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Thaizole based disperse dyes and their dyeing application on polyester fiber and their antimicrobial activity

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ABSTRACT

Various diazotized aryl amines were coupled with N-(2,4-dinitrophenyl)-2-[(4-phenyl-1,3-thiazol-2-yl)amino]acetamide to give the corresponding various azo disperse dyes (A1-A13). These dyes were applied to polyester fabric by HTHP method and their fastness properties were evaluated. Dyes were characterized by IR, elemental analysis and NMR spectral studies. These dyes showed very good anti-bacterial and anti-fungal activities.

Keywords: 2-amino-4-phenylthiazole, chloroacetylchloride, fastness, antibacterial, antifungal.

INTRODUCTION

Disperse dyes are organic colours having less water solubility, these are applied in colloidal aqueous dispersions to hydrophobic textile fibers in which the dyes literally dissolve and produce desired coloration. The development of disperse dyes is due to significant increase in the world production of polyester fibers[1] as compared to other fibers. Over 90% of disperse dyes usage is for the coloration of polyester and its blends. A monoazo dye with a heterocyclic system[2-8] is very useful class of disperse dyes. Derivatives of 2-aminothiazole[9-10] have a long history of use as heterocyclic components for various disperse dyes. The main approach of our work was to synthesize [11-16] the disperse dyes consisting thiazole with azo substituent which were further utilized to dye some hydrophobic fibers.

MATERIALS AND METHODS

All the chemicals used in dye synthesis were of laboratory grade and were further purified by crystallization and distillation. Melting points were determined by open capillary method. Thin layer chromatography was performed using silica gel (E. Merck) plates to access the reaction and purity of the synthesized compounds. The visible absorption spectra were measured using UV-160PC Simadzu Spectrophotometer. A Shimadzu-8400 FT-IR spectrometer was used for recording the IR spectra of the dyes using KBr pellets in the range between 4000 and 400 cm-1. Elemental analyses were estimated by means of a Perkin Elmer (USA) 2400 Series. ^{1}H-NMR spectra were recorded on a Varian (400 MHz) instrument using tetramethylsilane (TMS) as an internal standard and DMSO-d_6 as a solvent. The diazo component and 2-amino-4-phenylthiazole were prepared according to the literature. The synthesized dyes were screened for their antimicrobial activity[17-18] using the Kirby-Bauer method [17] measuring zone of inhibition in mm against different bacterial strains such as Escherichia coli, Pseudomonas aeruginosa, Staphylococcus aureus and fungi Candida Albicans at 40µg/ml concentration. Standards were used for comparison purpose like Metranidazole, Amplicilline and Flucanazole in antimicrobial activity. The dyeing of the polyester fabric samples was carried out by HTHP dyeing method [19-21]. The characteristic data of different molecules were studied and further applied on polyester fibers to study their light, wash and sublimation fastness [9-10].

Preparation of N-(2,4-dinitrophenyl)-2-[(4-phenyl-1, 3-thiazol-2-yl) amino Aa.

In a 250 ml R.B.F., 2, 4-dinitro aniline (3.66g, 0.02 mole) in dry benzene (60 ml) was cooled to 0-5°C add 2-3 drop TEA. Chloroacetyl chloride (2.26ml, 0.02 mole) dissolved in dry benzene (20ml) was slowly added to RBF with vigorous stirring. When addition was complete, the reaction mixture was refluxed for 3hr. Then adds 2-amino-4-phenyl thiazole (3.52gm 0.02mole) and then further reflux for 4hr. Benzene was removed in vacuo. The crude product was dried and recrystalised from ethanol., Yield: 67% M.P.: 124°C, Aa: IR (KBr, cm^{-1}) 3284 (N-H str.), 1685 (C=O str. of amide),1512 (-NO_2 str.),1341 (C-N str. of tertiary amine), 1265 (C-N str. of secondary amine); ^{1}H NMR (399.76 MHz, DMSO),δ, ppm: 4.211 (S,2H,-CO-CH_2), 8.245 (S, 2H, two –NH group),7.243 (S,1H, thiazole ring), 8.750 (1H,dd, Ha),8.491 (1H,dd, Hb,), 8.183 (1H,dd, Hc), 8.103-7.230 (5H,m, aromatic ring)

Diazotization and coupling reaction

The solution of different aryl amines (0.01 mole) dissolved in HCl (6 ml, 50 %) was cooled to 0 – 5°C in an ice – bath. A solution of sodium nitrite (0.01 mole, 0.69 gm) in water (4 ml) previously cooled to 0°C was added over a period of five minutes with stirring and maintaining the temperature at 0-5°C , stirring was continued for an hour, maintaining the same temperature with positive test for nitrous acid on starch iodide paper. Excess of nitrous acid was destroyed by adding required amount of sulphamic acid. The resulting solution was used for coupling reaction.

Aa (0.01mole) was dissolved in glacial acetic acid (30 ml). It was cooled below 5°C in an ice–bath. To this well stirred solution, above mentioned diazonium chloride solution was added drop wise over a period of 10-15 minutes, maintaining the pH 7.5 to 8.0 by simultaneous addition of aqueous sodium acetate. (10 % w/v). The stirring was continued for 3 hours at 0–5°C. To the reaction mixture was pour into ice the coloring material was precipitated. The dye was dried at 70°C. It was crystallized from acetone.

Dye A1: IR (KBr, cm^{-1}) 3282 (N-H str.), 1688 (C=O str.of amide),1509 (-NO_2 str.),1340 (C-N str. of tertiary amine), 1263 (C-N str. of secondary amine); ^{1}H NMR (399.76 MHz, DMSO),δ,ppm: 4.442 (S,2H,-CO-CH_2),8.369 (S, 2H, two –NH group), 8.761 (1H,dd, Ha),8.548 (1H,dd, Hb,), 8.217 (1H,dd, Hc), 8.195-7.070 (10H,m, aromatic ring)

Dye A2: IR (KBr, cm^{-1}) 3285 (N-H str.), 1692 (C=O str.of amide),1511 (-NO_2 str.),1342(C-N str. of tertiary amine), 1268 (C-N str. of secondary amine); ^1H NMR (399.76 MHz, DMSO),δ,ppm: 4.331 (S,2H,-CO-CH_2),8.402 (S, 2H, two –NH group), 8.764 (1H,dd, Ha),8.375 (1H,dd, Hb,), 8.021 (1H,dd, Hc), 8.010-7.450 (9H,m, aromatic ring)

Dye A3: IR (KBr, cm^{-1}) 3291 (N-H str.), 1695 (C=O str.of amide),1515 (-NO_2 str.),1343 (C-N str. of tertiary amine), 1258 (C-N str. of secondary amine); ^1H NMR (399.76 MHz, DMSO),δ,ppm: 4.424 (S,2H,-CO-CH_2),8.445 (S, 2H, two –NH group), 8.766 (1H,dd, Ha),8.376 (1H,dd, Hb,), 8.020 (1H,dd, Hc), 8.018-7.50 (9H,m, aromatic ring)

Dye A4: IR (KBr, cm^{-1}) 3285 (N-H str.), 1693 (C=O str.of amide),1514 (-NO_2 str.),1348 (C-N str. of tertiary amine), 1262 (C-N str. of secondary amine); ^1H NMR (399.76 MHz, DMSO),δ,ppm: 4.440 (S,2H,-CO-CH_2),8.420 (S, 2H, two –NH group), 8.768 (1H,dd, Ha),8.377 (1H,dd, Hb,), 8.019 (1H,dd, Hc), 8.766 (1H,dd, a' Between –CN & -NO_2 group), 8.362-7.340 (7H,m, aromatic ring)

Dye A5: IR (KBr, cm^{-1}) 3287 (N-H str.), 1689 (C=O str.of amide),1516 (-NO_2 str.),1344 (C-N str. of tertiary amine), 1260(C-N str. of secondary amine); ^1H NMR (399.76 MHz, DMSO),δ,ppm: 4.220 (S,2H,-CO-CH_2),8.388 (S, 2H, two –NH group), 8.766 (1H,dd, Ha),8.370 (1H,dd, Hb,), 8.017 (1H,dd, Hc), 7.710-7.045 (9H,m, aromatic ring)

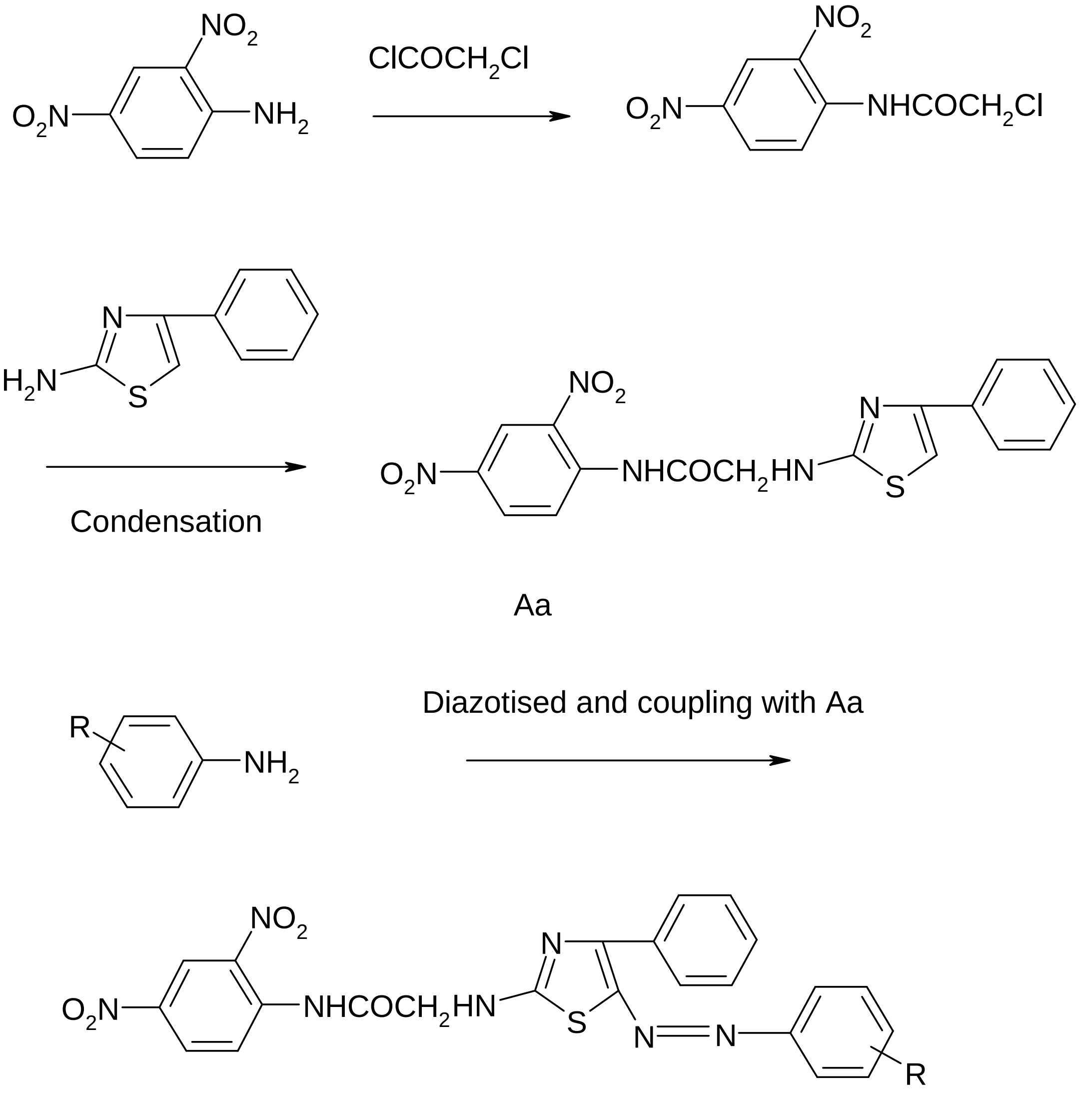
Dye A6: IR (KBr, cm^{-1}) 3295 (N-H str.), 1684 (C=O str.of amide),1510 (-NO_2 str.),1345 (C-N str. of tertiary amine), 1267 (C-N str. of secondary amine); ^1H NMR (399.76 MHz, DMSO),δ,ppm: 4.223 (S,2H,-CO-CH_2),8.390 (S, 2H, two –NH group), 8.765 (1H,dd, Ha), 7.948 (1H,dd, Hb,), 8.021 (1H,dd, Hc), 7.650-6.930 (9H,m, aromatic ring)

Dye A7: IR (KBr, cm^{-1}) 3290 (N-H str.), 1688 (C=O str.of amide),1515 (-NO_2 str.),1340 (C-N str. of tertiary amine), 1266 (C-N str. of secondary amine); ^1H NMR (399.76 MHz, DMSO),δ,ppm: 4.220 (S,2H,-CO-CH_2),8.423 (S, 2H, two –NH group), 8.770 (1H,dd, Ha),8.376 (1H,dd, Hb,), 8.018 (1H,dd, Hc), 7.596-7.063 (9H,m, aromatic ring)

Dye A8: IR (KBr, cm^{-1}) 3288 (N-H str.), 1692 (C=O str.of amide),1512 (-NO_2 str.),1344 (C-N str. of tertiary amine), 1266 (C-N str. of secondary amine); ^1H NMR (399.76 MHz, DMSO),δ,ppm: 2.114(3H,S,-CH_3 group) 4.221 (S,2H,-CO-CH_2),8.240 (S, 2H, two –NH group), 8.760 (1H,dd, Ha),8.376 (1H,dd, Hb,), 8.022 (1H,dd, Hc), 7.630-7.210 (9H,m, aromatic ring)

Dye A9: IR (KBr, cm^{-1}) 3293 (N-H str.), 1690 (C=O str.of amide),1514 (-NO_2 str.),1334 (C-N str. of tertiary amine), 1259 (C-N str. of secondary amine); ^1H NMR (399.76 MHz, DMSO),δ,ppm: 2.118 (3H,S,-CH_3 group) 4.230 (S,2H,-CO-CH_2),8.285 (S, 2H, two –NH group), 8.766 (1H,dd, Ha),8.025 (1H,dd, Hb,), 7.950 (1H,dd, Hc), 7.632-7.142 (9H,m, aromatic ring)

Dye A10: IR (KBr, cm^{-1}) 3301 (N-H str.), 1693 (C=O str.of amide),1517 (-NO_2 str.),1341 (C-N str. of tertiary amine), 1260 (C-N str. of secondary amine); ^1H NMR (399.76 MHz, DMSO),δ,ppm: 4.223 (S,2H,-CO-CH_2),8.480 (S, 2H, two –NH group), 8.766 (1H,dd, Ha),8.376 (1H,dd, Hb,), 8.022 (1H,dd, Hc), 7.620-7.220 (9H,m, aromatic ring)



Here,
R= H,-OH,-CH_3,-NO_2, -Cl, -Br

Dye A11: IR (KBr, cm^{-1}) 3288 (N-H str.), 1678 (C=O str.of amide),1515 (-NO_2 str.),1340 (C-N str. of tertiary amine), 1253 (C-N str. of secondary amine); ^{1}H NMR (399.76 MHz, DMSO),δ,ppm: 4.410 (S,2H,-CO-CH_2),8.330 (S, 2H, two –NH group), 8.768 (1H,dd, Ha), 8.375 (1H,dd, Hb,), 8.021 (1H,dd, Hc), 8.778 (1H,dd, a1), 8.512 (1H,dd, b1), 7.782-7.392 (6H,m, aromatic ring)

Dye A12: IR (KBr, cm^{-1}) 3280 (N-H str.), 1683 (C=O str.of amide),1512 (-NO_2 str.),1344 (C-N str. of tertiary amine), 1259 (C-N str. of secondary amine); ^{1}H NMR (399.76 MHz, DMSO),δ,ppm: 4.464 (S,2H,-CO-CH_2),8.332 (S, 2H, two –NH group), 8.766 (1H,dd, Ha), 7.940 (1H,dd, Hb,), 8.020 (1H,dd, Hc), 8.615 (1H,dd, a1), 8.530 (1H,dd, b1), 7.990-7.730 (5H,m, aromatic ring)

Dye A13: IR (KBr, cm^{-1}) 3283 (N-H str.), 1681 (C=O str.of amide),1510 (-NO_2 str.),1346(C-N str. of tertiary amine), 1258 (C-N str. of secondary amine); ^{1}H NMR (399.76 MHz, DMSO),δ,ppm: 4.318 (S,2H,-CO-CH_2),8.412 (S, 2H, two –NH group), 8.767 (1H,dd, Ha), 8.472 (1H,dd, Hb,), 8.024 (1H,dd, Hc), 8.778 (1H,dd, a1), 8.512 (1H,dd, b1), 7.964-7.580 (7H,m, aromatic ring)

Table-1: Yield, λmax, Melting points and Nitrogen analysis of disperse dyes

Dye No.	R	Yield (%)	λmax (nm)	M.P. °C	Nitrogen (%) Found	Nitrogen (%) Calcd.
A1	H-	79	472	152	19.45	19.47
A2	4-NO_2-	75	493	148	20.45	20.43
A3	3-NO_2-	68	463	110	20.48	20.43
A4	2-CN,4-NO_2-	66	408	180	21.95	21.98
A5	2-OH-	70	414	116	18.90	18.87
A6	3-OH-	73	413	123	18.83	18.87
A7	4-OH-	72	415	117	18.84	18.87
A8	4-CH_3 -	78	479	136	19.0	18.95
A9	3-CH_3-	70	473	140	18.94	18.95
A10	4-Cl-	69	464	129	18.26	18.23
A11	2,4-$(NO_2)_2$-	64	432	141	21.26	21.24
A12	2,4-$(NO_2)_2$,6-Cl-	58	492	166	20.10	20.08
A13	2,6-$(Br)_2$,4-NO_2-	62	441	179	15.82	15.87

Table-2: Shade, Fastness properties, K/S value, R value and % Exhaustion of the dyes

Dye No.	Shade on Polyester	Fastness to Light	Fastness to Washing	Sublimation at 180°C Staining of Cotton	Sublimation at 180°C Staining of Polyester	K/S Value	R Value	% Exhaustion
A1	Light Orange	4	4-5	4	4	5.69	7.82	76.10
A2	Red	4-5	3	5	4-5	7.20	5.98	81.50
A3	Dull yellow	4-5	5	4	5	5.17	8.70	79.42
A4	Cream	4	4	3-4	4	8.94	4.64	71.70
A5	Cream	4-5	5	5	4-5	5.73	7.75	68.75
A6	Greenish Cream	5	4	4-5	4	8.31	5.06	74.30
A7	Light Yellow	4	4	4	4	5.69	7.82	76.20
A8	Dark Yellow	3-4	4	3-4	4	3.86	11.97	71.45
A9	Reddish yellow	4	4	3-4	4	5.28	8.49	71.50
A10	Reddish yellow	4	5	4	4-5	4.20	10.93	77.34
A11	Light yellow	5	4-5	5	5	5.18	8.68	80.05
A12	Yellowish orange	4-5	4-5	5	5	7.91	5.36	79.30
A13	Yellow	4	3-4	4	4	6.37	6.88	70.05

Table-3: Antimicrobial screening results of disperse dyes.

Dye Compound	R	Zone of inhibition (mm)			
		Antibacterial activity			Antifungal activity
		E.coli	*P.aeruginosa*	*S.aureus*	*C.albicans*
A1	H-	14	14	15	12
A2	4-NO_2-	14	11	12	14
A3	3-NO_2-	12	13	16	13
A4	2-CN,4-NO_2-	13	15	13	15
A5	2-OH-	15	15	16	11
A6	3-OH-	10	13	14	10
A7	4-OH-	13	14	16	13
A8	4-CH_3-	11	13	15	11
A9	3-CH_3-	10	14	13	14
A10	4-Cl-	14	15	14	10
A11	2,4-$(NO_2)_2$-	12	11	12	15
A12	2,4-$(NO_2)_2$,6-Cl-	14	15	17	11
A13	2,6-$(Br)_2$,4-NO_2-	14	14	15	11
Metranidazole		15	16	21	-
Amplicilline		15	15	13	-
Flucanazole		-	-	-	19

RESULTS AND DISCUSSION

The characterization data for the dyes (A1-A13) are given in table-1. Table-2 shows moderate to fairly good light fastness. However, compounds A3, A5, A12 and A13 show some what better light fastness. The wash-fastness of all the compounds, as may be anticipated on the basis of their increased molecular weight and polarity to less substituted analogues, was also of an acceptably very good order. Introducing the terminal amino group for better dispersibility observes no notable change in the percentage exhaustion. Overall, the prepared dyes gave generally good dyeing on polyester fibers. All the samples showed moderate activities against E.coli and P. aeruginosa. All dyes showed good antibacterial activity against above bacteria, while A4 and A11 showed good anti fungal activity against C.Albicans. Standard drugs like Metranidazole, Amplicilline and Flucanazole were used for the comparison purpose are shown in table-3.

CONCLUSION

The present study revealed that prepared thiazole derivatives showed wide range of shades. They showed good dyeing performance on polyester fiber and some of the compounds showed good antibacterial and antifungal activities.

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