Original Research Paper

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Chemistry

SYNTHESIS, CHARACTERIZATION AND DYEING PERFORMANCE OF MONO AZO DISPERSE DYES BASED ON 2-AMINO 5-(4'-NITRO PHENYL) 1,3,4-THIADIAZOLE MOIETY

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ABSTRACT In this paper synthesis of some new mono azo disperse dyes based on 2-amino 5-(4'-nitro phenyl) 1,3,4-thiadiazole moiety has been reported. Preparation of mono azo disperse dyes via condensation and finally diazotization of substituted primary amine and condensed with N-(4-(4'-chlorophenyl)thiazol-2-yl)-2-((5-(4'-nitrophenyl)-1,3,4-thiadiazol-2-yl)amino)acetamide (RR) to give a series of mono azo dyes (RR_1-RR_{15}). All the dyes were characterized by IR, ^{1}H NMR, UV-Visible and elemental analysis and their dyeing performance evaluated using High Temperature High Pressure method (HTHP) at 130°C on polyester fabric. All dyes gave good to excellent fastness properties.

KEYWORDS : Thiadiazole, Thiazole, Mono azo disperse dyes, Primary amine, Fastness properties.

INTRODUCTION

The modern era of colour Chemistry began in 1856 when William Henry Perkins synthesized the first "coal tar" dye-mauve. Since then thousands of colorants have been synthesized to colour everything from textiles to food [1-2], and dyes and pigments [3]. According to the colour index [4], disperse dye is a class of sparingly water soluble dyes originally introduced for dyeing cellulose acetate and usually applied from fine aqueous dispersion.

Disperse dyes have also been defined as sparingly water soluble, nonionic dyes applied to hydrophobic fibres from aqueous dispersions [5]. The most dominant group of disperse dyes is the azo disperse dyes which account for over 70% of all disperse dyes manufactured. Disperse dyes, were originally developed to dye cellulose acetate, but now usefully applied to other hydrophobic fibres such as polyester [6]. In recent years many diazo components have been broadly used in production of disperse dyes [7-8].

Here we focused on the synthesis of disperse dyes based on 2-amino 5(4'-nitro phenyl)1,3,4-thiadiazole moiety, which is prepared according to reported method [9-12]. Chloro acetylation of 2-amino 4(4'-chloro phenyl) 1,3-thiazole [13-20] and condensation with 2-amino 5-(4'-nitro phenyl) 1,3,4-thiadiazole [17] to give a new heterocyclic moiety (RR), finally diazotization of substituted primary amine and condensed with RR to give a series of successive dyes (RR_1-RR_{15}).

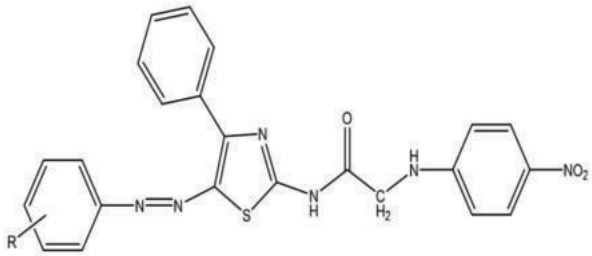
MATERIALS AND METHODS:

All chemicals were analytical grade reagents and were used directly. Melting points were measured using a Stuart SMP 10 melting point apparatus and are uncorrected. The purity and R_f value of all dyes were determined by thin-layer chromatography (TLC) using silica gel-G coated Al-plates (0.5 mm thickness, Merck) using methanol and toluene (4:1) as the solvent system and spots were visualized under UV radiation. The UV spectra were measured on UV-1800 Shimadzu Spectrophotometer. IR spectra were recorded on Perkin-Elmer 1600 FTIR in KBr disc in the range between 4000 cm^{-1} to 400 cm^{-1}. ^{1}H NMR spectra were taken on a Bruker Avance II 500 MH_Z NMR in DMSO-d_6 as solvent and TMS as internal standard.

The disperse dyes RR_1-RR_{15} were applied at 2% depth on polyester fabric. The dyeing of the polyester fabric samples was carried out by high temperature (130°C) and high pressure (24-30psi) dyeing method. Fastness properties to light, wash, sublimation, perspiration and rubbing were evaluated in accordance with ISO 105 [21-22]. Computer Color Matching properties (L*, a*, b*, C*, h°, and K/S) were recorded on a reflectance spectrometer. Dye bath exhaustion (%) and fixation (%) values were determined according to reported method [21-25].

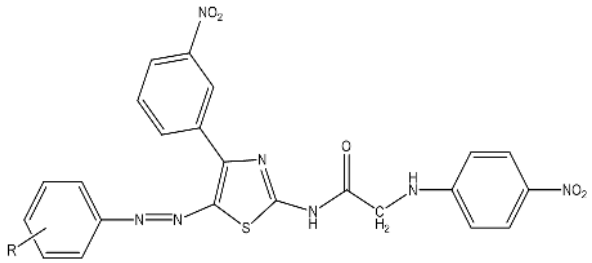
PREVIOUS WORK:

S. K. Zadafiya et.al (2012) [26]



Where R= -H, $-CH_3$ $-NO_2$, -Cl, -Br etc.

Sandip K. Patel et al (2014) [17]



Where R= -H, $-CH_3$, $-NO_2$, -F, -Cl, -Br, -OH, $-OCH_3$, $-NHCOCH_3$ etc.

REACTION SCHEME:

Step-1: Synthesis of 2-amino 5-(4'-nitro phenyl)1,3,4-thiadiazole (A_1): Mixture of 4-Nitro benzoic acid (0.01 mole;1.67 gm) and thiosemicarbazide (0.01 mole;9.1 gm) was dissolved in ethanol (70 mL) and 10 mL conc. H_2SO_4 was added as a cyclizing agent, reaction mixture was refluxed in water bath at 90°C for 7 hrs. reaction was monitored by TLC (n- hexane: ethyl acetate), after completion of reaction, mixture was poured into crushed ice and basified with liq. ammonia to give white solid, filtered, dried and recrystallized from ethanol to give pure compound.(R_f = 0.1923). White solid, yield 82%, m.p. 218-220°C; Anal. Calc. For $C_8H_6N_4O_2S$; N, 25.21%; Found: N, 24.12%

Step-2: Synthesis of 2-amino 4-(4'-chloro phenyl) 1,3-thiazole (N_1): 4-chloro acetophenone (0.01 mole;1.54 gm) and iodine (0.01 mole; 1.26 gm) was stirred in 2 necked RBF. Then thiourea (0.02 mole;1.52 gm) was added and mixture was refluxed at 110°C in oil bath for over night, hot water was then added to it and heated till clear solution obtained, filtered while hot in condition to remove impurities, and filtrate was cooled at room temperature and basified with NH_4OH

solution to give solid. It was washed with cold water, dried and recrystallized from ethanol to give light yellow needle shaped crystal. (R_f = 0.4090). Yellow solid, yield 85%, m.p. 154°C; Anal. Calc. For $C_9H_7ClN_2S$; N, 13.22%; Found: N, 13.30%

Step-3: Synthesis of 2-chloro-N-(4-(4'-chloro phenyl)thiazol-2-yl)acetamide: 2-Amino 4-(4'-chloro phenyl) 1,3-thiazole (0.01 mole; 2.1 gm) was dissolved in glacial CH_3COOH (50 mL) containing saturated solution of CH_3COONa the solution was heated till clear solution obtained then cooled below 5°C, chloro acetyl chloride (0.02 mole; 1.59 mL) was added drop wise with continuous stirring during time period of 30 minutes. After completion of addition, the mixture was stirred for 1 hr. below 5°C, then at 25-30°C for 1 hr., temperature was gradually increased to 80°C and stirred for 1 hr. followed by stirring at room temperature for 6 hours, after completion of reaction (checked by TLC) mixture was dumped into crushed ice to give white solid, it was filtered, dried and recrystallized from ethanol. (R_f = 0.7407). Yellow solid, yield 70%, m.p. 135-137°C; Anal. Calc. For $C_{11}H_8Cl_2N_2OS$; N, 9.76%; Found: N, 9.70%

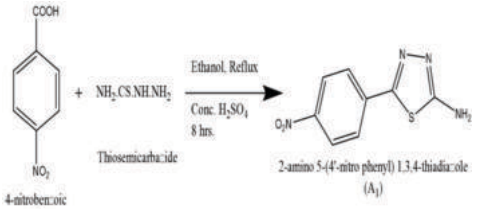
Step-4: Synthesis of N-(4-(4'-chlorophenyl)thiazol-2-yl)-2-((5-(4'-nitrophenyl)-1,3,4-thiadiazol-2-yl)amino)acetamide (RR): 2-chloro-N-(4-(4'-chlorophenyl)thiazol-2-yl)acetamide (0.01 mole;2.87 gm) was dissolved in glacial CH_3COOH (80 mL), mixture of 2-amino 5-(4'-nitro phenyl)1,3,4-thiadiazole (0.01 mole;2.22 gm) and K_2CO_3 (0.01 mole;1.38 gm) was added portion wise, then reaction mixture was refluxed at 80°C for 8 hrs., after completion of reaction (checked by TLC) mixture was poured into crushed ice to give yellow solid, which was filtered, dried and recrystallized from ethanol to give pure compound (R_f = 0.4782). Golden Yellow solid, yield 72%, m.p. 162-164°C; Anal. Calc. For $C_{19}H_{13}ClN_6O_3S_2$; N, 17.77%; Found: N, 17.50%

Step-5: Diazotization of primary amine and coupling with N-(4-(4'-chlorophenyl)thiazol-2-yl)-2-((5-(4'-nitrophenyl)-1,3,4-thiadiazol-2-yl)amino)acetamide(RR_1-RR_{15}): Aniline (0.01 mole; 0.93 gm) was dissolved in HCl (5 mL) and cooled to 0-5°C in an ice bath, a solution of $NaNO_2$ (0.01 mole; 0.69 gm) in water (10 mL) was cooled to 0-5°C, the solution of aniline added to the solution of $NaNO_2$ with taking care of no evolution of yellow-brown gas during the time period of 10 minutes. Stirring was continued, maintaining the temperature at 0-5°C. The reaction mixture was stirred further for an hour keeping constant positive test on starch iodide paper. The excess of nitrous acid was removed by using sulphamic acid. The resulting solution was used for coupling reaction.

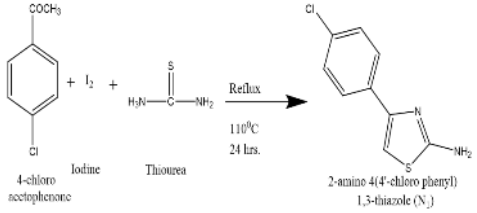
N-(4-(4'-chlorophenyl)thiazol-2-yl)-2-((5-(4'-nitrophenyl)-1,3,4-thiadiazol-2-yl)amino)acetamide (RR) (0.01 mole;4.725 gm) was dissolved in glacial CH_3COOH (40 mL) then cooled at 0-5°C in an ice bath. The above mention diazonium chloride solution was added drop wise over a period of 10 minutes, maintaining the pH 7.0 to 7.5 by simultaneous addition of aqueous sodium acetate (20% w/v), further the reaction mixture was stirred for 3 hrs. at 0-5°C. The solid dye RR_1 was yielded. The dye was filtered, washed with cold water to remove acid completely and dried, recrystallized from acetone. Remaining dyes RR_2-RR_{15} were synthesized following same method by using different substituted primary amine.

Reaction scheme:

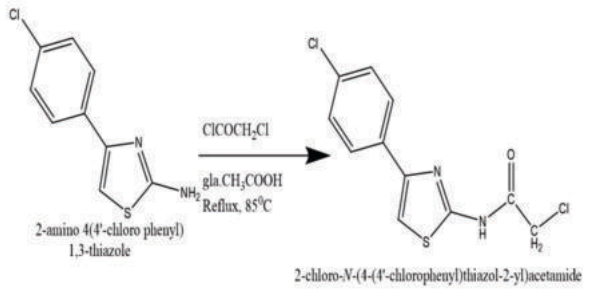
Step-1: Synthesis of 2-amino 5-(4'-nitro phenyl)1,3,4-thiadiazole (A_1):



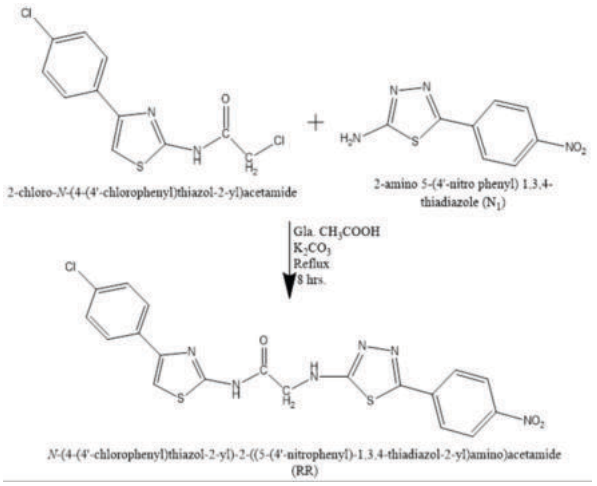
Step-2: Synthesis of 2-amino 4-(4'-chloro phenyl) 1,3-thiazole (N_1):



Step-3: Synthesis of 2-chloro-N-(4-(4'-chloro phenyl)thiazol-2-yl)acetamide:



Step-4: Synthesis of N-(4-(4'-chlorophenyl)thiazol-2-yl)-2-((5-(4'-nitrophenyl)-1,3,4-thiadiazol-2-yl)amino)acetamide (RR):



Step-5: Diazotization of primary amine and coupling with N-(4-(4'-chlorophenyl)thiazol-2-yl)-2-((5-(4'-nitrophenyl)-1,3,4-thiadiazol-2-yl)amino)acetamide(RR_1-RR_{15}):

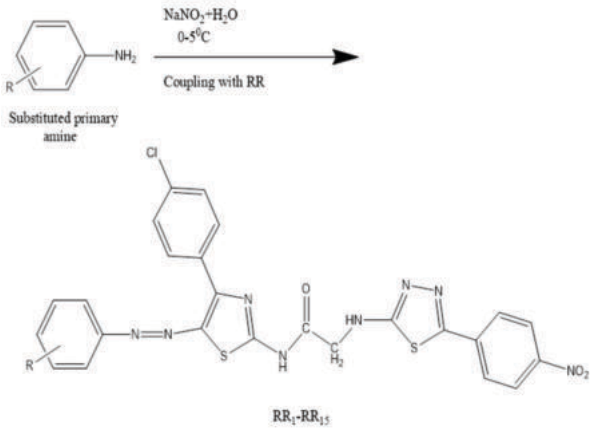


Table-1: The Characterization Data Of Dyes (RR_1-RR_{15}):

Dye No.	Substituent (R)	M. F.	M.W.	Melting point (°C)	Yield %	%Nitrogen	
						Found	Calc.
RR_1	-H	$C_{25}H_{17}ClN_8O_3S_2$	577.03	168-170	82	19.42	19.40
RR_2	2-NO_2	$C_{25}H_{16}ClN_9O_5S_2$	622.03	172-174	75	20.27	20.25
RR_3	4-NO_2	$C_{25}H_{16}ClN_9O_5S_2$	622.03	178-181	77	20.27	20.24
RR_4	4-Cl	$C_{25}H_{16}Cl_2N_8O_3S_2$	611.48	163-165	62	18.33	18.30
RR_5	4-F	$C_{25}H_{16}ClFN_8O_3S_2$	595.02	168-172	68	18.83	18.80
RR_6	4-CH_3	$C_{26}H_{19}ClN_8O_3S_2$	591.06	175-180	85	18.96	18.94
RR_7	2-Cl, 4-NO_2	$C_{25}H_{15}Cl_2N_9O_5S_2$	656.47	186-190	74	19.20	19.17
RR_8	2-NO_2, 4-Cl	$C_{25}H_{15}Cl_2N_9O_5S_2$	655.00	177-181	63	19.20	19.17
RR_9	2,5-di chloro	$C_{25}H_{15}Cl_3N_8O_3S_2$	645.92	180-184	81	17.35	17.34
RR_{10}	2-CN, 6-Br, 4-NO_2	$C_{26}H_{14}BrClN_{10}O_5S_2$	725.94	183-190	68	19.30	19.28

RR_{11}	2-OCH_3, 3-NO_2	$C_{26}H_{18}ClN_9O_6S_2$	652.06	167-170	61	19.33	19.29
RR_{12}	3-CH_3	$C_{26}H_{19}ClN_8O_3S_2$	591.06	189-192	72	18.96	18.94
RR_{13}	3-NO_2	$C_{25}H_{16}ClN_9O_3S_2$	622.03	195-200	83	20.27	20.24
RR_{14}	2-CH_3	$C_{26}H_{19}ClN_8O_3S_2$	591.06	187-191	67	18.96	18.93
RR_{15}	4-Br	$C_{25}H_{16}BrClN_8O_3S_2$	655.93	174-178	80	17.08	17.05

Table-2: Shade And Fastness Properties Of Dyes (RR_1-RR_{15}) On Polyester Fabrics:

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
RR_1	Orange	4	4	3	2	4	4-5	4-5	4
RR_2	Reddish orange	3	5	2	2	4	4-5	4-5	4
RR_3	Pale violet red	4-5	4	3	2	4	4	4	4
RR_4	Yellowish orange	4	4-5	2-3	2	4-5	5	4	4
RR_5	Reddish orange	4	3-4	2	2	4	4	5	4
RR_6	Golden yellow	4	4	3-4	3	4	3-4	5	5
RR_7	Plum	3-4	3	3	3	5	5	4	4
RR_8	Brick red	3	4	4	3	4	5	4	4
RR_9	Yellowish red	4	4	3	2-3	5	4-5	3-4	4
RR_{10}	Thistle	5	4	2	1-2	4	4-5	5	5
RR_{11}	Tropical orange	4	3-4	3-4	3-4	3-4	4	4	4
RR_{12}	Bright yellow	3-4	5	3	2	4-5	3	5	4
RR_{13}	Fire	4	4	3	2	5	4-5	5	4
RR_{14}	Chrome yellow	4	4	2-3	1-2	4	5	5	4
RR_{15}	Mari gold	4	4	3	3	4	4-5	4	4

Abbreviations:
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.

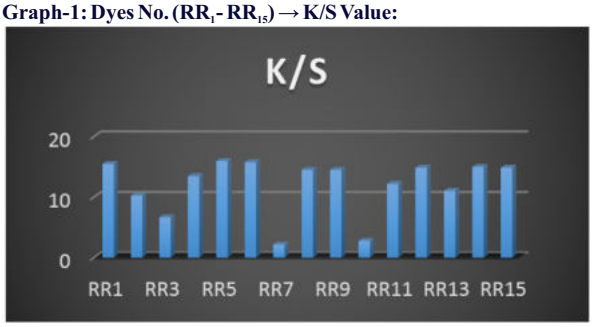
Table-3: UV-Visible Spectroscopic Data And Color Coordinates Of The Dyes (RR_1-RR_{15}):

Dye No.	λ_{max}(nm)	R_f	L^*	a^*	b^*	C^*	h^o	K/S Value	% Exhaustion	% Fixation
RR_1	463.04	0.82	57.31	33.68	62.92	71.36	61.84	15.46	82.23	73.70
RR_2	479.44	0.74	47.67	34.91	36.43	50.45	46.22	10.25	82.50	81.20
RR_3	516.56	0.84	46.75	39.25	16.94	42.75	23.35	6.65	72.30	75.60
RR_4	473.46	0.88	60.28	34.50	64.10	72.80	61.71	13.45	75.80	78.90
RR_5	472.95	0.78	45.12	48.45	42.88	64.70	41.51	15.96	78.00	78.20
RR_6	472.99	0.74	61.21	33.62	69.84	77.51	64.29	15.74	82.75	75.67
RR_7	546.11	0.67	54.50	25.03	1.36	25.06	3.12	2.18	84.56	76.62
RR_8	498.22	0.80	41.20	42.99	32.11	53.65	36.76	14.5	85.50	78.30
RR_9	488.22	0.72	51.57	47.81	49.52	68.83	46.01	14.5	86.42	89.20
RR_{10}	568.40	0.70	46.50	11.81	-6.50	13.48	331.18	2.79	74.24	72.50
RR_{11}	488.78	0.74	49.78	42.06	40.77	58.58	44.11	12.18	76.12	72.60
RR_{12}	428.15	0.85	55.95	30.26	58.19	65.59	62.52	14.86	78.90	73.70
RR_{13}	479.15	0.86	61.53	36.51	60.61	70.76	58.93	11.03	77.42	85.67
RR_{14}	463.13	0.84	56.78	36.38	61.23	71.22	59.28	14.99	72.25	75.08
RR_{15}	473.15	0.72	58.58	39.22	63.88	74.96	58.45	14.86	79.91	82.30

Table-4: IR and ^{1}H NMR Spectral Properties Of Synthesized Dyes (RR_7& RR_{15}):

Dye No.	IR (KBr, cm^{-1})	^{1}H NMR (500 MH_Z DMSO-d_6) chemical shift in δ_H ppm
RR_7	3468.44 (-NH str.), 3104.66 (-C-H str.), 1708.86 (-C=O str.), 1582.43 (-C=C str.), 1524.92 (-N=N str.), 1480.86 (-C-S-C str. In thiazole), 1312.48 (Ar-NO_2 str.), 1203.65 (-C-N str.), 718.53 (Ar-Cl str.).	4.35-4.40 (d, 2H, -CH_2), 6.5-8.5 (m, 11H, Ar-H), 8.8 (s, 1H, -NH), 12.27 (s, 1H, -NH)
RR_{15}	3431.79 (-NH str.), 3110.27 (-C-H str.), 2991.15 (-C-H str. Asymm.), 1718.53 (-C=O str.), 1576.17 (-C=C str.), 1516.64 (-N=N str.), 1478.91 (-C-S-C str. In thiazole), 1358.58 (Ar-NO_2 str.), 1198.53 (-C-N str.), 717.48 (-C-Cl str.), 608.71 (Ar-Br str.).	4.35-4.39 (d, 2H, -CH_2), 7.4-8.5 (m, 12H, Ar-H), 9.0 (s, 1H, -NH), 12.26 (s, 1H, -NH)

Graph-1: Dyes No. (RR_1- RR_{15}) → K/S Value:



RESULT AND DISCUSSION:
Chloroacetylation of 2-amino 4-(4'-chloro phenyl) 1,3-thiazole with chloroacetyl chloride at 0-5°C gave 2-chloro-N-(4-(4'-chloro phenyl)thiazol-2-yl)acetamide which was condensed with 2-amino 5-(4'-nitro phenyl)1,3,4-thiadiazole to give N-(4-(4'-chlorophenyl) thiazol-2-yl)-2-((5-(4'-nitrophenyl)-1,3,4-thiadiazol-2-yl)amino)acetamide (RR). Finally diazotization of substituted primary amine and coupling with N-(4-(4'-chlorophenyl)thiazol-2-yl)-2-((5-(4'-nitrophenyl)-1,3,4-thiadiazol-2 yl)amino)acetamide (RR) to yield the series of dyes (RR_1-RR_{15}). Physical appearance of newly synthesized dyes was yellow, orange, red, pink coloured crystalline solid. The newly synthesized compounds were confirmed by elemental analysis IR, ^{1}H NMR, UV-absorbance data.

Spectral properties of Dyes:
IR and ^{1}H NMR Spectra:
IR and ^{1}H NMR spectral analysis has been given in Table-4 for RR_7 and RR_{15} as representative dyes.

The IR spectra of RR_7 and RR_{15} dyes showed a peak at 3469-3431 cm^{-1} corresponding to the –NH group. The IR spectra of the dyes showed the stretching vibrations of alkyl group appeared at 3111-3104 cm^{-1} and for asymmetric at 2991.15 cm^{-1}, stretching for amide group at 1719-1708 cm^{-1}. Also –C=C stretching appeared at 1583-1576 cm^{-1}, the –N=N group streching vibration appeared at 1525-1516 cm^{-1}, the –C-S-C stretching in thiazole appeared at 1480-1478 cm^{-1}, Ar-NO_2 streching vibration at 1359-1312 cm^{-1}, C-N stretching at 1204-1198 cm^{-1}, Ar-X stretching vibration apperared at 719-608 cm^{-1}.

^{1}H NMR spectra of dye RR_7 shows doublet in the region at δ 4.35-4.40 ppm for two protons of CO-CH_2-NH group, one proton of –NH attached to –CH_2 appears singlet at δ 8.8 ppm, one proton of –NH attached to thiazole ring appears singlet at δ 12.27 ppm, which shows the deshielding effect of –C=O to this proton lies at higher δ value, and eleven proton of aromatic ring shows multiplet at δ 6.5-8.5 ppm.

^{1}H NMR spectra of dye RR_{15} shows doublet in the region at δ 4.35-4.39 ppm for two protons of CO-CH_2-NH group, one proton of –NH attached to –CH_2 appears singlet at δ 9.0 ppm, one proton of –NH attached to thiazole ring appears singlet at δ 12.26 ppm, which shows the deshielding effect of –C=O to this proton lies at higher δ value, and twelve proton of aromatic ring shows multiplet at δ 7.4-8.5 ppm.

Dyeing Properties:
All the synthesized dyes RR_1-RR_{15} were applied on polyester fibre in 2% depth according to literature procedure by HTHP method [23, 27-28]. The variation in the hues of the dyed fabric results from the alternation in the coupling components. The remarkable degree of levelness and brightness after washing indicates good penetration and excellent affinity of these dyes to the fabric. The colour strength (K/S) values of all dyes for polyester fabric were found to be in the order: RR5>RR6>RR_1>RR_{14}>RR_{12}=RR_{15}>RR_8=RR_9>RR_4>RR_{11}>RR_{13}>RR_2>RR_3>RR_{10}>RR_7 which is presented by graph-1.

Fastness properties of dyed fabric:
The fastness ratings and colour shade of all dyes are shown in Table-2. The light fastness properties were assessed in accordance with BS:

1006-1378 and the wash fastness test in accordance with IS: 765-1979. The light fastness of all the dyes exhibited the rating 3-6 for polyester which is showed that light fastness was moderate to very good. The wash fastness of all the dyes had the rating 3-5 for polyester which indicated that wash fastness was good to excellent and the sublimation fastness of all the dyes had the rating 1-4 for polyester which showed sublimation fastness was poor to very good. The perspiration fastness in acid and basic medium of all the dyes had the ratings 3-5 for polyester which indicated that perspiration fastness was good to excellent. The rubbing fastness in dry and wet condition of all dyes had the ratings 3-5 for polyester which indicated that rubbing fastness was good to excellent.

CONCLUSIONS

Substituted primary amine was diazotized and coupled with N-(4-(4'-chlorophenyl)thiazol-2-yl)-2-((5-(4'-nitrophenyl)-1,3,4-thiadiazol-2-yl)amino)acetamide (RR) to give a series of dyes (RR_1-RR_{15}). Physical appearance of newly synthesized dyes was yellow, orange, red, pink coloured crystalline solid. The newly synthesized compounds were confirmed by elemental analysis IR, ^{1}H NMR, UV-absorbance spectral data. These dyes gave yellow, orange, red, pink, and violet shade on polyester fabric and showed wide range of fastness properties.

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