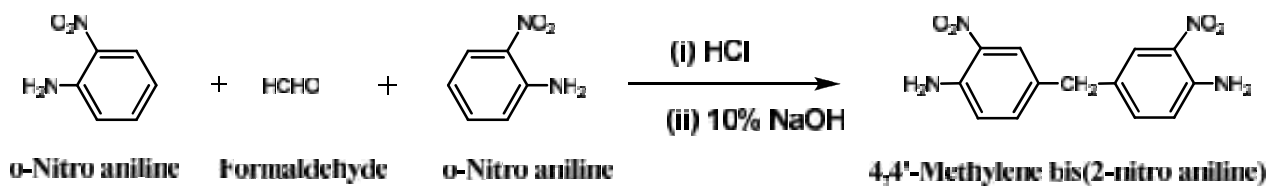
The visible absorption spectra of all synthesized dyes have been recorded on in water and observed in the range of 535-410 nm. The colour observed for each dye is due to the oscillation of electrons and presence of substituents.

IR Spectra

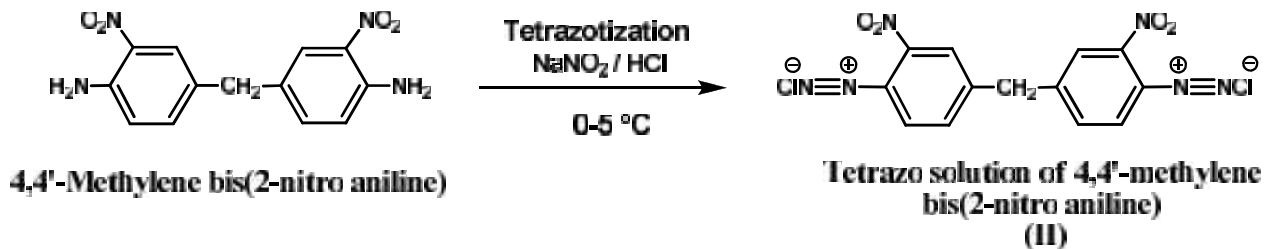
IR spectra of dye **D₁** showed O-H stretching vibration of -OH group at 3580 cm⁻¹, N-H stretching vibration of secondary amine at 3423 cm⁻¹, C-H stretching vibration of -OCH₃ and -CH₂- groups at 2930 cm⁻¹ and 2852 cm⁻¹, C=C stretching vibration of aromatic ring at 1622 cm⁻¹ and 1422 cm⁻¹, N=N stretching vibration of azo group at 1450 cm⁻¹, C-N stretching vibration of tertiary amine group at 1300 cm⁻¹, S=O stretching vibration of SO₂ group at 1318 cm⁻¹ and 1150 cm⁻¹, S=O stretching vibration of -SO₃Na group at 1194 cm⁻¹ and 1077 cm⁻¹, C-Cl stretching vibration of chloro group at 740 cm⁻¹. IR spectra of dye **D₂** showed O-H stretching vibration of -OH group at 3580 cm⁻¹, N-H stretching vibration of secondary amine at 3444 cm⁻¹, C-H stretching vibration of -OCH₃ and -CH₂- groups at 2911 cm⁻¹ and 2850 cm⁻¹, C=C stretching vibration of aromatic ring at 1622 cm⁻¹ and 1420 cm⁻¹, N=N stretching vibration of azo group at 1450 cm⁻¹, C-N stretching vibration of tertiary amine group at 1314 cm⁻¹, S=O stretching vibration of SO₂ group at 1340 cm⁻¹ and 1155 cm⁻¹, S=O stretching vibration of -SO₃Na group at 1185 cm⁻¹ and 1083 cm⁻¹, C-Cl stretching vibration of chloro group at 741 cm⁻¹.

IR spectra of dye **D₅** showed O-H stretching vibration of -OH group at 3580 cm⁻¹, N-H stretching vibration of secondary amine at 3429 cm⁻¹, C-H stretching vibration of -OCH₃ and -CH₂- groups at 2912 cm⁻¹ and 2850 cm⁻¹, C=C stretching vibration of aromatic ring at 1621 cm⁻¹ and 1423 cm⁻¹, N=N stretching vibration of azo group at 1457 cm⁻¹, C-N stretching vibration of tertiary amine group at 1310 cm⁻¹, S=O stretching vibration of SO₂ group at 1350 cm⁻¹ and 1150 cm⁻¹, S=O stretching vibration of -SO₃Na group at 1184 cm⁻¹ and 1077 cm⁻¹, C-Cl stretching vibration of chloro group at 741 cm⁻¹.

Reaction scheme

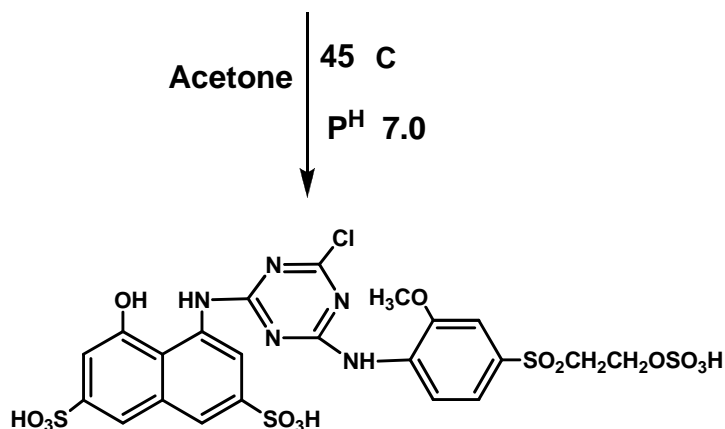
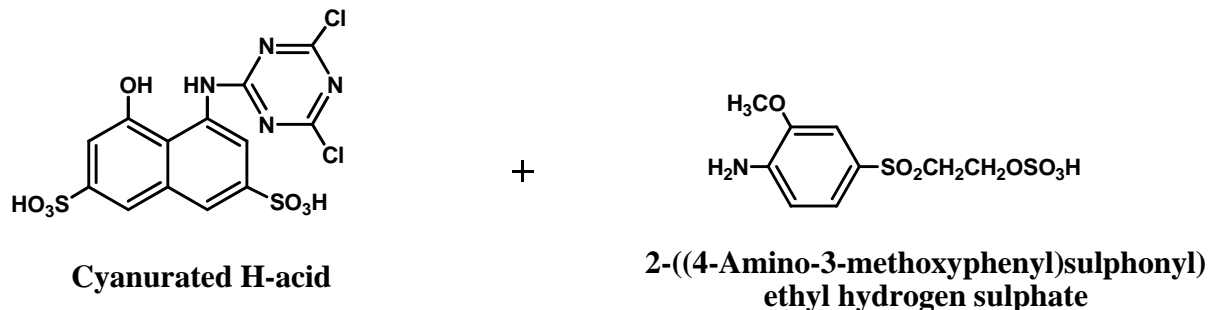
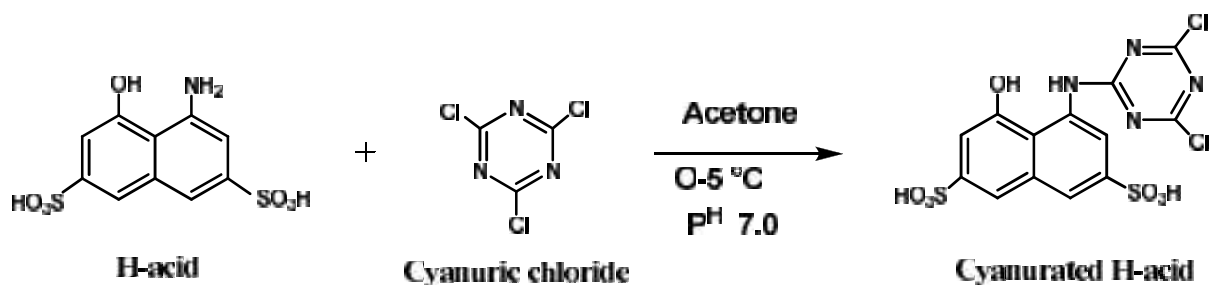


(I)

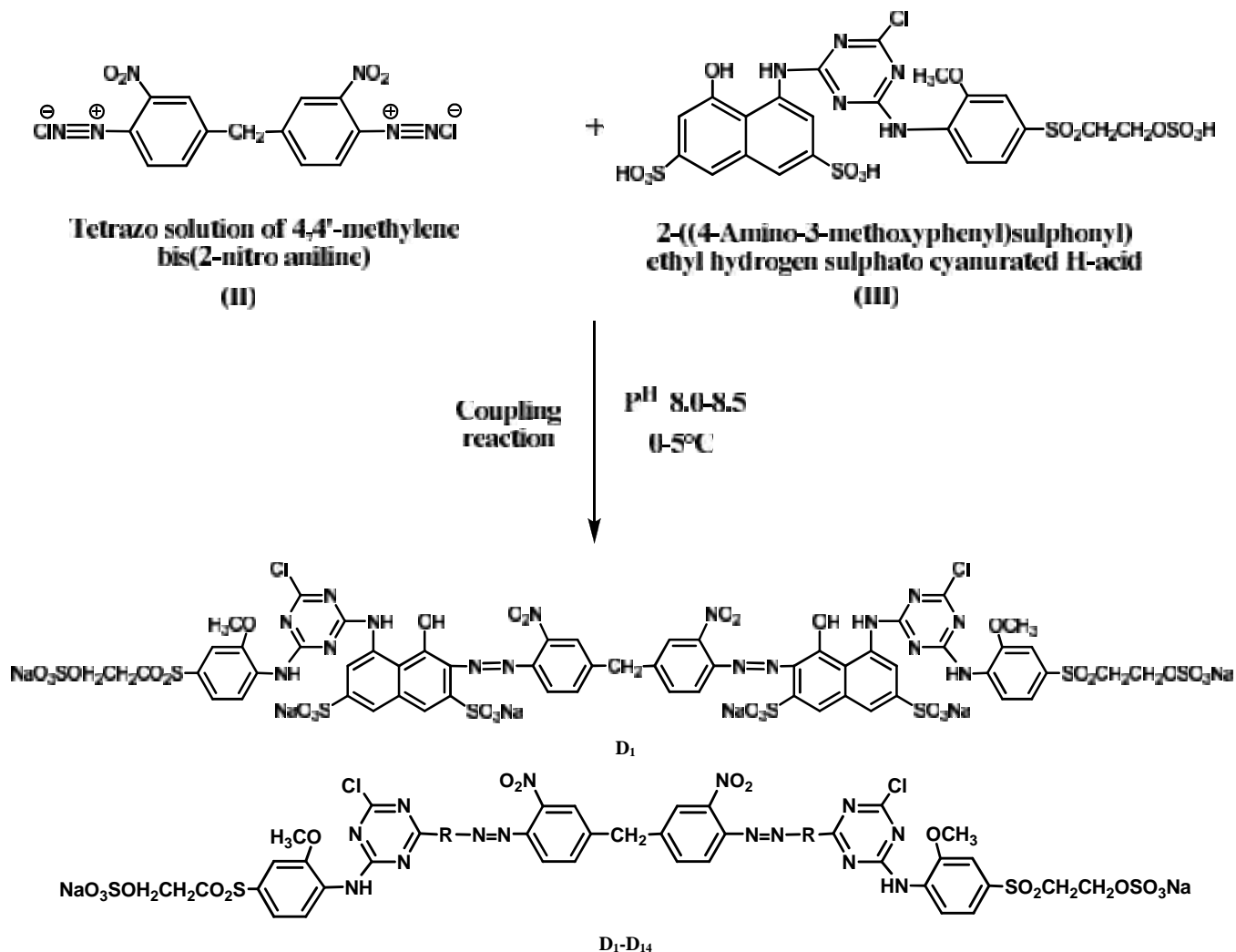


(II)

(II)



2-((4-Amino-3-methoxyphenyl)sulphonyl)ethyl hydrogen sulphato cyanurated H-acid (III)



Where, R= various coupling components to synthesize D₁ to D₁₄ (Table-1)

¹H NMR Spectra

¹H NMR Spectra of dye D₁ showed signals at 1.98(s, 6H, -OCH₃), 3.38-3.41(t, 4H, -CH₂-protons), 3.56(s, 2H, -CH₂-protons), 3.78(s, 2H, -NH-protons), 3.88-3.91(t, 4H, -CH₂-protons), 4.70(s, 2H, -OH proton) and 6.68-8.48(m, 18H, aromatic protons).

Application

Dyeing of fibres

All the dyes were applied on silk, wool and cotton fibres by using the standard procedure (Shenai V.A., 1973).

Table-1 Characterization of hot brand reactive dyes

Dye No.	Coupling component (R)	Molecular Formula	Mol. Weight g/mol	Yield (%)	M.P. °C	Nitrogen %		Rf Value
						Found	Req.	
D ₁	H-acid	C ₅₇ H ₄₀ N ₁₆ S ₈ O ₃₂ Cl ₂ Na ₆	1926	80	>300	11.58	11.64	0.57
D ₂	Gamma acid	C ₅₇ H ₄₂ N ₁₆ S ₆ O ₂₆ Cl ₂ Na ₄	1722	79	>300	12.96	13.01	0.56
D ₃	S-acid	C ₅₇ H ₄₂ N ₁₆ S ₆ O ₂₆ Cl ₂ Na ₄	1722	84	>300	12.97	13.01	0.58
D ₄	Peri acid	C ₅₇ H ₄₂ N ₁₆ S ₆ O ₂₄ Cl ₂ Na ₄	1690	81	>300	13.20	13.26	0.59
D ₅	J-acid	C ₅₇ H ₄₂ N ₁₆ S ₆ O ₂₆ Cl ₂ Na ₄	1722	80	>300	12.95	13.01	0.54
D ₆	N-Methyl J-acid	C ₅₉ H ₄₆ N ₁₆ S ₆ O ₂₆ Cl ₂ Na ₄	1750	77	>300	12.75	12.81	0.63
D ₇	N-Phenyl J-acid	C ₆₉ H ₅₀ N ₁₆ S ₆ O ₂₆ Cl ₂ Na ₄	1875	83	>300	11.90	11.95	0.52
D ₈	N-Benzoyl J-acid	C ₇₁ H ₅₀ N ₁₆ S ₆ O ₂₈ Cl ₂ Na ₄	1931	80	>300	11.55	11.61	0.56
D ₉	K-acid	C ₅₇ H ₄₀ N ₁₆ S ₈ O ₃₂ Cl ₂ Na ₆	1926	79	>300	11.56	11.64	0.59
D ₁₀	Koch acid	C ₅₇ H ₃₈ N ₁₆ S ₁₀ O ₃₆ Cl ₂ Na ₈	2098	83	>300	10.62	10.68	0.60
D ₁₁	Bronner acid	C ₅₇ H ₄₂ N ₁₆ S ₆ O ₂₄ Cl ₂ Na ₄	1690	79	>300	13.20	13.26	0.62
D ₁₂	Laurent acid	C ₅₇ H ₄₂ N ₁₆ S ₆ O ₂₄ Cl ₂ Na ₄	1690	84	>300	13.18	13.26	0.54
D ₁₃	Cleve acid	C ₅₇ H ₄₂ N ₁₆ S ₆ O ₂₄ Cl ₂ Na ₄	1690	76	>300	13.22	13.26	0.53
D ₁₄	Tobias acid	C ₅₇ H ₄₄ N ₁₆ S ₄ O ₁₈ Cl ₂ Na ₂	1486	75	>300	15.0	15.07	0.50

Exhaustion and fixation study

The percentage exhaustion of 2% dyeing on silk fabric ranges from 63-75%, for wool ranges from 65-76 % and for cotton ranges from 67-80%. The percentage fixation of 2% dyeing on silk fabric ranges from 85-91 %, for wool ranges from 85-90% and for cotton ranges from 85-94% (Table-2).

spectrophotometer were calculated at max and are directly correlated with the dye concentration on the substrate according to the Kubelka–Munk equation (Billmeyer *et al.*, 1981; Volz H.G., 1995). The colorimetric data of silk, wool and cotton fibres for all dyes are summarized in Table-4.

Table-2 Dyeing performance of Dyes D₁ to D₁₄

Dye No.	Shade on silk fabric	Shade on wool fabric	Shade on cotton fabric	max	% Exhaustion			% Fixation		
					S	W	C	S	W	C
D ₁	Maroon red	Maroon	Purple	535	75.55	76.25	79.50	90.73	89.84	94.34
D ₂	Dark brown	Dark brown	Brown	525	72.00	74.50	78.75	88.19	89.26	92.70
D ₃	Light pink	Light pink	Light pink	410	66.00	71.75	70.25	85.61	86.41	87.54
D ₄	Light orange	Orange	Pale orange	475	71.50	72.00	71.75	86.71	85.42	85.02
D ₅	Orange	Light orange	Pink	495	70.75	73.00	72.00	88.34	89.73	91.67
D ₆	Dark orange	Light maroon	Pink	505	73.25	74.25	70.25	88.04	88.89	86.12
D ₇	Dark brown	Dark brown	Light purple	535	71.00	74.00	73.00	86.62	87.84	91.10
D ₈	Orange	Dark orange	Light orange	495	74.25	73.25	68.75	89.56	89.42	87.27
D ₉	Cream	Turmeric yellow	Cream	410	68.75	70.75	67.00	86.55	86.22	86.57
D ₁₀	Light cream	Cream	Light cream	410	67.25	69.25	70.75	86.24	85.92	86.93
D ₁₁	Light yellow	Light orange	Peech	410	66.25	68.00	69.50	86.04	86.03	87.05
D ₁₂	Light ivory	Pale yellow	Peech	410	65.50	67.00	68.25	86.26	85.82	86.45
D ₁₃	Brown	Brown	Light orange	480	64.75	70.00	71.25	86.49	87.32	88.42
D ₁₄	Cream	Light yellow	Cream	410	63.50	65.75	69.00	85.83	85.17	86.23

S= silk, W= wool and C=cotton

Fastness properties

Fastness to light was assessed in accordance with BS: 1006-1978(Standard Test Method, 1994). The rubbing fastness test was carried out by using Crock meter (Atlas) in accordance with the AATCC-1961(AATCC Test, 1961) and the wash fastness was carried out in accordance with IS:765-1979 (Indian Standard,1979). All the dyes showed generally fair to good light fastness properties. The washing and rubbing fastness properties ranges from very good to excellent fastness on silk, wool and cotton (Table-3).

The K/S value graph of all the dyes is shown in Figure-1 and graph of b* versus a* for silk, wool and cotton fibres are shown in Figure-2, 3 and 4 respectively.

For silk fibre, the data summarized in Table-4 showed that dye D₁₄ is more lighter and D₇ is less lighter, D₈ is more redder and D₁₄ is less redder, D₈ is more yellower and D₁ is less yellower, D₈ is more brighter and D₇ is less brighter, D₁₄ has more K/S value and D₇ has less K/S value.

Table-3 Fastness properties of Dyes D₁ to D₁₄

Dye No.	Light fastness			Wash fastness			Rubbing fastness					
	S	W	C	S	W	C	Dry			Wet		
							S	W	C	S	W	C
D ₁	6	5-6	5	4-5	5	4-5	4	5	5	5	4-5	5
D ₂	5-6	4-5	5-6	5	4-5	4-5	3-4	4	5	4-5	4-5	4-5
D ₃	4	3-4	3-4	4	3-4	3-4	5	4	4	3-4	3-4	4
D ₄	4-5	5	3-4	4-5	5	3-4	4	5	4	4	5	3
D ₅	5-6	4-5	4	4-5	4-5	4-5	4	4-5	4	5	4-5	4
D ₆	5	5	4-5	4-5	4-5	4	4	4-5	4	4-5	4	4
D ₇	5	4-5	4-5	4-5	4	5	4-5	4	5	4	4	4
D ₈	5-6	5-6	4-5	4-5	6	4	5	4-5	4-5	4-5	4-5	4
D ₉	4-5	3-4	3-4	4	3-4	3-4	3-4	3-4	4	5	3-4	3-4
D ₁₀	3-4	4	3	4	4	3	3-4	4	4	3-4	4	4
D ₁₁	4	4-5	4	4	4-5	4	3-4	5	4	4	4-5	4
D ₁₂	4	4	4	4	3-4	4	4	4	3-4	3-4	4	4
D ₁₃	6	4-5	4	4-5	4	4	4-5	3-4	3-4	4	3-4	4
D ₁₄	5	4	4	4-5	4	4	4	3-4	4	3-4	3-4	4

S= silk, W= wool and C=cotton

Light fastness: 1-Poor, 2-Slight,3-Moderate, 4-Fair , 5-Good, 6-Very good,7-Excellent,8-Maximum

Wash and Rubbing fastness: 1-Poor, 2-Fair,3-Good, 4-Very good, 5-Excellent

Computer Colour Matching Data (CCM)

The colour on silk, wool and cotton fibres are expressed in terms of CIELab values and the coordinates measured are lightness (L*), chroma (C*), hue angle from 0° to 360°(H), a* value represent the degree of redness (positive) and greenness (negative) and b* represents the degree of yellowness (positive) and blueness (negative). K/S values given by the reflectance

For wool fibre, the data showed that dye D₁₄ is more lighter and D₇ is less lighter, D₈ is more redder and D₁₄ is less redder, D₈ is more yellower and D₁ is more bluer, D₈ is more brighter and D₃ is less brighter, D₁₄ has more K/S value and D₇ has less K/S value.

Table-4 Color measurement (CIELab) data of Dyes D₁ to D₁₄

Dye No.	L*			a*			b*			C*			H*			K/S		
	S	W	C	S	W	C	S	W	C	S	W	C	S	W	C	S	W	C
D ₁	32.15	30.38	52.05	36.45	33.19	25.63	1.84	-0.05	-7.64	36.50	33.19	26.74	2.89	359.92	343.40	3.38	3.18	14.16
D ₂	26.09	22.17	33.28	21.51	21.19	17.87	8.09	8.36	6.41	22.98	22.78	18.98	20.60	21.53	19.73	3.04	2.14	5.54
D ₃	59.50	57.07	76.37	16.60	17.94	7.57	6.08	9.51	1.53	17.67	20.31	7.73	20.11	27.92	11.45	19.45	15.52	45.49
D ₄	51.12	48.49	66.21	33.86	33.19	16.88	39.25	41.32	17.33	51.83	53.00	24.19	49.22	51.23	45.75	5.01	3.90	23.52
D ₅	56.16	59.45	67.34	31.10	27.67	20.04	25.26	22.45	11.65	40.07	35.63	23.18	39.09	39.06	30.18	11.22	14.38	26.25
D ₆	44.27	42.44	65.05	34.66	35.41	22.41	18.56	18.72	5.69	39.31	40.15	23.12	28.17	27.80	14.25	5.77	4.99	23.81
D ₇	23.65	21.09	48.98	16.83	20.68	13.93	2.81	4.94	-4.17	17.06	21.26	14.54	9.49	13.43	343.34	2.95	2.11	14.62
D ₈	49.56	56.46	72.25	50.03	40.83	18.26	43.11	42.62	14.64	66.06	59.02	23.40	40.75	46.23	38.73	3.53	6.20	30.54
D ₉	64.51	60.22	82.29	15.02	16.58	6.80	36.96	38.19	17.00	39.90	41.63	18.31	67.89	66.53	68.22	9.99	7.09	39.61
D ₁₀	61.64	65.27	84.14	15.93	11.95	3.43	29.11	23.24	10.66	33.19	26.13	11.20	61.31	62.79	72.16	11.34	15.69	48.50
D ₁₁	62.63	54.75	80.48	23.05	26.73	11.52	33.40	38.55	20.10	40.58	46.92	23.16	55.40	55.26	60.18	11.20	5.84	36.69
D ₁₂	60.70	56.55	74.32	16.59	17.10	10.64	29.34	30.32	17.41	33.71	34.81	20.40	60.52	60.57	58.56	10.52	8.20	30.29
D ₁₃	35.52	33.92	62.95	30.38	31.11	16.49	23.36	27.91	15.16	38.32	41.80	22.40	37.55	41.90	42.59	3.32	2.26	21.46
D ₁₄	79.38	74.38	81.66	9.00	8.71	9.25	31.11	31.42	17.49	32.39	32.61	19.78	73.86	74.52	62.13	22.26	17.40	38.14

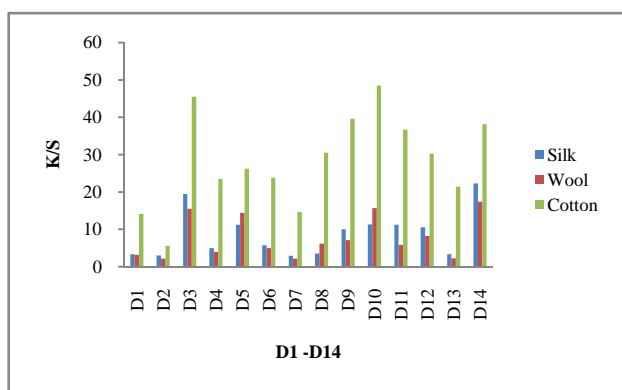


Figure-1 Graph of K/S values of dyes D₁ to D₁₄

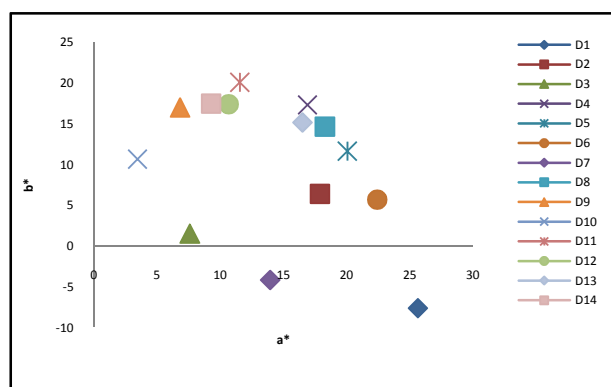


Figure-4 Graph of b* vs a* for cotton fibre

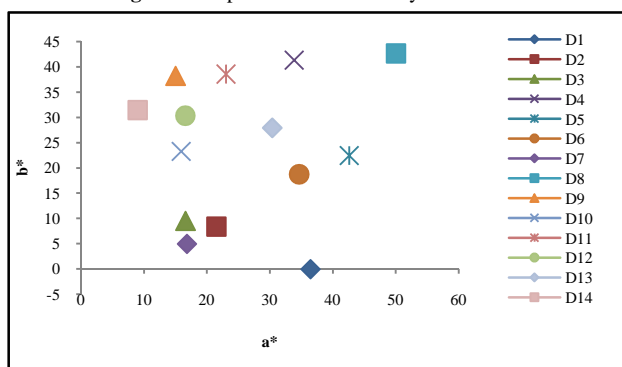


Figure-2 Graph of b* vs a* for silk fibre

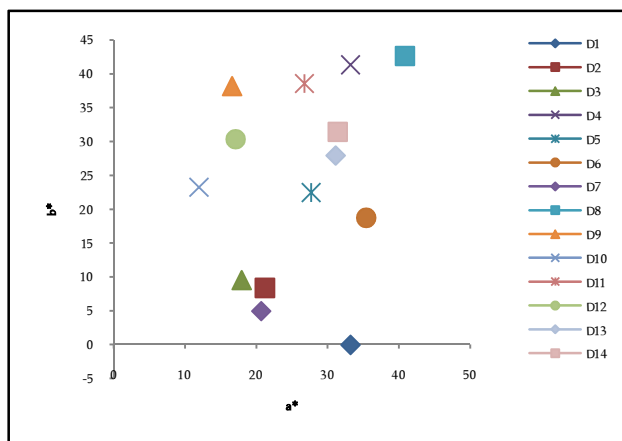


Figure-3 Graph of b* vs a* for wool fibre

For cotton fibre, the data showed that dye D₁₀ is more lighter and D₂ is less lighter, D₁ is more redder and D₁₀ is less redder, D₁₁ is more yellower and D₁ is more bluer, D₁ is more brighter and D₃ is less brighter, D₁₀ has more K/S value and D₂ has less K/S value.

CONCLUSION

4,4'-Methylene bis(2-nitro aniline) was tetrazotized and coupled with various 2-((4-amino-3-methoxy phenyl) sulphonyl)ethyl hydrogen sulphato cyanurated coupling components gave corresponding hot brand reactive dyes (D₁ to D₁₄). These dyes gave violet to yellow shade on silk, wool and cotton fibres and showed fair to good light fastness, good to excellent fastness to washing and rubbing fastness. Exhaustion and fixation of these dyes are very good in order. The presence of triazine group of dye molecule improves the exhaustion, fixation and fastness properties. D₁₄ has highest K/S value for silk fibre and wool fibre, D₁₀ has highest K/S value for cotton fibre. Thus, D₁₄ has more dye concentration on silk and wool fibre and D₁₀ has more dye concentration on cotton fibre.

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