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ARTICLE

Synthesis of *bis* Azo Disperse Dyes derived from 2-Amino-4(4′-nitro phenyl)thiazole having *tertiary*-Amines as Coupling Component, their Characterization and Evolution of Dyeing Characteristics

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ABSTRACT

2-Amino-4(4′-nitro phenyl)-1,3-thiazole was used to couple with diazotized 2-amino-5(4′-nitro phenyl)-1,3,4-thiadiazole to gave mono azo disperse dye with good yield. This mono azo disperse dye was further diazotized and coupled with different tertiary amines which were couplers that yielded *bis* azo disperse dyes (**AJ_1-AJ_{15}**). The synthesized dyes were analyzed *via* elemental analysis, IR, ^{1}H NMR spectral analysis. All the synthesized dyes were applied on polyester fiber by using HTHP method and their dyeing performance and their fastness characteristics were studied.

KEYWORDS

2-Amino-5(4′-nitro phenyl)-1,3,4-thiadiazole, 2-Amino-4(4′-nitro phenyl)-1,3-thiazole, *bis* azo dyes, Polyester.

INTRODUCTION

The azo disperse dyes are common type of azo dyes, accounting for more than 70% to 72% of all disperse dyes available commercially. These disperse dyes were designed to colour cellulose acetate, but they are currently also used to dye some hydrophobic fibers like polyester [1]. Many diazo components have been widely used in the manufacturing of disperse dyes [2,3]. For the synthesis of disperse dyes 2-amino thiazole derivatives are utilized as heterocyclic diazo components [4]. The dyes, which are synthesized using 2-amino-5-nitro thiazole reported to possess high extinction coefficient. Dyes possessing enhanced fastness to light as well as fastness to sublimation were synthesized using aniline type couplers comprising one or more *N*-alkyl groups which were substituted with –CN, -OR, pyrimidine and sulphato [5,6] as coupling ingredients. An increase in patent literatures related to thiazolyl azo disperse dyes [7-9] revealed that the level of interest has increased in the last decades. A notable growth in size of data has been observed other than that of patent literature [10-12].

In this article, we have reported *bis* azo disperse dyes prepared by using 2-amino-4(4′-nitro phenyl)-1,3-thiazole. 2-Amino-5(4′-nitro phenyl)-1,3,4-thiadiazole was prepared by cyclization method by reported procedure [13-18]. 2-Amino-

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4(4′-nitro phenyl)-1,3-thiazole was prepared by using 4-nitro acetophenone, iodine and thiourea and ethanol as solvent. Then diazotization of 2-amino-5-(4′-nitro phenyl)-1,3,4-thiadiazole was carried out to be coupled with 2-amino-4(4′-nitro phenyl)-1,3-thiazole to give mono azo disperse dyes, which were further diazotized and coupled with different tertiary amines as coupling components that yielded a series of *bis* azoic disperse dyes (**AJ_1-AJ_{15}**) [19-21]. All the synthesized dyes were characterized by UV-visible, IR, ^{1}H NMR spectral analysis and applied on polyester fabric using HTHP method and the dyeing performance was evaluated. All the newly synthesized dyes showed good to excellent fastness characteristics.

EXPERIMENTAL

For the purpose of synthesis the chemicals and solvents used were of AR grade reagents and used directly. Melting points of these compounds were taken using a staurt SMP 10 melting point apparatus and are uncorrected. The purity of dyes and their R_f value were determined using thin layer chromatography (TLC) silica gel G coated Al plates of a 0.5 mm thickness (Merck) were used, methanol and toluene (4:1) was the solvent system. The spots were visualized by the use of UV radiation. The UV spectra measurement were carried out using UV-1800 Shimadzu spectrophotometer. Perkin-Elmer 1600 FTIR in KBr disc in the range 4000 to 400 cm^{-1} were used to record IR spectra. ^{1}H NMR spectra recorded with the help of Bruker Avance II 500 MHz, DMSO-d_6 was the solvent and TMS used as internal standard.

The disperse dyes **AJ_1-AJ_{15}** were used at 2% depth on polyester fabric. High temperature (130 ºC) and high pressure (24-30 psi) dyeing method was used to dye the polyester fabric samples. Light fastness, wash fastness, sublimation fastness and fastness to perspiration and rubbing were tested in accordance with ISO 105 [22,23]. Reflactance spectrometer was used to carry out Computer Color Matching (CCM) properties (L*, a*, b*, C*, h° and K/S). The value of dye bath exhaustion (%) and fixation (%) were studied as per reported procedure [22-26].

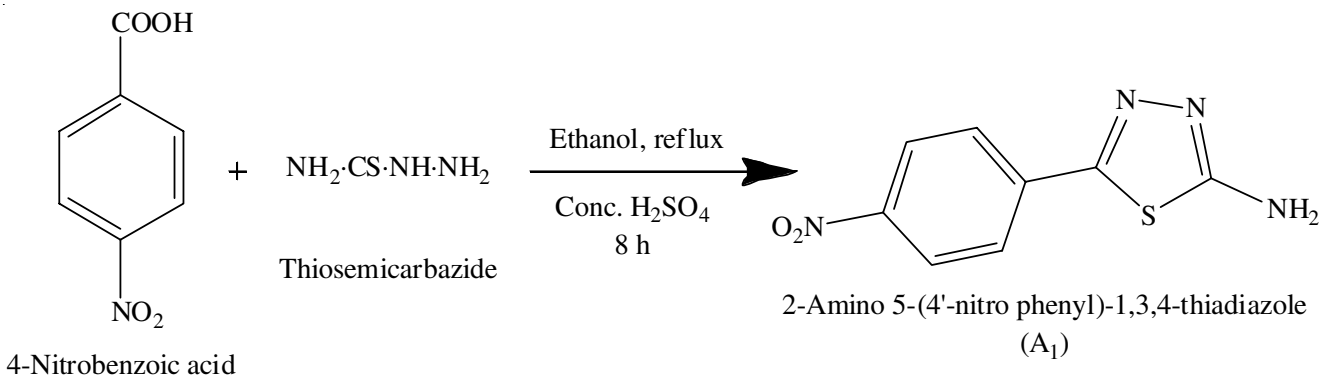
Synthesis of 2-amino-5-(4′-nitro phenyl)-1,3,4-thiadiazole (A_1) (Step-1): 4-Nitro benzoic acid (0.01 mol; 1.67 g) and thiosemicarbazide (0.01 mol; 0.91 g) was dissolved in ethanol (70 mL), 10 mL conc. H_2SO_4 was added as a cyclizing agent, to this mixture then reaction mixture was refluxed in water bath at 90 ºC for 7 h, monitoring of reaction was done by TLC (*n*-hexane:ethyl acetate), when the reaction completed the mixture was poured into crushed ice and basified with liq. ammonia which gave white solid, it was filtered, dried and recrystallized using ethanol to obtain pure compound (R_f = 0.1923) (**Scheme-I**). White solid, yield 82%, m.p. 218-220 ºC; Anal. calcd. (found) % for $C_8H_6N_4O_2S$; N, 25.21 (24.12).

Synthesis of 2-amino 4-(4′-nitro phenyl)-1,3-thiazole (S_1) (Step-2): 4-Nitro acetophenone (0.01 mol; 1.65 g) and iodine (0.01 mol; 1.26 g) was stirred in two-necked round bottom flask. Thiourea (0.02 mol; 1.52 g) was added to mixture and refluxed at 110 ºC in the oil bath till over night, hot water was added and the mixture was then heated till clear solution obtained. It was filtered while hot in condition to remove impurities, the filtrate was then cooled at room temperature and basified with NH_4OH solution to get solid, washed with cool water, dried and recrystallization was carried out with ethanol to get needle shaped crystal (R_f = 0.5909) (**Scheme-II**). Yellowish brown solid, yield 78%, m.p. 268-270 °C. Anal. calcd. (found) % for $C_9H_7N_3O_2S$; N, 7.31 (7.50).

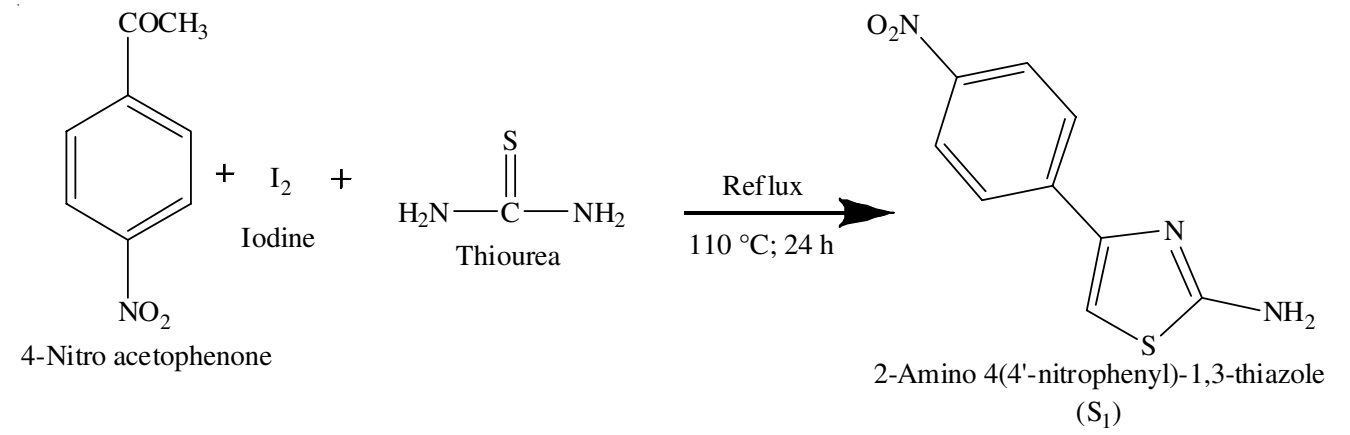
Synthesis of 4-(4′-nitrophenyl)-5-((5-(4′-nitrophenyl)-1,3,4-thiadiazol-2-yl)diazenyl)thiazol-2-amine (M) (Step-3): A solution of 2-amino-5-(4′-nitro phenyl)-1,3,4-thiadiazole (0.01 mol; 2.22 g) in mixture of glacial acetic acid and hydrochloric acid was stirred at 0-5 ºC. The solution of sodium nitrite (0.01 mol; 0.69 g) in 8 mL distilled water was added dropwise and the same temperature was maintained during addition. The resultant reaction mixture was stirred for 1 h at 0-5 ºC, with positive test for nitrous acid on starch iodide paper and excess of nitrous acid was removed by addition of required amount of sulphamic acid.

2-Amino-4-(4′-nitro phenyl)-1,3-thiazole (0.01 mol; 2.21 g) in mixture of acetic acid and hydrochloric acid was stirred at 0-5 ºC. The above mentioned diazonium salt was added dropwise at the same temperature and maintaining pH 5.5 to 6.0 by addition of 20% w/v sodium acetate. The resulting reaction mixture was stirred for 4 h at 0-5 ºC. The solid obtained was filtered off. It was washed with cold water, dried and obtained as dark yellow solid (**Scheme-III**). Yield: 85%, m.p. 184-188 ºC. Anal. calcd. (found) % for $C_{17}H_{10}N_8O_4S_2$; N, 24.66 (23.98).

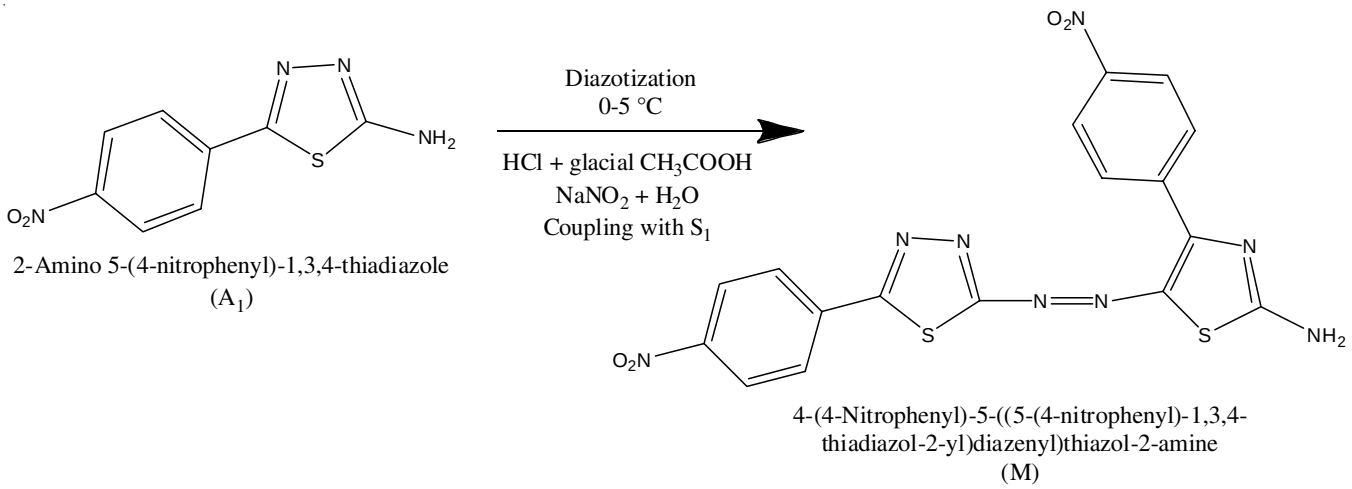
Synthesis of *bis* azo dyes (AJ_1-AJ_{15}) (Step-4): The mono azo dye M (0.01 mol; 4.54 g) was stirred in 5 mL sulphuric acid at 0-5 ºC. Sodium nitrite solution in concentrate sulphuric acid (2 mL) heated up to 60 ºC till all the solid dissolved then it was rapidly cooled at 0-5 ºC dropwise addition was done at the same temperature. This reaction mixture was stirred for 1 h



Scheme-I



Scheme-II



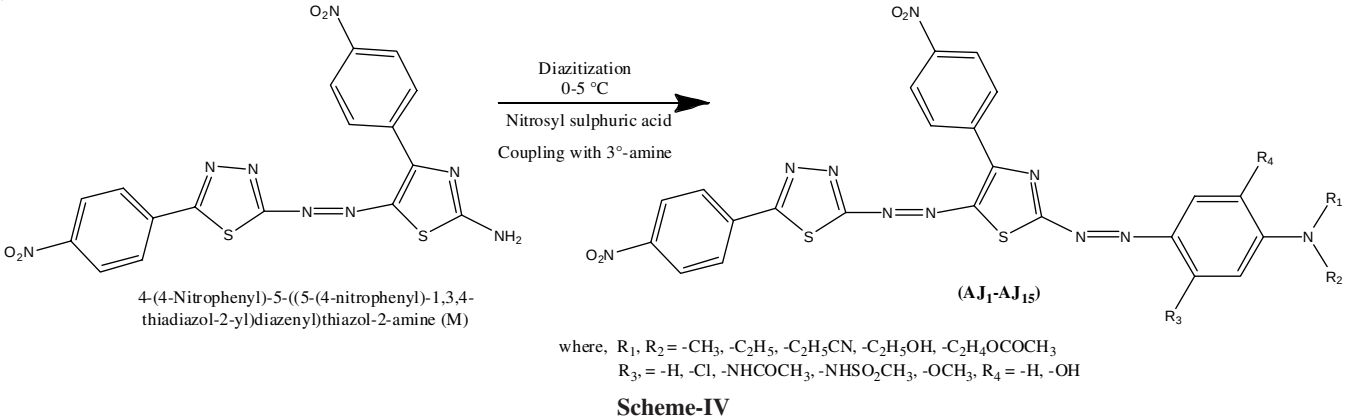
Scheme-III

at 0-5 ºC, with positive test for nitrous acid on starch iodide paper and excess of nitrous acid was removed by adding necessary amount of sulphamic acid and this diazonium salt was used for further coupling reaction.

N,*N*-Dimethyl aniline (0.01 mol; 1.21 g) in acetic acid was stirred at 0-5 ºC. To this well stirred solution above mentioned diazonium salt solution was added dropwise; maintaining pH 5.5 to 6.0 by adding solution of 20% sodium acetate. The resulting reaction mixture was stirred for further 3 h at 0-5 ºC. The coloured dye **AJ$_1$** was filtered and dried at 60-70 ºC and recrystallized from acetone (**Scheme-IV**). Remaining dyes (**AJ$_2$-AJ$_{15}$**) were synthesized by same method by using substituted tertiary amine couplers.

RESULTS AND DISCUSSION

Diazotization of compound 2-amino-5(4′-nitro phenyl)-1,3,4-thiadiazole by nitrous acid and coupled with 2-amino-4(4′-nitro phenyl)-1,3-thiazole resulted mono azo dyes (**M**). This was further diazotized using nitrosyl sulphuric acid and then coupling with substituted *tert.*-amine coupling components yielded a series of *bis* azo disperse dyes (**AJ$_1$-AJ$_{15}$**). The physico-chemical parameters of the novel synthesized dyes (**AJ$_1$-AJ$_{15}$**)



where, R_1, R_2 = -CH_3, -C_2H_5, -C_2H_5CN, -C_2H_5OH, -$C_2H_4OCOCH_3$
R_3, = -H, -Cl, -$NHCOCH_3$, -$NHSO_2CH_3$, -OCH_3, R_4 = -H, -OH

Scheme-IV

are given in Table-1. All the synthesized dyes were confirmed *via* nitrogen elemental analysis, UV-visible, IR, ^{1}H NMR spectral analysis.

IR and ^{1}H NMR spectra: The results of IR and ^{1}H NMR spectral analysis are given in Table-2 for **AJ_1** and **AJ_2** as representative dye. The IR spectrum of the synthesized dyes, exhibited absorption band at 3117-3115 cm^{-1} for asymmetric and 2991-2990 cm^{-1} for symmetric stretching is due to the –CH group. The C=C stretching observed at 1643-1641 cm^{-1}, N=N stretching vibration appeared at 1603-1601 cm^{-1}, C=N stretching vibration observed at 1528-1526 cm^{-1}, Ar-NO_2 stretching vibration appeared at 1347-1278 cm^{-1}. Absorption band of C-N stretching appeared at 1279-1169 cm^{-1}, *p*-substitued benzene ring shows absorption band at 843-841 cm^{-1}. The C-Cl stretching vibration appeared at 788-786 cm^{-1}, whereas C-S stretching vibration appeared at 716-715 cm^{-1}.

^{1}H NMR spectra of dyes **AJ_1** shows triplet in the region at δ 1.30 ppm for 6 proton of 2 methyl group and twelve proton of aromatic ring show multiplet at δ 7.19-8.18 ppm. ^{1}H NMR spectra of dyes **AJ_2** shows triplet in the region at δ 1.30 ppm for six proton of two methyl group, four hydrogen of –CH_2 group shows quatrate at δ 4.28-4.29 ppm and twelve proton of aromatic ring shows multiplet at δ 7.19-8.18 ppm.

Dyeing properties: The synthesized dyes gave good depth on polyester fabric (2% depth) as exhibited in Table-3. The visible absorption spectra of dyes were performed in DMF. Their absorption maxima were in the range of 415 to 546 nm (Table-4). Sysnthesized dyes **AJ_1-AJ_{15}** show the moderate to good light fastness and very good to excellent washing fastness. Fastness to sublimation was found to be good to very good in **AJ_4**, **AJ_9**, **AJ_{12}**, **AJ_{14}** and **AJ_{15}** and also found to be poor to good in **AJ_1**, **AJ_2**, **AJ_3**, **AJ_5**, **AJ_6**, **AJ_7**, **AJ_8**, **AJ_{10}**, **AJ_{11}** and **AJ_{13}**. Dyes **AJ_1-AJ_{15}** shows good to excellent perspiration and rubbing fastness properties on polyester fabrics (Table-3). The purity of all dyes were evaluated by TLC using methanol: toluene (4:1) as the eluting system and the R_f values of all dyes were in the range from 0.62 to 0.82. The values of K/S as colour strength for all dyes for polyester fabric were found as: **AJ_9 > AJ_5 > AJ_{11} > AJ_{14} > AJ_{15} > AJ_6 > AJ_{13} > AJ_7 > AJ_1 > AJ_4 > AJ_3 > AJ_{12} > AJ_2 > AJ_{10} > AJ_8** which is presented in Fig. 1. The colour of dyeing is expressed in terms of CIELAB values were good on polyester fabric to indicate that the dyes have good affinity to polyester fabric shown in Table-3. The exhaustion of disperse dyes for polyester ranges from 68.30% to 79.50% and the fixation of disperse dyes ranges from 75.52% to 92.27% (Table-4).

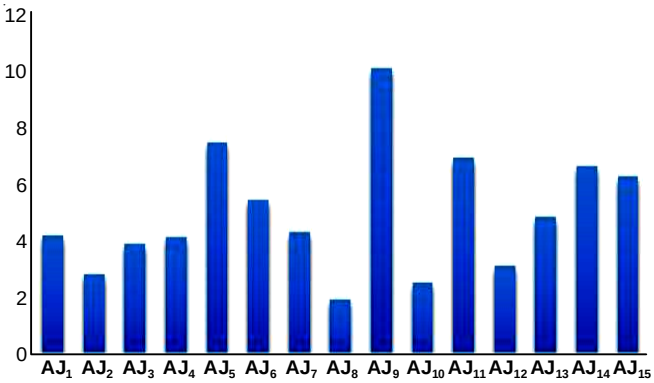


Fig. 1. Dye No. (**AJ_1-AJ_{15}**) → K/S value

TABLE-1
SUBSTITUENT AND PHYSICAL PROPERTIES OF THE DYES (**AJ_1-AJ_{15}**)

Dye No.	Substituent				m.f.	m.w.	Yield (%)	m.p. (°C)	Nitrogen (%)	
	R_1	R_2	R_3	R_4					Found	Calcd.
AJ_1	-CH_3	-CH_3	-H	-H	$C_{25}H_{18}N_{10}O_4S_2$	586.61	80	190-194	21.23	23.88
AJ_2	-C_2H_5	-C_2H_5	-H	-H	$C_{27}H_{22}N_{10}O_4S_2$	614.66	78	100-104	21.20	22.79
AJ_3	-C_2H_5	-C_2H_5	-$NHCOCH_3$	-H	$C_{29}H_{25}N_{11}O_5S_2$	671.71	75	115-119	20.80	22.94
AJ_4	-C_2H_5	-C_2H_5	-$NHSO_2CH_3$	-H	$C_{28}H_{25}N_{11}O_6S_3$	707.76	84	120-124	19.98	21.77
AJ_5	-C_2H_4OH	-C_2H_4OH	-$NHCOCH_3$	-H	$C_{29}H_{25}N_{11}O_7S_2$	703.71	58	78-82	20.24	21.89
AJ_6	-C_2H_4OH	-C_2H_4OH	-Cl	-H	$C_{27}H_{21}ClN_{10}O_6S_2$	681.10	67	145-149	18.87	20.57
AJ_7	-C_2H_4OH	-C_2H_4OH	-CH_3	-H	$C_{28}H_{24}N_{10}O_6S_2$	660.68	76	142-146	18.94	21.20
AJ_8	-C_2H_4CN	-C_2H_4CN	-H	-H	$C_{29}H_{20}N_{12}O_4S_2$	664.68	74	150-154	24.10	25.29
AJ_9	-C_2H_5	-C_2H_4CN	-H	-H	$C_{28}H_{21}N_{11}O_4S_2$	639.67	63	148-152	23.12	24.09
AJ_{10}	-C_2H_4CN	-C_2H_4CN	-CH_3	-H	$C_{30}H_{22}N_{12}O_4S_2$	678.71	79	85-89	22.4	24.77
AJ_{11}	-C_2H_4CN	-$C_2H_4OCOCH_3$	-$NHCOCH_3$	-H	$C_{32}H_{26}N_{12}O_7S_2$	754.76	80	158-162	20.24	22.27
AJ_{12}	-C_2H_4CN	-C_2H_4OH	-$NHCOCH_3$	-OH	$C_{30}H_{24}N_{12}O_7S_2$	728.72	69	120-124	21.22	23.07
AJ_{13}	-$C_2H_4OCOCH_3$	-$C_2H_4OCOCH_3$	-Cl	-H	$C_{31}H_{25}ClN_{10}O_8S_2$	765.17	73	80-84	17.73	18.31
AJ_{14}	-C_2H_5	-C_2H_4CN	-$NHCOCH_3$	-H	$C_{30}H_{24}N_{12}O_5S_2$	696.72	68	134-138	23.20	24.13
AJ_{15}	-C_2H_4CN	-C_2H_5	-H	-H	$C_{28}H_{21}N_{11}O_4S_2$	639.67	72	168-172	22.27	24.09

TABLE-2
IR AND ^{1}H NMR SPECTRAL PROPERTIES OF SYNTHESIZED DYES (**AJ_1** and **AJ_2**)

Dye No.	IR (KBr, cm^{-1})	^{1}H NMR (500 MHz DMSO-d_6) chemical shift in δ_H ppm
AJ_1	3116.21 (CH str. asym.), 2990.63 (CH str. sym.), 1641.75 (C=C str.), 1601.22 (N=N str.), 1526.82 (C=N str.), 1347.09 (Ar-NO_2 str.), 1278.92 (C-N str.), 841.93 (*p*-sub. benzene), 787.35 (C-Cl str.), 715.65 (C-S str.)	1.30 (t, 6H, 2-CH_3), 7.19-8.18 (m, 12H, Ar-H)
AJ_2	3116.65 (CH str. asym.), 2990.08 (CH str. sym.), 1642.55 (C=C str.), 1602.32 (N=N str.), 1527.65 (C=N str.), 1278.54 (Ar-NO_2 str.), 1169.84 (C-N str.), 842.90 (*p*-sub. benzene), 786.87 (C-Cl str.), 715.88 (C-S str.)	1.30 (t, 6H, 2-CH_3), 4.28-4.29 (quartet, 4H, 2-CH_2), 7.19-8.18 (m, 12H, Ar-H)

TABLE-3
SHADE AND FASTNESS PROPERTIES OF DYES ON POLYESTER FABRIC (AJ_1-AJ_{15})

Dye No.	Shade on polyester	Fastness to		Sublimation on polyester		Perspiration		Rubbing	
		Light	Washing	Staining at 180 °C	Staining at 210 °C	Acid	Basic	Dry	Wet
AJ_1	Amberglow	4	4-5	2	1	4-5	4	4	4
AJ_2	Ruby	4	4	3	2-3	5	4-5	4	4
AJ_3	Ruby pink	3-4	4	2-3	2	4	4-5	4-5	4
AJ_4	Cerise	4-5	4-5	3-4	3	3-4	4	4-5	4
AJ_5	Outrageous	4	4	3	2-3	4	4	4	3-4
AJ_6	Ginger orange	4	4	3	2	4	4	4	4
AJ_7	Ferrari	3-4	4	2	1	4-5	4-5	4	4
AJ_8	Princeton orange	4-5	4-5	2-3	1-2	4-5	4	5	4
AJ_9	Orange yellow	4	4-5	3-4	3	3-4	4	4-5	3-4
AJ_{10}	Aerospace orange	3-4	4	3	2-3	4-5	4	4	4
AJ_{11}	Sun rise	4-5	5	2-3	1-2	4	4	4	4
AJ_{12}	Punch	4	4-5	3-4	3	4	4	4-5	4
AJ_{13}	Brick	3-4	4	3	2-3	4-5	4-5	4	3-4
AJ_{14}	Chestnut	4-5	4-5	3	3	4	4	4-5	4
AJ_{15}	Aerospace orange	4	4	3-4	3	4-5	4-5	4-5	4

Light fastness: 1-poor, 2-slight, 3-moderate, 4-fair, 5-good, 6-very good, 7-excellent. Fastness of washing, sublimation, perspiration, rubbing: 1-poor, 2-fair, 3-good, 4-very good, 5-excellent.

TABLE-4
UV-VISIBLE SPECTROSCOPIC DATA AND COLOR COORDINATION OF THE DYE (AJ_1-AJ_{15})

Dye No.	λ_{max} (nm)	R_f	L*	a*	b*	C*	H°	K/S value	Exhaustion (%)	Fixation (%)
AJ_1	419.6	0.74	62.59	17.52	17.48	24.75	44.94	4.21	78.34	90.20
AJ_2	533.3	0.80	52.64	25.65	5.78	26.30	12.7	2.81	68.30	75.52
AJ_3	545.2	0.68	50.48	37.82	-1.58	37.86	357.6	3.90	78.57	90.23
AJ_4	534.7	0.69	51.66	40.12	4.80	40.41	6.82	4.15	79.30	92.27
AJ_5	415.2	0.72	74.80	14.20	45.50	47.90	70.20	7.50	72.23	80.23
AJ_6	420.2	0.71	55.80	25.80	9.20	27.61	20.21	5.48	74.28	84.53
AJ_7	504.4	0.78	52.94	31.50	24.20	39.72	37.54	4.34	70.30	81.20
AJ_8	497.7	0.74	70.60	27.41	33.08	42.96	50.35	1.91	70.68	80.17
AJ_9	417.2	0.82	67.20	42.31	57.20	70.02	55.20	10.20	78.65	88.87
AJ_{10}	504.5	0.62	65.10	31.88	28.76	42.94	42.06	2.5	79.50	90.78
AJ_{11}	422.3	0.80	67.25	35.50	32.20	48.30	44.57	7.01	71.23	84.26
AJ_{12}	422.3	0.72	55.81	17.40	5.44	18.23	17.37	3.12	72.35	84.57
AJ_{13}	418.6	0.73	53.57	18.22	13.06	22.42	35.63	4.85	74.35	89.97
AJ_{14}	416.5	0.71	56.86	6.87	15.60	17.05	66.22	6.70	73.25	86.30
AJ_{15}	480.2	0.80	65.30	42.20	51.50	67.30	52.40	6.30	75.50	82.28

Conclusion

A series of *bis* azo disperse dyes was synthesized by the conventional method and their dyeing performance was examined on polyester fabrics. Synthesized *bis* azo disperse dyes gave wide range of properties. Good strength of colour on polyester fabric after washing shows the good affinity towards polyester fabrics.

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