



## **SYNTHESIS OF SOME NEW BROMINE CONTAINING REACTIVE DYES: THEIR APPLICATIONS AND MICROBIAL STUDIES**

**DIVYESH R. PATEL, AMIT L. PATEL, BHAVESH M. PATEL<sup>a</sup> and  
KESHAV C. PATEL<sup>\*</sup>**

Department of Chemistry, New Synthetic Organic Chemistry Research Laboratory, Veer Narmad South  
Gujarat University, SURAT - 395007 (Guj.) INDIA

<sup>a</sup>Naran Lala College of Professional & Applied Science, NAVSARI - 396450 (Guj.) INDIA

### **ABSTRACT**

Various hot brand reactive dyes have been prepared by coupling tetrazotised 4, 4'-methylene-bis-o-bromo aniline with various m-toluidino cyanurated coupling components and their dyeing performance as reactive dyes has been assessed on silk, wool and cotton fibres. The purity of all dyes has been checked by TLC. All the dyes were characterized by their percentage yield, UV-Visible spectra, elemental analysis, IR and <sup>1</sup>H-NMR spectroscopy. The percentage dye bath exhaustion of different fibres has found to be good and acceptable. The dyed fibres show fair to very good fastness to light, wash and rubbing. All the synthesized dyes have been screened for their antimicrobial activity.

**Key words:** Hot brand reactive dyes, Dyeing, Silk, Wool, Cotton, Microbial.

### **INTRODUCTION**

Reactive dyes constitute a very important class for dyeing cellulosic fibres and a high rate of growth is predicted in future. The most recent technological contribution to textile has been the development of reactive dyes.

Reactive dyes are unique as this is the only class of dyes that actually form a chemical bond with textile fibres and this bond gives them their excellent wash fastness properties. All the reactive dyes contain a chemical group that is attracted to the hydroxyl group in cellulosic fibres or to the amino group in protein fibres<sup>1</sup>.

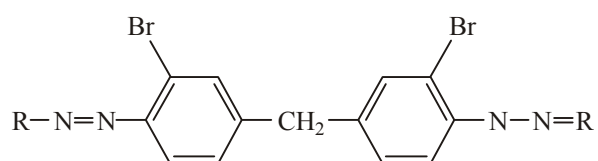
It was for the first time that dyeing has been done by chemical reaction between the

---

<sup>\*</sup> Author for correspondence; E-mail: divyeshpatel\_905@yahoo.com

dye and the fibre, enabling one to get assortment of bright, attractive shades of adequate fastness with considerable ease of dyeing. It can easily be understood that dyes with two reactive groups give a higher fixation yield than dyes with one reactive group, as one of the two dye fibre bond is hydrolysed and another is still left for fixation<sup>2,3</sup>.

Earlier we have studied bis azo reactive dyes<sup>4-6</sup> with good properties and compatibility with the fibres like silk, wool and cotton. Ayyangar et al.<sup>7,8</sup> reported that bis azo dyes are tinctorially stronger than mono azo dyes, making them potentially economical. Here, a series of bis azo reactive dyes of a general formula shown in Fig. 1 were synthesized and tested on silk, wool and cotton fibres. In addition to the characterization of the dyes, evaluations of their technical properties were also performed.



**Fig. 1: General structure formula**

Where R = Different m-toluidino cyanurated coupling components (Table 1)

## EXPERIMENTAL

All the chemicals used in the synthesis of dyes **D<sub>1</sub>-D<sub>12</sub>** were of commercial grade and were further purified by crystallization and distillation. All solvents used were of spectroscopic grade. Melting points (m.p.) were determined in open capillary method and are uncorrected. The purity of all the dyes was checked by thin layer chromatography<sup>9</sup>. IR spectra were recorded in KBr pellets on a Perkin-Elmer Model 881 spectrophotometer and <sup>1</sup>H NMR spectra on a Bruker DRX-300 instrument 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 were carried out on Carlo Erba 1108 instrument. The light fastness was assessed in accordance with BS: 1006-1978<sup>10</sup>. The rubbing fastness test was carried out with a Crockmeter (Atlas) in accordance with AATCC-1961<sup>11</sup> and the wash fastness test in accordance with IS: 765-1979<sup>12</sup>.

### Synthesis of 4, 4'-methylene bis-acetanilide (A)

A mixture of acetic anhydride (30 mL) and glacial acetic acid (60 mL) and 4,4'-methylene bis-aniline (17.2 g, 0.1 mole) was refluxed for 30 minutes on a hot water bath.

The product was precipitated by pouring the mixture into cold water and crystallized from 75% acetic acid. Yield 81 %, m.p. 236-237°C

### **Synthesis of 4, 4'-methylene bis-o-bromo acetanilide (B)**

4, 4'-Methylene bis-acetanilide (**A**) (0.282 g, 0.01 mole) was dissolved in acetic acid (30 mL) and bromine in acetic acid (2 mL, 10%) solution was added to it slowly with continuous stirring. The reaction mixture was kept for 4 hrs in water bath. Then it was treated with ice water. The solid separated was filtered, washed with sodium thiosulphate solution and then with water, dried and crystallized from absolute alcohol. Yield 78 %, m.p. 193°C.

### **Synthesis of 4, 4'-methylene bis-o-bromo aniline (C)**

4, 4'-Methylene bis-o-bromo acetanilide (**B**) (4.40 g, 0.01 mole) and 70% H<sub>2</sub>SO<sub>4</sub> was refluxed for 30 minutes on a hot water bath. The clear solution was poured into ice cold water and precipitated by adding excess of 10% NaOH solution and crystallized from alcohol. Yield 65 %, m.p. 110°C.

**IR (KBr) cm<sup>-1</sup>:** 3522, 3395 (-NH<sub>2</sub>), 2895 (-CH<sub>2</sub>), 3010 (= CH-), 650 (C-Br).

### **Tetrazotisation of 4, 4'-methylene bis-o-bromo aniline (D)**

4, 4'-Methylene bis-o-bromo aniline (**C**) (1.78 g, 0.005 mole) was suspended in water (60 mL). Hydrochloric acid (0.86 g) was added dropwise to this well stirred suspension. The mixture was gradually heated up to 70°C, till a clear solution was obtained. The solution was cooled at 0-5°C in an ice bath. A solution of NaNO<sub>2</sub> (0.7 g) in water (4 mL) (previously cooled to 0°C) was then added over a period of five minutes with stirring. The stirring was continued for an hour, maintaining the same temperature, with positive test for nitrous acid on starch iodide paper. After just destroying excess of nitrous acid with required amount of a solution of sulphamic acid, the clear tetrazo solution (**D**) at 0-5°C was used for subsequent coupling reaction.

### **Preparation of m-toluidino cyanurated H-acid**

The preparation of m-toluidino cyanurated H-acid involves two steps -

- (i) Cyanuration of H-acid (R') and
- (ii) Condensation with m-toluidine (Formation of m-toluidino cyanurated H-acid)

### Cyanuration of H-acid (**R'**)

Cyanuric chloride (1.85g, 0.01 mole) was stirred in acetone (25 mL) at a temperature below 5°C for a period of an hour. A neutral solution of H-acid (3.19 g, 0.01 mole) in aqueous sodium carbonate solution (10% w/v) was then added in small lots in about an hour. The pH was maintained to be neutral by simultaneous addition of sodium carbonate solution (1% w/v). The reaction mass was stirred at 0-5°C for further 4 hours. Then clear solution was obtained. The cyanurated H-acid solution (**R'**) was used for subsequent coupling reaction.

### Condensation with m-toluidine

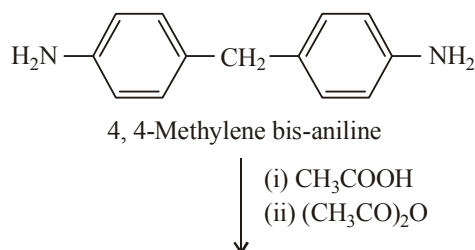
#### [Formation of m-toluidino cyanurated H-acid (**R**)]

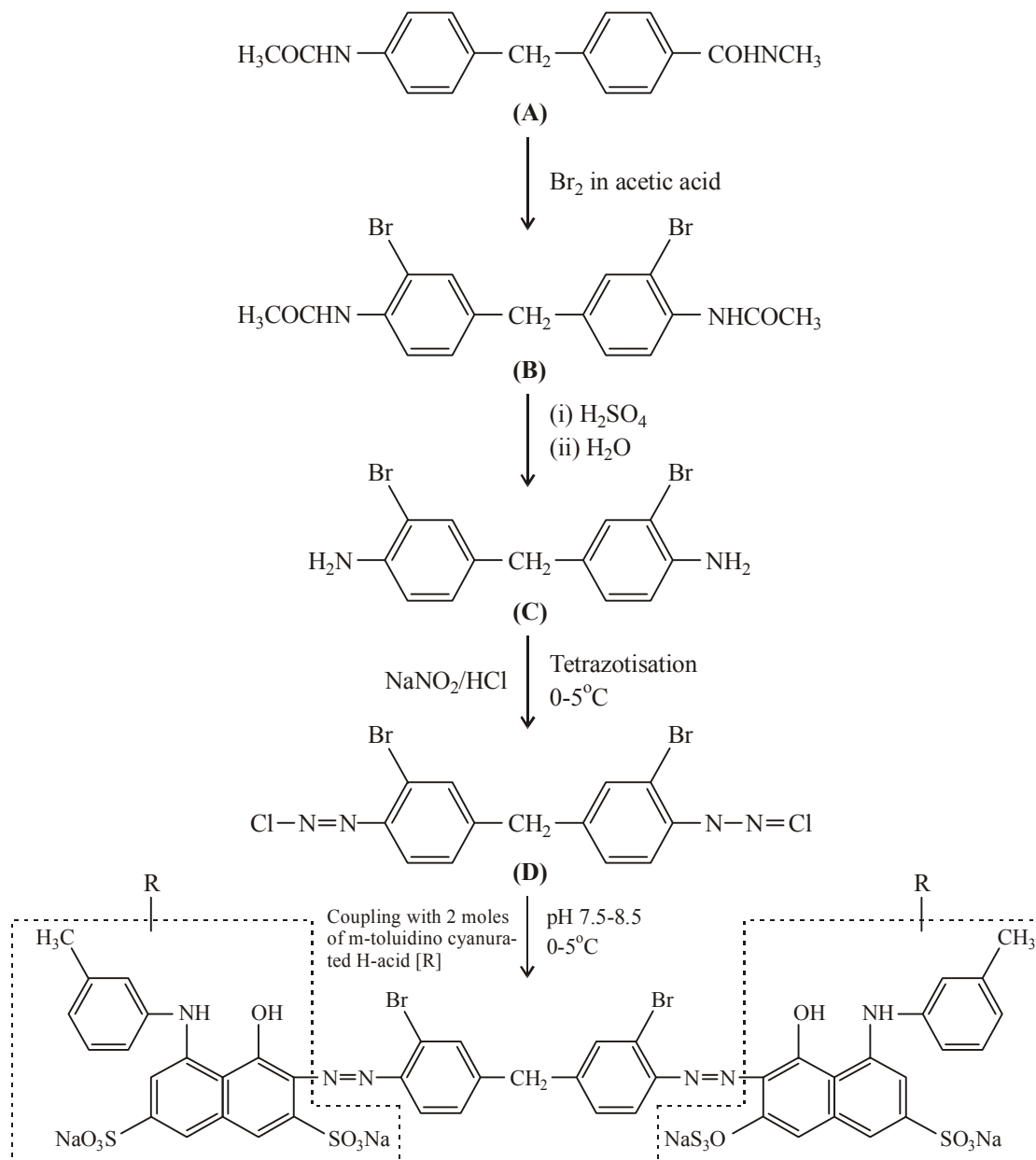
The temperature of ice-cooled well stirred solution of cyanurated H-acid (4.67 g, 0.01 mole) was gradually raised to 45°C for half an hour. To this cyanurated H-acid, the m-toluidine (1.07 g, 0.01 mole) was added dropwise at same temperature, during a period of 30 minutes, maintaining the pH at neutral by simultaneous addition of sodium bicarbonate solution (1% w/v). After the addition was completed, stirring was continued for further 3 hours. The m-toluidino cyanurated H-acid solution (**R**) thus obtained was subsequently used for further coupling reaction.

### Formation of dyes (**D**<sub>1</sub>)

To an ice cold and stirred solution of m-toluidino cyanurated H-acid (5.37 g, 0.01 mole) a freshly prepared solution of tetrazo solution (**D**) (as previously prepared) was added dropwise over a period of 10-15 minutes. The pH was maintained at 7.5 to 8.5 during addition and the stirring was continued for 4 hours, maintaining the temperature below 5°C. Sodium chloride (12 g) was then added and the mixture was stirred for an hour. The solid violet dye separated out, which was filtered, washed with minimum amount of acetone and dried at room temperature. Yield 85%

The same procedure was used to prepare other reactive dyes (**D**<sub>2</sub> to **D**<sub>12</sub>) using different m-toluidino cyanurated coupling components (Table 1).





**Reaction Scheme**  
**Synthesis of dyes (D<sub>1</sub> to D<sub>12</sub>)**

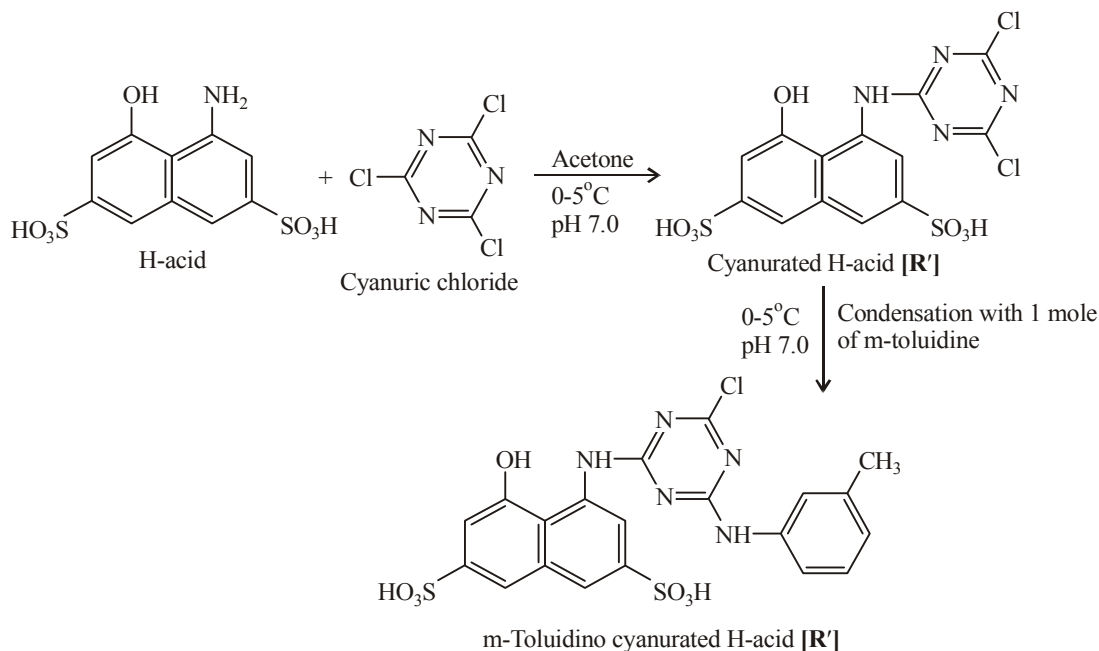
Where R = Various m-toluidino cyanurated coupling components for the formation of **D<sub>1</sub>** to **D<sub>12</sub>** (Table 1).

**Table 1: Characterisation data of reactive dyes (D<sub>1</sub> to D<sub>12</sub>)**

Dye No.	Coupling component (R)	Mol. formula	Mol. Wt.	Yield (%)	Elemental analysis Found (Required)			R <sub>f</sub> <sup>a</sup> value
					%C	%H	%N	
D <sub>1</sub>	H-acid	C <sub>53</sub> H <sub>34</sub> O <sub>14</sub> N <sub>14</sub> S <sub>4</sub> Cl <sub>2</sub> Na <sub>4</sub> Br <sub>2</sub>	1542	82	41.22 (41.29)	2.18 (2.22)	12.66 (12.72)	0.45
D <sub>2</sub>	J-acid	C <sub>53</sub> H <sub>36</sub> O <sub>8</sub> N <sub>14</sub> S <sub>2</sub> Cl <sub>2</sub> Na <sub>2</sub> Br <sub>2</sub>	1338	80	47.51 (47.58)	2.66 (2.71)	14.61 (14.66)	0.42
D <sub>3</sub>	N-Phenyl-J-acid	C <sub>65</sub> H <sub>44</sub> O <sub>8</sub> N <sub>14</sub> S <sub>2</sub> Cl <sub>2</sub> Na <sub>2</sub> Br <sub>2</sub>	1490	78	52.35 (52.40)	2.95 (2.98)	13.11 (13.16)	0.35
D <sub>4</sub>	N-Methyl-J-acid	C <sub>55</sub> H <sub>40</sub> O <sub>8</sub> N <sub>14</sub> S <sub>2</sub> Cl <sub>2</sub> Na <sub>2</sub> Br <sub>2</sub>	1366	84	48.30 (48.37)	2.88 (2.95)	14.28 (14.36)	0.38
D <sub>5</sub>	Bronner acid	C <sub>53</sub> H <sub>36</sub> O <sub>6</sub> N <sub>14</sub> S <sub>2</sub> Cl <sub>2</sub> Na <sub>2</sub> Br <sub>2</sub>	1306	80	48.72 (48.75)	2.72 (2.78)	14.98 (15.02)	0.37
D <sub>6</sub>	Gamma acid	C <sub>53</sub> H <sub>36</sub> O <sub>8</sub> N <sub>14</sub> S <sub>2</sub> Cl <sub>2</sub> Na <sub>2</sub> Br <sub>2</sub>	1338	78	47.50 (47.58)	2.64 (2.71)	14.58 (14.66)	0.40
D <sub>7</sub>	Chicago acid	C <sub>53</sub> H <sub>34</sub> O <sub>14</sub> N <sub>14</sub> S <sub>4</sub> Cl <sub>2</sub> Na <sub>4</sub> Br <sub>2</sub>	1542	75	41.23 (41.29)	2.15 (2.22)	12.67 (12.72)	0.42
D <sub>8</sub>	Tobias acid	C <sub>53</sub> H <sub>38</sub> N <sub>14</sub> Cl <sub>2</sub> Br <sub>2</sub>	1102	77	57.71 (57.78)	3.42 (3.48)	17.75 (17.80)	0.40
D <sub>9</sub>	Sulfo tobias acid	C <sub>53</sub> H <sub>36</sub> O <sub>6</sub> N <sub>14</sub> S <sub>2</sub> Cl <sub>2</sub> Na <sub>2</sub> Br <sub>2</sub>	1306	80	48.72 (48.77)	2.73 (2.78)	14.98 (15.02)	0.37
D <sub>10</sub>	Laurant acid	C <sub>53</sub> H <sub>36</sub> O <sub>6</sub> N <sub>14</sub> S <sub>2</sub> Cl <sub>2</sub> Na <sub>2</sub> Br <sub>2</sub>	1306	82	48.74 (48.77)	2.70 (2.78)	14.95 (15.02)	0.35
D <sub>11</sub>	K-acid	C <sub>53</sub> H <sub>34</sub> O <sub>14</sub> N <sub>14</sub> S <sub>4</sub> Cl <sub>2</sub> Na <sub>4</sub> Br <sub>2</sub>	1542	80	41.24 (41.29)	2.15 (2.22)	12.67 (12.72)	0.42
D <sub>12</sub>	Peri acid	C <sub>53</sub> H <sub>36</sub> O <sub>6</sub> N <sub>14</sub> S <sub>2</sub> Cl <sub>2</sub> Na <sub>2</sub> Br <sub>2</sub>	1306	78	48.70 (48.77)	2.71 (2.78)	14.94 (15.02)	0.38

<sup>a</sup>Determined by TLC using benzyl alcohol : DMF : water (30 : 20 : 20) solvent system on Silica gel G F<sub>254</sub> TLC plate

### Preparation of m-toluidino cyanurated H-acid [R]



## RESULTS AND DISCUSSION

A new series of bis azo reactive dyes has been prepared by tetrazotisation of 4, 4'-methylene-bis-o-bromo aniline followed by coupling with various m-toluidino cyanurated coupling components. All the dyes show purple, yellow, orange and red hues on different fibres like silk, wool and cotton.

### IR and $^1\text{H}$ NMR spectral study

IR spectra<sup>13-14</sup> of all the dyes, in general showed O-H and N-H stretching vibrations in the region 3350-3520  $\text{cm}^{-1}$ , N-H bending vibration at 1520-1530  $\text{cm}^{-1}$ , C=C stretching vibration at 1430-1660  $\text{cm}^{-1}$ , N = N stretching vibration at 1580-1620  $\text{cm}^{-1}$ , C-N stretching vibration at 1380-1560  $\text{cm}^{-1}$ , S = O stretching vibration at 1030-1180  $\text{cm}^{-1}$ , C-Br stretching vibration at 620-650  $\text{cm}^{-1}$  and C-Cl stretching vibration at 750-780  $\text{cm}^{-1}$  (Table 5).

The  $^1\text{H}$  NMR<sup>15</sup> spectra of representative dye showed all the desired signals (Table 6).

### Dyeing of fibres

All the dyes (**D**<sub>1</sub> to **D**<sub>12</sub>) were applied on cotton, wool and silk fabrics in 2% shade according to the usual procedure<sup>16</sup> having the dye bath material given in Table 2.

**Table 2: Dye-bath materials**

Materials	For silk	For wool	For cotton
Fabric	2.0 g	2.0 g	2.0 g
Amount of dye	40 mg	40 mg	40 mg
Glauber's salt (20% w/v)	1.0 mL	1.5 mL	1.0 mL
Soda ash (10% w/v)	-	-	1.0 mL
Acetic acid (10% w/v)	1.0 mL	-	-
Formic acid (10% w/v)	-	1.5 mL	-
pH	3	3	8
MLR	1:40	1:40	1:40
Dyeing time	40 min	60 min	90 min
Dyeing temp.	60-80 °C	60-80 °C	60-80 °C
Total volume	80 mL	80 mL	80 mL

### Colour and spectral properties of dyes

The absorption maxima of the dyes **D**<sub>1</sub> to **D**<sub>12</sub> recorded in water are shown in Table 3. All the dyes give pink to yellow shades with absorption maxima between 470-525 nm. The values of logarithm of the molar extinction coefficient (log  $\epsilon$ ) of the dyes were in the range of 4.10-4.32, which indicates their good absorption intensity.

**Table 3: Percentage exhaustion and fixation of reactive dyes (**D**<sub>1</sub> to **D**<sub>12</sub>) on silk, wool and cotton**

Dye No.	$\lambda_{\max}^b$	log $\epsilon$	% Exhaustion			% Fixation		
			S	W	C	S	W	C
<b>D</b> <sub>1</sub>	525	4.32	64.50	65.82	68.45	88.37	91.90	86.19
<b>D</b> <sub>2</sub>	480	4.28	67.55	76.00	70.75	92.52	82.60	84.80
<b>D</b> <sub>3</sub>	475	4.18	72.80	74.60	69.80	89.97	91.15	87.39
<b>D</b> <sub>4</sub>	485	4.30	77.50	65.67	75.70	83.22	87.55	91.80

Cont...



Dye No.	$\lambda_{\max}^b$	log $\epsilon$	% Exhaustion			% Fixation		
			S	W	C	S	W	C
D <sub>5</sub>	490	4.26	71.80	66.95	69.35	87.74	91.85	85.80
D <sub>6</sub>	495	4.15	66.60	79.62	66.45	87.84	86.03	83.52
D <sub>7</sub>	510	4.23	68.75	67.80	72.90	86.54	86.28	92.93
D <sub>8</sub>	482	4.22	67.65	67.55	68.15	88.69	91.78	88.43
D <sub>9</sub>	470	4.18	73.50	69.45	67.02	88.43	88.55	90.26
D <sub>10</sub>	472	4.10	68.90	66.07	68.22	87.23	88.53	90.88
D <sub>11</sub>	505	4.20	70.85	73.45	67.65	93.86	80.33	84.99
D <sub>12</sub>	486	4.28	68.15	71.88	66.52	92.44	82.78	89.44

<sup>b</sup>Determined in water at 28 °C at  $2.0 \times 10^{-3}$  M dye concentration S-Silk, W-Wool and C-Cotton

### Exhaustion and fixation study

The percentage exhaustion of 2% dyeing on silk fabric ranges from 64 to 78 %, for wool ranges from 65 to 76 % and for cotton ranges from 66 to 75 %. The percentage fixation of 2% dyeing on silk fabric ranges from 83 to 93%, for wool ranges from 82 to 91% and for cotton ranges from 83 to 92% (Table 3).

### Fastness properties

Data on fastness properties show that the light fastness ranges from moderate to very good on silk, wool and cotton. The fastness to washing and rubbing ranges from good to excellent on silk, wool and cotton. (Table 4)

**Table 4: Fastness properties of reactive dyes (D<sub>1</sub> to D<sub>12</sub>) on silk, wool and cotton**

Dye No.	Light fastness			Wash fastness			Rubbing fastness					
	S	W	C	S	W	C	Dry			Wet		
							S	W	C	S	W	C
D <sub>1</sub>	6	6	6	5	5	5	5	5	4	4	5	4-5
D <sub>2</sub>	5-6	5	5	4-5	4-5	4-5	4	4-5	3	4-5	4-5	4

Cont...

Dye No.	Light fastness			Wash fastness			Rubbing fastness					
	S	W	C	S	W	C	Dry			Wet		
							S	W	C	S	W	C
D <sub>3</sub>	3	3	3-4	3-4	3	3	3	4	4-5	3	3	3
D <sub>4</sub>	5-6	5	6	5	5	5	4-5	4-5	4	4	4	4-5
D <sub>5</sub>	5	4-5	5	3	4	4	4	4	3-4	3-4	4	4
D <sub>6</sub>	3-4	4	3	4	3-4	2-3	4	4	3	3	3	3-4
D <sub>7</sub>	5	3-4	6	3-4	4	3-4	3	3	3-4	3	3-4	5
D <sub>8</sub>	6	5	3-4	5	3-4	5	4-5	4-5	4	4-5	4	3-4
D <sub>9</sub>	6	6	4	5	5	3	4	4	3	4	3-4	3
D <sub>10</sub>	3	3	3-4	3	4	3-4	3-4	4	5	3	3	4
D <sub>11</sub>	5-6	5-6	6	5	3	5	5	5	3-4	4-5	4	4
D <sub>12</sub>	3	3	4-5	4	4-5	3	3	4	3-4	3	3	5

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

**Wash and Rubbing fastness:** 1-Poor, 2-Fair, 3-Good, 4-Very good, 5-Excellent. S-Silk, W-Wool and C-Cotton

**Table 5: IR spectral data of reactive dyes (D<sub>1</sub> to D<sub>12</sub>)**

Dye No.	IR spectra (cm <sup>-1</sup> )
D <sub>1</sub>	3350-3520 (O-H & N-H stretching), 1523 (N-H bending), 1580 (N=N stretching), 1540, 1414, 830 (C-N stretching), 1206, 1043 (S=O, asym & sym stretching), 750 (C-Cl stretching), 655 (C-Br stretching)
D <sub>2</sub>	3345-3530 (O-H & N-H stretching), 1530 (N-H bending), 1585 (N=N stretching), 1542, 1418, 835 (C-N stretching), 1210, 1042 (S=O, asym & sym stretching), 755 (C-Cl stretching), 650 (C-Br stretching)
D <sub>3</sub>	3315-3500 (O-H & N-H stretching), 1535 (N-H bending), 1605 (N=N stretching), 1545, 1420, 822(C-N stretching), 1215, 1040 (S=O, asym & sym stretching), 752 (C-Cl stretching), 652 (C-Br stretching)

Cont...

Dye No.	IR spectra (cm <sup>-1</sup> )
D <sub>4</sub>	3365-3510 (O-H & N-H stretching), 1542 (N-H bending), 1605 (N=N stretching), 1552, 1415, 812 (C-N stretching), 1225, 1056 (S=O, asym & sym stretching), 745 (C-Cl stretching), 650 (C-Br stretching)
D <sub>5</sub>	3350-3540 (O-H & N-H stretching), 1520 (N-H bending), 1590 (N=N stretching), 1535, 1420, 835 (C-N stretching), 1220, 1050 (S=O, asym & sym stretching), 762 (C-Cl stretching), 642 (C-Br stretching)
D <sub>6</sub>	3350-3545 (O-H & N-H stretching), 1532 (N-H bending), 1582 (N=N stretching), 1545, 1412, 832 (C-N stretching), 1198, 1040 (S=O, asym & sym stretching), 752 (C-Cl stretching), 645 (C-Br stretching)
D <sub>7</sub>	3345-3562 (O-H & N-H stretching), 1540 (N-H bending), 1598 (N=N stretching), 1555, 1420, 810 (C-N stretching), 1186, 1053 (S=O, asym & sym stretching), 748(C-Cl stretching), 642 (C-Br stretching)
D <sub>8</sub>	3340-3562 (O-H & N-H stretching), 1538 (N-H bending), 1602 (N=N stretching), 1548, 1425, 812 (C-N stretching), 1222, 1050 (S=O, asym & sym stretching), 755 (C-Cl stretching), 652 (C-Br stretching)
D <sub>9</sub>	3410-3565 (O-H & N-H stretching), 1543 (N-H bending), 1610 (N=N stretching), 1555, 1435, 812 (C-N stretching), 1225, 1052 (S=O, asym & sym stretching), 765 (C-Cl stretching), 642 (C-Br stretching)
D <sub>10</sub>	3395-3545 (O-H & N-H stretching), 1548 (N-H bending), 1612 (N=N stretching), 1560, 1430, 822 (C-N stretching), 1226, 1048 (S=O, asym & sym stretching), 760 (C-Cl stretching), 650 (C-Br stretching)
D <sub>11</sub>	3365-3552 (O-H & N-H stretching), 1522 (N-H bending), 1602 (N=N stretching), 1565, 1428, 822 (C-N stretching), 1218, 1042 (S=O, asym & sym stretching), 765 (C-Cl stretching), 645 (C-Br stretching)
D <sub>12</sub>	3352-3560 (O-H & N-H stretching), 1520 (N-H bending), 1605 (N=N stretching), 1542, 1418, 822 (C-N stretching), 1206, 1032 (S=O, asym & sym stretching), 758 (C-Cl stretching), 652 (C-Br stretching)

### Microbial studies

All the bromine containing reactive dyes are inactive against both Gram positive bacteria (*Pseudomonas Sp.* & *B. Subtilis*) and Gram negative bacteria (*Ceretium* & *E. Coli*) at 100 µg/mL and 200 µg/mL concentration, respectively as compared to amphotericine-B.

**Table 6:  $^1\text{H}$  NMR spectral data of reactive dyes ( $\text{D}_1$  to  $\text{D}_{12}$ )**

<b>Dye No.</b>	<b><math>^1\text{H}</math> NMR Spectra (DMSO-<math>d_6</math>) (chemical shift in <math>\delta</math> ppm)</b>
<b>D<sub>1</sub></b>	$-\text{CH}_3$ (6H, s, 0.95), $-\text{CH}_2-$ (2H, s, 2.10), $-\text{OH}$ (2H, s, 3.38), $>\text{NH}$ (4H, s, 4.22), Ar-H (20H, m, 6.82-8.10)
<b>D<sub>2</sub></b>	$-\text{CH}_3$ (6H, s, 0.92), $-\text{CH}_2-$ (2H, s, 2.15), $-\text{OH}$ (2H, s, 3.42), $>\text{NH}$ (4H, s, 4.35), Ar-H (22H, m, 6.78-8.12)
<b>D<sub>3</sub></b>	$-\text{CH}_3$ (6H, s, 0.98), $-\text{CH}_2-$ (2H, s, 2.14), $-\text{OH}$ (2H, s, 3.35), $>\text{NH}$ (2H, s, 4.08), Ar-H (32H, m, 6.85-8.22)
<b>D<sub>4</sub></b>	$-\text{CH}_3$ (12H, s, 0.88), $-\text{CH}_2-$ (2H, s, 2.22), $-\text{OH}$ (2H, s, 3.32), $>\text{NH}$ (2H, s, 4.15), Ar-H (22H, m, 6.72-8.15)
<b>D<sub>5</sub></b>	$-\text{CH}_3$ (6H, s, 0.85), $-\text{CH}_2-$ (2H, s, 2.05), $>\text{NH}$ (4H, s, 4.30), Ar-H (24H, m, 6.72-8.22)
<b>D<sub>6</sub></b>	$-\text{CH}_3$ (6H, s, 0.82), $-\text{CH}_2-$ (2H, s, 2.26), $-\text{OH}$ (2H, s, 3.22), $>\text{NH}$ (4H, s, 4.35), Ar-H (22H, m, 6.78-8.22)
<b>D<sub>7</sub></b>	$-\text{CH}_3$ (6H, s, 0.95), $-\text{CH}_2-$ (2H, s, 2.18), $-\text{OH}$ (2H, s, 3.25), $>\text{NH}$ (4H, s, 4.32), Ar-H (20H, m, 6.72-8.15)
<b>D<sub>8</sub></b>	$-\text{CH}_3$ (6H, s, 0.92), $-\text{CH}_2-$ (2H, s, 2.15), $>\text{NH}$ (4H, s, 4.35), Ar-H (26H, m, 6.75-8.22)
<b>D<sub>9</sub></b>	$-\text{CH}_3$ (6H, s, 0.98), $-\text{CH}_2-$ (2H, s, 2.25), $>\text{NH}$ (4H, s, 4.32), Ar-H (24H, m, 6.82-8.30)
<b>D<sub>10</sub></b>	$-\text{CH}_3$ (6H, s, 0.88), $-\text{CH}_2-$ (2H, s, 2.28), $>\text{NH}$ (4H, s, 4.28), Ar-H (24H, m, 6.78-8.28)
<b>D<sub>11</sub></b>	$-\text{CH}_3$ (6H, s, 0.85), $-\text{CH}_2-$ (2H, s, 2.33), $-\text{OH}$ (2H, s, 3.42), $>\text{NH}$ (4H, s, 4.35), Ar-H (20H, m, 6.85-8.32)
<b>D<sub>12</sub></b>	$-\text{CH}_3$ (6H, s, 0.95), $-\text{CH}_2-$ (2H, s, 2.12), $>\text{NH}$ (4H, s, 4.35), Ar-H (24H, m, 6.75-8.30)

Abbreviations in  $^1\text{H}$  NMR data: s, singlet; d, doublet; t, triplet; m, multiplet

## CONCLUSION

A series of reactive dyes have been synthesized by conventional method and their colour properties have been examined both; in solution and on application to silk, wool and cotton fibres. The dyes give yellow to pink shades on silk, wool and cotton depending on the coupling component used, having moderate to very good light fastness on silk, wool and cotton. The washing and rubbing fastness properties ranges from good to excellent on silk, wool and cotton. This indicates good substantivity of these dyes for silk, wool and cotton. A remarkable degree of levelness after washing indicates good penetration and affinity of these dyes to the fabrics.

## ACKNOWLEDGEMENT

The authors express their gratitude to Head of the Department of Chemistry, Veer Narmad South Gujarat University, Surat for providing necessary research facilities, SAIF, Chandigarh for  $^1\text{H}$  NMR spectra and Atul Ltd., Atul (Valsad) for providing dyeing and analytical facilities.

## REFERENCES

1. K. M. Shah, Handbook of Synthetic Dyes and Pigments, **Vol. I**, Chapter-14, (1998) p. 229.
2. K. Bredereck and C. Schumacher, Dyes and Pigments, **21**, 23 (1993)
3. M. Masaki, M. Ulrich and H. Zolinger, J. Soc. Dyers Colour., **104**, 425 (1998).
4. J. R. Mehta, S. K. Patel and K. C. Patel, Colourage, **10**, 85 (2008).
5. N. B. Patel, S. K. Patel and K. C. Patel, Chemistry-An Indian Journal, **3(1)**, 35 (2006).
6. A. L. Patel, N. B. Patel, B. M. Patel and K. C. Patel, Asian J. Chem., **19(1)**, 67 (2007).
7. N. R. Ayyangar, G. S. Jadhav, S. V. Joshi and K. V. Srinivasan, Dyes and Pigments, **8(4)**, 301 (1987).
8. N. R. Ayyangar, R. J. Lahoti, K. V. Srinivasan, T. Daniel and H. K. Venkataramaih, Dyes and Pigments, **17(4)**, 279 (1991).
9. B. Fried and J. Sherma, Thin Layer Chromatography: Techniques and Applications, Marcell-Dekker, Inc., New York and Basel (1982).
10. Standard Test Method BS 1006, (1978) (UK): ISO India, 105 (1994).
11. AATCC Test Method, (1961) p. 8.

12. Indian Standard ISO: 765 (1979).
13. N. B. Colthup, L. H. Daly and S. E. Wiberley, Introduction to Infrared and Raman Spectroscopy, Academic Press, New York (1991).
14. G. C. Bassler, R. M. Silverstein and J. C. Morrill, Spectrophotometric Identification of Organic Compounds, 5<sup>th</sup> Ed., Wiley. New York (1991).
15. J. A. Dean, Clin. Chem., **14**, 324 (1968)
16. V. A. Shenai, Chemistry of Dyes and Principles of Dyeing, Sevak Publication. Bombay (1973).

*Accepted : 03.11.2009*