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Synthesis, Characterization and Dyeing Performance of Novel Bisazo-Bisazomethine Disperse Dyes on Polyester Fabric

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ABSTRACT

Azo dyes and pigment forms the largest group of all the synthetic colorants and play a prominent role in almost every type of applications. In the last couple of decades a number of major changes have occurred primarily aimed at minimizing the major weakness of anthraquinone dyes. For this purpose attempts were made to replace anthraquinone dyes by azo dyes. For this purpose we have undertaken the work in this direction with a view to synthesize novel bisazo-bisazomethine disperse dyes. Therefore, it is worthwhile to describe the chemistry and development of azo and bisazo dyes in brief for the sake of better understanding. Bisazo and bisazomethine dyes are of current important, however there are very few reports regarding bisazo-bisazomethine disperse dye. In the textile industry, different fibers used for dyeing are all natural fiber like cellulose (linen, cotton) or animal protein fibers (wool, silk, animal hair) and synthetic fibers like nylon, polyester, polyacrylonitrile and blends of two or more fibers. The development of a new substrate presents new dyes and dyeing methods and it influence dyeing technology in a fundamental manner. New bisazo-bisazomethine disperse dyes were prepared by the coupling of diazotized solutions of various aromatic primary amines with 2, 2'-{1,4-phenylenebis [nitrilomethylylidene]} diphenol (Schiff base). Schiff base was prepared by the condensation of 2-hydroxybenzaldehyde with 4-aminoaniline. The resultant dyes were characterized by elemental analysis, IR and 1H NMR Spectral Studies. The UV-visible absorption spectral data were investigated in dimethylformamide (DMF) and are discussed in terms of structure property relationship. The dyes when applied on polyester fabric, gave pale yellow to orange shades having fairly good to good light fastness, very good to excellent washing, perspiration and sublimation fastness and good to very good rubbing fastness properties.

KEYWORD: *Bisazo-bisazomethine; Schiff base; Polyester; Disperse dyes*

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1. INTRODUCTION

Azo dyes are marketed in the form of bisazo and polyazo disperse dyes as dyeing materials [1-4]. 8-quinoline sulfonamide based bisazo disperse dyes have been prepared and their dyeing performance on polyester fabric was assessed [5]. Also quinoline based various bisazo disperse dyes synthesized [6-8]. Thus the field of bisazo disperse dyes are current interest. Recently there are also reports regarding bisazomethine dyes in which imine linkage formed by the Schiff reaction of aromatic aldehyde with aromatic amine [9-10].

Literature survey shows that both bisazo and bisazomethine dyes are of current importance, however there are hardly few reports regarding bisazo-bisazomethine disperse dyes in which both bisazo and bisazomethine chromophoric groups lying in a single molecular framework [11]. Hence, the present work comprises synthesis of a series of novel bisazo-bisazomethine disperse dyes. In addition to characterization of the above dyes, they have also been accessed successfully as disperse dyes on polyester fabrics.

2. Experimental

2.1. Materials and methods

All the chemicals used were of analytical grade and were further purified as and when required. 4-aminoanilin0, 4-hydroxybenzaldehyde, different aromatic primary amines, sodium hydroxide, sodium nitrite, hydrochloric acid were purchased from local market. The organic solvents used were purified by standard methods [12]. The aromatic amines used for diazotization are listed in Scheme 1. Melting points were determined by open capillary method and were found uncorrected. The visible absorption spectra of all dyes obtained for solution (1×10-4 gm/ml) prepared in DMF and were recorded on a Carl Zeiss UV/VIS Specord spectrometer, and elemental analysis was carried out on Perkin Elmer CHNS/O Analyzer 2400 Series II. Infrared spectra were recorded on a PerkinElmer Spectrum GX FT-IR model between 4000 and 400 cm-1 using a KBr pellets and 1H NMR spectra were recorded on Bruker DRX-400 FT-NMR spectrometer at 400 MHz using in DMSO-d6 solvent (Chemical shift in ä ppm). The purity of all the dyes was checked by TLC using chloroform:methanol (4:1) solvent system [13]. The C, H, N contents of Schiff base and bisazo-bisazomethine disperse dye were carried out on perkin Elmer CHNS analyzer 2400 series II. The number of diazo group in each disperse dye was determined by the reported process [14], indicating that there are two diazo group (-N=N-) present in the disperse dye molecule. Fastness to light of dyed patterns were assessed in accordance with method developed by Park and D. J. Smith [15-17], sublimation and perspiration fastness test were carried out in accordance with BS:1006-1978, washing fastness test in accordance with IS:765-1979 [18] and fastness to rubbing was tested using Crockmeter (Atlas) in accordance with ATCC1961[19]. The dyeing of polyester fabric was carried out according to method described in the literature[20]. The percentage dye bath exhaustion (%E) and percentage dye bath fixation (%F) of the dyed fabric was determined according to the known method [21]

2.2. Synthesis of 2,2'-{1,4-phenylenebis [nitrilomethylylidene]} diphenol (SB)

The title bisanil compound was synthesized by the reported method [22]. A solution of 4-aminoaniline (2.16 g, 0.02 mol) in absolute alcohol (50 ml) was prepared. To the above solution an ethanolic solution (100 ml) of salicylaldehyde (4.88 g, 0.04 mol) was added slowly under stirring to produce a orange crystalline solid product upon cooling at room temperature. The product thus obtained was filtered, washed with ethanol and dried in a vacuum desiccator. Yield: 91% m.p. 200-204 ^{0}C (uncorrected). The synthetic route is shown in Scheme - 1.

Elemental analysis: Found: C 75.68%; H 4.97%; N 8.67%; $C_{20}H_{16}N_2O_2$ (Required C 75.94%; H 5.06%; N 8.89%). IR (KBr, cm^{-1}): 3020 (C-H stretching, aromatic); 1605, 1514 (C-C multiple bonding aromatic); 3615, 1172, 1309 (phenol O-H stretching, O-H bending, C-O stretching); 1630 (>C=N- stretching). ^{1}H-NMR (400 MHz, DMSO-d_6, δ ppm) 6.72-7.86 (12H, m, Ar–H), 8.49 (2H, s, N═CH), 12.62 (2H, s, Ar–OH).

2.3. Synthesis of the bisazo-bisazomethine disperse dyes D_n

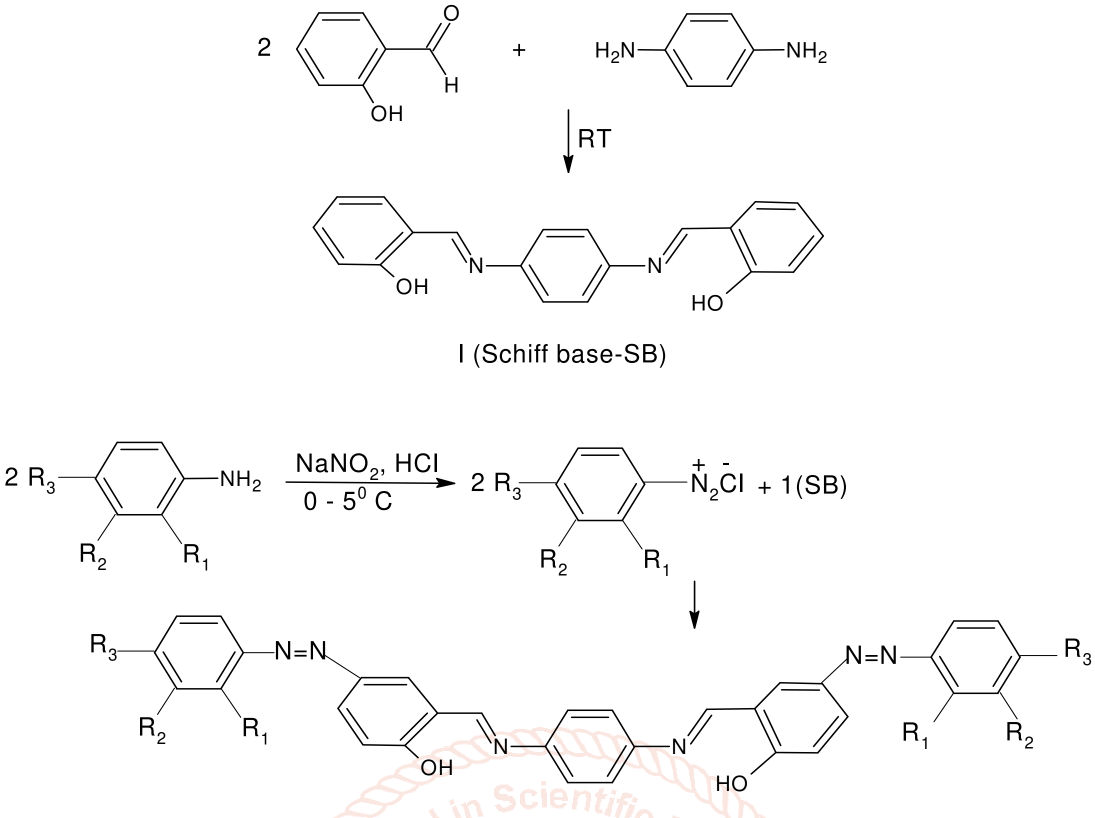
2.3.1. Diazotization:

Diazotization of various aromatic amines (listed in Scheme 1) was carried out by the reported method [23]. The clear diazonium salt solution thus obtained was used immediately in the coupling reaction.

2.3.2. General coupling procedure

The coupling of above mentioned diazotized aromatic amines were carried out by the reported method [23]. Accordingly, the general procedure adopted for coupling reaction is given below:

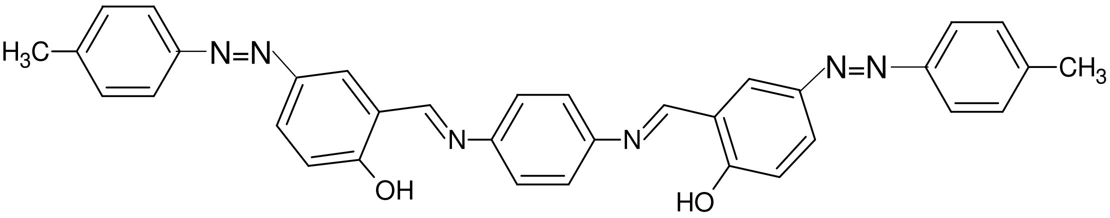
2, 2'-{1,4-phenylenebis [nitrilomethylylidene]} diphenol (SB) (3.16 gm, 0.01 mol) was dissolved in 25 ml sodium hydroxide (1.0 M) solution. The obtained clear solution was cooled in an ice-bath and the diazonium solution of 4-methyl aniline was added drop wise over a period of 30 min with vigorous stirring. The pH of the mixture was maintained between 8.0 and 9.0 by simultaneous addition of the 10% (w/v) sodium carbonate solution. Stirring was continued for further ~2h by maintaining the temperature at 0-5 0C. Then the pH of reaction mixture was maintained at 7.0 by addition of dilute acetic acid (2 M) solution. Thus the dye D1 was precipitated out was then filtered off, washed with distilled water until it was free from acid and salt and, then it was dried at 50 0C in an oven. The obtained dyes were recrystallized by ethanol. Following the above procedure, other bisazo-bisazomethine disperse dyes D_2-D_{10} were synthesized using diazotized various aromatic amines and bisanil compound as a coupler. The synthetic route is shown in **Scheme 1.** The purity of all the dyes were checked by TLC using chloroform: methanol (4:1) solvent system. The melting points of the purified dyes were measured and are uncorrected. The visible absorption spectroscopic properties of the dyes were recorded in DMF solution. The dyes prepared are given in chart 1 and the characteristic data of all the dyes are recorded in Table 1.



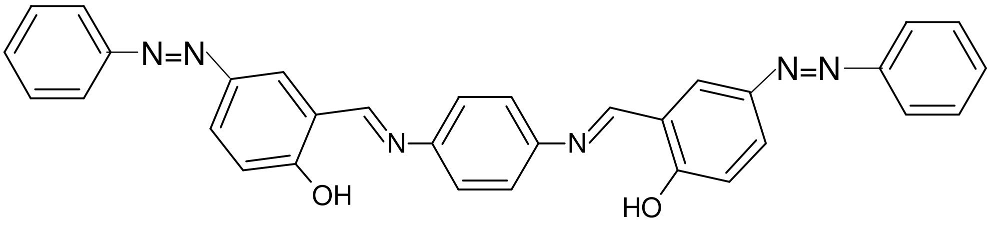
Scheme 1: Synthetic route for the preparation of bisazo-bisazomethine disperse dyes

Where substituent R1, R2 and R3 of aromatic amines are given below:

Dye No.	R_1	R_2	R_3	Amines
D_1	H	H	CH_3	4-methylaniline
D_2	H	H	H	Aminobenzene
D_3	H	H	NO_2	4-nitroaniline
D_4	H	NO_2	H	3-nitroaniline
D_5	H	Cl	H	3-chloroaniline
D_6	NO_2	H	H	2-nitroaniline
D_7	H	H	Cl	4-chloroaniline
D_8	H	H	OH	4-hydroxyaniline
D_9	H	OH	H	3-hydroxyaniline
D_{10}	H	H	Br	4-bromoaniline



Dye D_1



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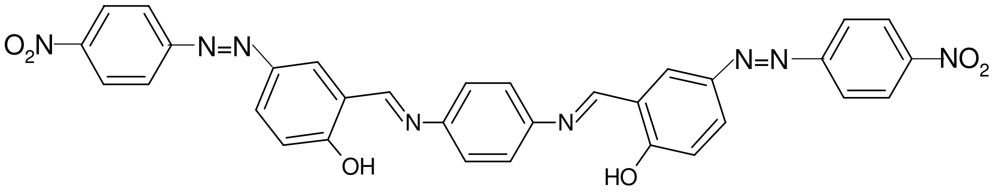
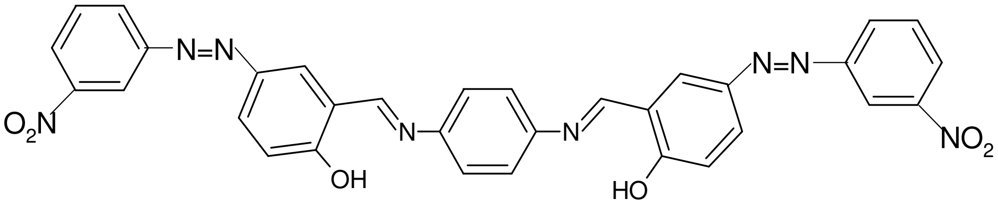


Dye D_3



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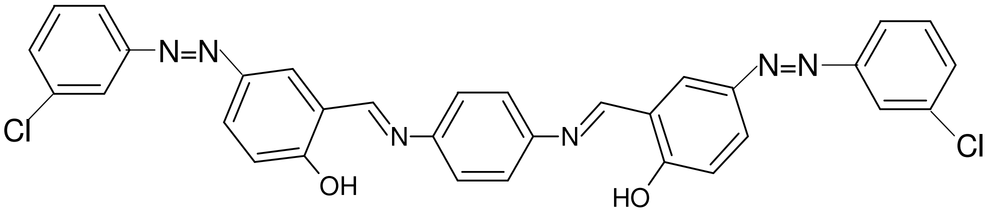
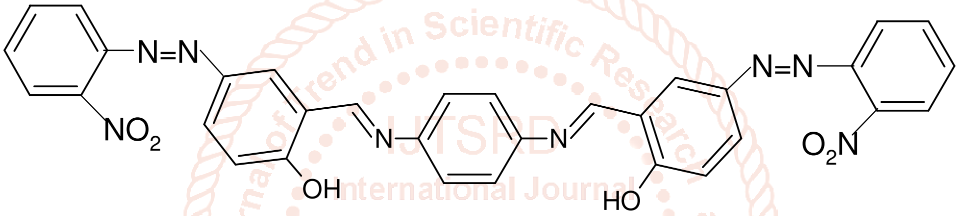


Dye D_5



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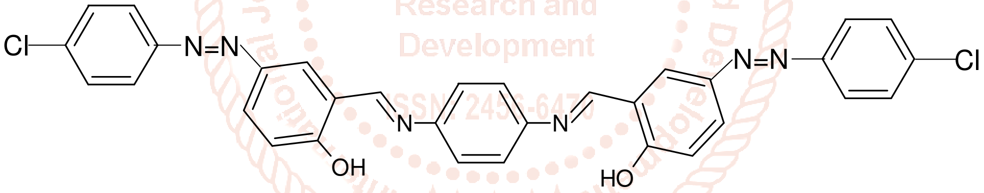
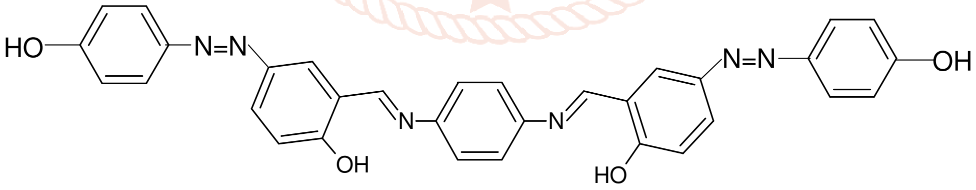


Dye D_7



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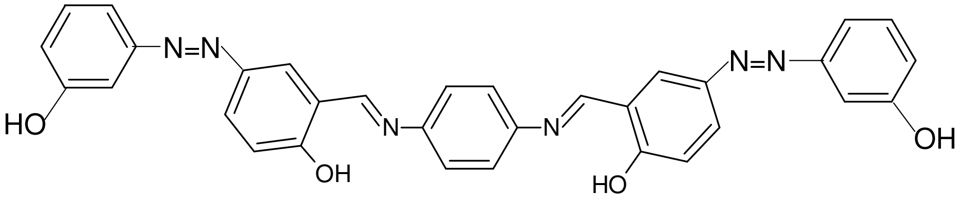
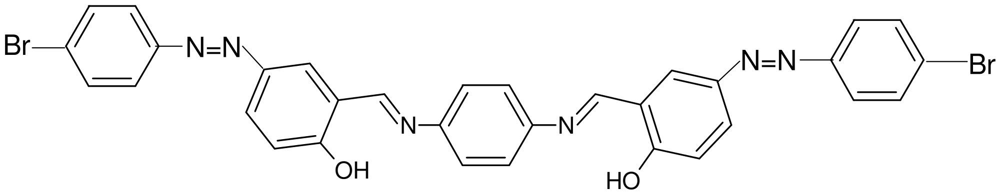


Dye D_9



Dye D_{10}

Chart 1 The structure of dyes ($D_1 – D_{10}$)

Table 1 Elemental analysis of disperse dyes

Dye no.	Molecular formula	Mol. wt. g/mol	%C		%H		%N		%X (Cl, Br)	
			M	F	M	F	M	F	M	F
D_1	$C_{41}H_{34}O_2N_6$	642	76.61	76.41	5.29	5.15	13.08	13.02	-	-
D_2	$C_{39}H_{30}O_2N_6$	614	76.22	76.05	4.88	4.65	13.68	13.45	-	-
D_3	$C_{39}H_{28}O_6N_8$	704	66.47	66.25	3.97	3.85	15.90	15.75	-	-
D_4	$C_{39}H_{28}O_6N_8$	704	66.47	66.30	3.97	3.80	15.90	15.70	-	-
D_5	$C_{39}H_{28}O_2N_6Cl_2$	683	68.52	68.35	4.09	4.00	12.29	12.05	10.39	10.32
D_6	$C_{39}H_{28}O_6N_8$	704	66.47	66.35	3.97	3.82	15.90	15.75	-	-
D_7	$C_{39}H_{28}O_2N_6Cl_2$	683	68.52	68.34	4.09	3.95	12.29	12.15	10.39	10.25
D_8	$C_{39}H_{30}O_4N_6$	646	72.44	72.24	4.64	4.52	13.00	12.85	-	-
D_9	$C_{39}H_{30}O_4N_6$	646	72.44	72.30	4.64	4.57	13.00	12.75	-	-
D_{10}	$C_{39}H_{28}O_2N_6Br_2$	772	60.62	60.45	3.62	3.51	10.88	10.65	20.72	20.55

M= maximum, F= found.

3. Results and discussion:

3.1. Preparation of dye D_1-D_{10}

The key intermediate, bisanil compound I was satisfactorily prepared. Various substituted aromatic amines were diazotized effectively at 0-5^0 C by sodium nitrite and hydrochloric acid. In order to determine the end point of diazotization, it was found useful to check for the presence of unreacted diazo component no longer persisted on TLC, the diazotization was ended. The diazonium salt solution was used immediately, since this decomposed on standing, even when cold. Subsequent coupling reactions took place readily on adding the resulting diazonium salt continuously to the solution of coupling component in sodium carbonate. Coupling was usually accompanied by some evidence of decomposition; however 60-94 % yield (except D_1) of the dye were usually obtained by careful addition of the diazonium salt solution at 0-5^0 C to a solution of coupling component.

3.2. Physical properties of dyes

All the dyes were obtained as amorphous powder ranging in color from yellow to reddish brown. The purity of the dyes was checked by TLC using chloroform: methanol (4:1) solvent system. When adsorbed onto silica chromatography plates, the dyes produced a single spot. All the dyes are soluble in acetone, DMF, acetic acid and insoluble in water, ether, n-hexane etc. Elemental analysis data are given in Table **1.**

3.3. IR and 1H NMR spectra

The infrared spectra of dyes D7 is shown in Figure 1. The data of IR spectrum features of all disperse dyes are shown in Table 3.2. Examination of the IR spectra of all the bisazo-bisazomethine disperse dyes reveals that all the spectrum shown some identical bands due to the presence of aromatic nuclei, azomethine and azo group in most of the disperse dye molecules. The infrared spectra of dye D_7 are shown in Figure 1. The data of IR spectrum features of all disperse dyes are shown in Table 3.2. Examination of the IR spectra of all the bisazo-bisazomethine disperse dyes reveals that all the spectrum shown some identical bands

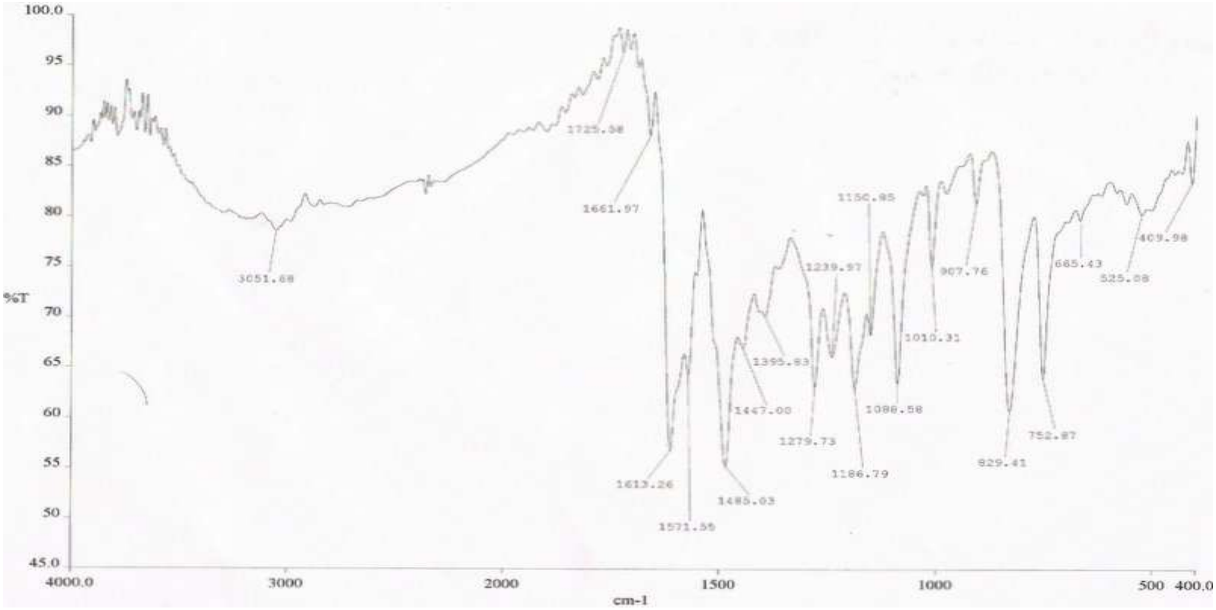


Fig. 1 IR spectrum of D7

Most of the spectrum comprise: A broad band at 3400-3600 cm^{-1} mainly arise due to free or hydrogen bonded –OH (phenolic) group and -1575 cm^{-1} are considered due to presence of azomethine and azo groups. The bands appeared at ~1600 and ~1500 cm^{-1} is due to aromatic stretching of the double bond. The variable bands at 1690-1640 cm^{-} and 1630 are considered due to presence of azomethine and azo groups respectively. In addition to this the IR spectrum of the disperse dyes have shown the characteristic absorption bands due to presence of $-NO_2$, -CI, -Br groups at 1570-1500 cm^{-1} and 1370-1300 cm^{-1}, 800-600 cm^{-1}, 600-500 cm^{-1} respectively. The substituents present at the para to the azo group in a diazo component of disperse dyes have shown characteristics absorption band at 830 cm^{-1}. The 1H NMR features of all the disperse dyes are shown in Table **2.**

Table 2 IR and ^{1}H NMR data of disperse dyes (D_1-D_{10}).

Dye no.	IR (KBr): *v* (cm^{-1})	^{1}H NMR (DMSO-d_6) (chemical shift in δ ppm)
D_1	2910($-CH_3$), 2950, 3018 (C–H), 1653 (>C=N–), 1599 (–N=N–), 1618, 1473 (C–C), 3416, 1348 (O–H & C-N).	2.28 (6H, s, Ar–CH_3), 3.74 (2H, s, Ar–CH_2–Ar), 6.88-8.16 (22H, m, Ar–H), 8.46 (2H, s, N=CH), 12.46 (2H, s, Ar–OH).
D_2	3015 (C–H), 1651 (>C=N–), 1605 (–N=N–), 1572, 1490 (C–C), 3595, 1340 (O–H & C-N).	3.76 (2H, s, Ar–CH_2–Ar), 7.26-8.16 (24H, m, Ar–H), 8.45 (2H, s, N=CH), 12.44 (2H, s, Ar–OH).
D_3	3020 (C–H), 1645 (>C=N–), 1610 (–N=N–), 1615, 1495 (C–C), 3610, 1340 (O–H & C-N), 1520, 1330 (C-NO_2).	3.75 (2H, s, Ar–CH_2–Ar), 7.21-8.17 (22H, m, Ar–H), 8.47 (2H, s, N=CH), 12.47 (2H, s, Ar–OH).
D_4	3028 (C–H), 1654 (>C=N–), 1619 (–N=N–), 1572, 1489 (C–C), 3650,1283 (O–H & C-N), 1525, 1351 (C-NO_2).	3.74 (2H, s, Ar–CH_2–Ar), 6.90-8.45 (22H, m, Ar–H), 8.49 (2H, s, N=CH), 12.43 (2H, s, Ar–OH).
D_5	3060 (C–H), 1633 (>C=N–), 1581 (–N=N–), 1605, 1480 (C–C), 3680, 1344 (O–H & C-N), 745 (C-Cl).	3.76 (2H, s, Ar–CH_2–Ar), 7.04-8.27 (22H, m, Ar–H), 8.44 (2H, s, N=CH), 12.47 (2H, s, Ar–OH).
D_6	3025 (C–H), 1651 (>C=N–), 1615 (–N=N–), 1580, 1505 (C–C), 3650, 1330 (O–H & C-N), 1523,1310 (C-NO_2).	3.75 (2H, s, Ar–CH_2–Ar), 6.94-8.23 (22H, m, Ar–H), 8.45 (2H, s, N=CH), 12.49 (2H, s, Ar–OH).
D_7	3025 (C–H), 1669 (>C=N–), 1618 (–N=N–), 1598, 1502 (C–C), 3670, 1310 (O–H & C-N), 751 (C-Cl).	3.74 (2H, s, Ar–CH_2–Ar), 7.20-8.15 (22H, m, Ar–H), 8.46 (2H, s, N=CH), 12.48 (2H, s, Ar–OH).
D_8	3025 (C–H), 1665 (>C=N–), 1615 (–N=N–), 1595, 1490 (C–C), 3660, 1325 (O–H & C-N).	3.73 (2H, s, Ar–CH_2–Ar), 6.87-8.17 (22H, m, Ar–H), 8.45 (2H, s, N=CH), 12.44 (2H, s, Ar–OH terminal), 10.84 (2H, s, Ar–OH).
D_9	3033 (C–H), 1667 (>C=N–), 1620 (–N=N–), 1595, 1505 (C–C), 3670, 1314 (O–H & C-N).	3.73 (2H, s, Ar–CH_2–Ar), 6.87-8.13 (22H, m, Ar–H), 8.46 (2H, s, N=CH), 9.91 (2H, s, Ar–OH terminal), 12.48 (2H, s, Ar–OH).
D_{10}	3036 (C–H), 1665 (>C=N–), 1625 (–N=N–), 1595, 1480 (C–C), 3645, 1305 (O–H & C-N), 543 (C-Br).	3.74 (2H, s, Ar–CH_2–Ar), 7.16-8.16 (22H, m, Ar–H), 8.44 (2H, s, N=CH), 12.42 (2H, s, Ar–OH).

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

3.4. UV–visible spectra

The absorption maxima (ëmax) of disperse dyes (D_1-D_{10}) fall in the range of 359-419 nm as shown in Table **3.** As far as absorption maxima are concerned ëmax are directly proportional to the electronic power of the substituents in the coupled ring system since the coupled component is identical in all the disperse dyes, the presence of electron donating or electron attracting groups did not bring about any bathochromic or hypsochromic shift. However considerable bathochromic shift observed in the disperse dyes D_1, D_5, D_7 and D_{10} because of *o*- and *p*- directing groups present on the aromatic ring, also deepen the color. The value of the logarithm of the molar extinction coefficient (log a_0) of all the dyes were in the range of 4.157 - 4.347 which consistent with their high absorption intensity. One cause of the increased intensity might be attributed to the greater planarity of the dyes because of the lower steric interaction of a phenyl ring compared to naphthyl ring.

3.5. Dyeing properties

All the dispersed dyes were applied at 2% depth on polyester fabrics. The percentage of exhaustion and fixation of the dyes D_1-D_{10} for polyester fabric ranges from 65% to 78% and 59% to 78% respectively. The variation in the shade of the dye fabric results from both the nature and position of the substituent present on the diazotized amine. The dyed fabric have fairly good to good light fastness; very good to excellent washing, perspiration and sublimation fastness and good to very good rubbing fastness for polyester fabric respectively. These dyes gave a narrow range of color ranging from yellow properties. All the fastness properties shown in Table **4**. are interrelated since they depend among other things, on the rate of diffusion of dye in the fabric. This rate is a function of the geometry of the dye molecule. The concentration of dye in the fabric appeared to be the most influential factor in the fastness of the dyeing a remarkable degree of levelness after washing was observed. This may be attributed to the good penetration and affinity of the dye for the fabric structure.

Table 3 Absorption maxima (λ_{max}), intensities (log ε), exhaustion (E) and fixation (F) of bisazo-bisazomethine disperse dyes on polyester fabric.

Dye Samples	λ_{max} nm	log ε	Disperse dyeing on polyester	
			%E	%F
D_1	362	4.347	70.12	69.73
D_2	359	4.225	65.37	71.09
D_3	400	4.346	76.87	72.68
D_4	390	4.320	72.62	64.19
D_5	403	4.311	73.12	58.97
D_6	384	4.310	71.87	65.21
D_7	391	4.309	70.87	74.95
D_8	419	4.250	78.12	64.48
D_9	395	4.225	72.60	77.99
D_{10}	401	4.157	76.10	62.91

Table 4 Evaluation of fastness properties of disperse dyeing on polyester:

Dye No.	Colour shades on polyester	Light fastness	Washing fastness	Perspiration fastness		Rubbing fastness		Sublimation fastness
				Acid	Alkaline	Dry	Wet	
D_1	Yellow brown	5	4	4	5	4	4	5
D_2	Reddish brown	5	5	5	5	4	4	5
D_3	Dark yellow	5	5	4	5	5	4.5	5
D_4	Light yellow	5	4	5	4	5	4	5
D_5	Dark yellow	4	4	4	5	4	4	5
D_6	Dark yellow	5	5	4	5	5	4	5
D_7	Light yellow	5	4	5	5	4	4.5	5
D_8	Off white	5	5	5	5	5	4.5	4
D_9	Light yellow	5	5	4	4	4	5	5
D_{10}	Orange	5	5	5	4	5	4	5

4. Conclusion

The symmetrical bisazo-bisazomethine disperse dyes have been synthesized and characterized. These dyes give mostly yellow, orange and brown shades on polyester fabrics, and yellow and reddish brown shades on nylon fabrics having overall good fastness properties. The nature of the substituent in the coupling component has little influence on the visible absorption and the shade of the dyed fabric. The exhaustion and fixation of these dyes are very good; this indicates that the dyes have good affinity and solubility with the polyester fabrics. The remarkable degree of levelness after washing indicates the good penetration and affinity of these dyes to the fabrics. The instrinsic conjugation in the dye structure results in the good color strength.

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