Synthesis of monoazo disperse dyes, their dyeing performance on polyester fibers and antimicrobial activity

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Abstract: Different monoazo disperse dyes N-(4-nitrophenyl)-2-((4-(3'-nitro phenyl)-1, 3-thiazol-2-yl) amino)acetamide derivatives has been synthesized in the current study using different diazotized aryl amines. These were characterized using elemental analysis, ¹H NMR and IR spectra. Their dyeing performance on polyester fiber was assessed and fastness properties of these dyes were evaluated by applying them to polyester fiber. These dyes showed moderate anti-bacterial and anti-fungal activities.

Keywords: 2-Amino-4-(3'-nitro phenyl) thiazole, chloroacetylchloride, 4-nitro aniline, dyeing performance, ¹H NMR, IR Spectra, antimicrobial activity.

I. Introduction

Disperse dyes are organic colorants and have less water solubility which are applied in colloidal dispersions to hydrophobic textile fibers to produce desired colour. There has been significant increase in the development of disperse dyes due to increasing use of polyester fibers and their blend [1-3], 2-amino thiazole derivatives [4-8] have been utilized as heterocyclic components since long for many disperse dyes. The synthesis of thiazole, 2-amino-4-phenyl thiazole and their derivatives has got significant importance because a large number of natural products and drugs comprise of this heterocyclic moiety [9-11]. It was our main objective to synthesize monoazo disperse dyes based on 2-chloro-N-(4-nitrophenyl)acetamide [12,13]. The characteristic data of synthesized dyes and dyeing properties also studied [14-18]. The newly synthesized monoazo disperse dyes sample were screened for antibacterial activity against Staphylococcus aureus, Escherichia coli, Bacillus subtilis and Pseudomonas aeruginosa and antifungal activity against candida albicans [19-24].

II. Materials and Methods

All reagents were of analytical reagent grade and were used without further purification. All the synthesized compounds were characterized by their spectral analysis. The melting points of the compounds were determined using Stuart smp 10 melting point apparatus and are uncorrected. The purity of dyes was determined by thin- layer chromatography (TLC) using silica gel-G coated Al-plates and spots were visualized under uv radiation. IR spectra were recorded on FTIR spectrophotometer Perkin Elmer RX-1 using KBr pellets. ¹H NMR spectra obtained on Bruker Avance-II 400 NMR spectrometer using DMSO solvent and TMS as internal reference (chemical shifts in δ , ppm). Elemental analysis carried on Perkin Elmer (USA) 2400 series instrument. The synthesized dyes were screened for their antimicrobial activity using the Kirby-Bauer method. All the compounds were screened for their in vitro antimicrobial activity against bacterial strains Escherichia coli, Pseudomonas aeruginosa, Staphylococcus aureus, Bacillus subtilis and fungi Candida albicans at 50µg/ml concentration and Ciprofloxacin and Flucanazole using as standards respectively. The fastness to light, wash and sublimation was assessed in accordance with ISO 105. A convenient laboratory method was used for dyeing polyester to employ high temperature (130°C) and high pressure (24-30psi.). The dye bath exhaustion (%E) of the dyed fiber was determined according to the method.

Preparation of 2-amino-4-(3'-Nitro phenyl) thiazole (1)

In 250 ml R.B.F mixture of (16.5 g, 0.1 mole) m-nitro acetophenone and thiourea (15.2 g, 0.2 mole) was added. Then bromine (23.97 g, 0.15 mole) drop wise added, after addition of bromine the reaction mixture was heated in water bath for 8-10 hours. It was cooled and content was washed with water. The residue left was dissolved in boiling water and filtered. The filtrate was made alkaline by addition of liquor ammonia. The solid (1) thus obtained was filtered, dried and recrystalized from ethanol.

Yield 81 %, M.P.: 185-188 °C IR (KBr cm⁻¹⁾ 3444 (N-H str.), 3113 (C-H str.), 1512 (NO₂ str.), 713 (C-S str.), 1339 (C-N str.).

Preparation of 2-chloro-N-(4-nitrophenyl)acetamide(2)

4-nitro aniline (2.76 g, 0.02 mole) and 15 ml glacial acetic acid was added to a 250 ml R.B.F. then cooled to 0-5 °C. Chloroacetyl chloride (3.39ml, 0.02 mole) was slowly added to R.B.F. with continuous stirring. When addition was complete, the reaction mixture was stirred at room temperature for 3 hours. The reaction mixture dumped into 20 % sodium acetate solution. The solid (2) thus obtained was filtered washed with water, dried and recrystalized from 80 % ethanol and 20 % water solution. Yield: 90 %, M.P.: 186-188 °C.

Preparation of N-(4-nitrophenyl)-2-((4-(3'-nitro phenyl)-1, 3-thiazol-2-yl) amino) acetamide (3)

In a 250 ml R.B.F. 2-amino-4-(3'-nitro phenyl) thiazole) (2.21 g, 0.01 mole) (1) and 2-chloro-N-(4-nitrophenyl)acetamide (2.14 g, 0.01 mole) (2) in 30 ml glacial acetic acid was taken, then slowly K_2CO_3 (6.9 g, 0.05 mole) was added into reaction mixture. When addition was completed, the reaction mixture was heated for 6 hours at 85-90 °C. The reaction mixture dumped into 20 % sodium acetate solution. The solid (3) thus obtained was filtered washed with water, dried and recrystalized from ethanol. Yield: 80 %, M.P.: 160-165 °C.

Diazotization and coupling reaction (SP₁-SP₁₅)

Aryl amines (0.01 mole) dissolved in HCl (5 ml, 50 %) was cooled to 0-5 $^{\circ}$ C in an ice-bath. A solution of sodium nitrite (0.01 mole, 0.69 g) in water (5 ml) previously cooled to 0-5 $^{\circ}$ C was added over a period of ten minutes with continuous stirring and maintaining the temperature at 0-5 $^{\circ}$ C, further the reaction mixture was stirred for an hour keeping constant positive test on starch iodide paper. The excess of nitrous acid was then destroyed using sulfamic acid. The resulting solution was used for coupling reaction.

N-(4-nitrophenyl)-2-((4-(3'-nitro phenyl)-1, 3-thiazol-2-yl) amino) acetamide (3) (0.01mole) was dissolved in glacial acetic acid (30 ml). Then cooled below 5 $^{\circ}$ C in an ice-bath. The above mentioned diazonium chloride solution was added drop wise over a period of 10-15 minutes, maintaining the pH 7.0 to 7.5 by simultaneous addition of aqueous sodium acetate (10 % w/v), further the reaction mixture was stirred for 3 hours at 0-5 $^{\circ}$ C. The solid dye was precipitated. The dye was filtered, washed with water to remove acid completely and dried and recrystalized from acetone.

III. Results And Discussion

Spectral Properties of Dyes

The visible absorption spectrum of dyes was recorded in DMF and their properties are shown in (Table 1). From (Table 1), it is clear that the value of λ_{max} depends on the nature and position of coupling component used.

IR spectra of synthesized dyes described in (Table 3), compound (3) showed N-H stretching vibration at 3417 cm⁻¹, C-H stretching vibration at 3115 cm⁻¹, C=O stretching vibration at 1649 cm⁻¹, N=N stretching vibration at 1575, C-S-C stretching vibration at 1471 cm⁻¹, C-S stretching in thiazole at 750 cm⁻¹, 1518 cm⁻¹ is due to presence of NO₂ group. The ¹H NMR of all dyes showed in (Table 3). The ¹H NMR spectra of all dyes were recorded in DMSO. The compound (2) showed spectra exhibited multiplets in the region at δ 7.06-8.75 ppm for 12 aromatic protons. The Methylene attached to carbonyl group showed singlet at δ 3.40 ppm. While presence of –NH in thiazole and aromatic ring showed singlet at δ 4.24 ppm and δ 7.00 ppm, respectively.

Dyeing Properties

Dyeing method

All the synthesized dyes SP_1 - SP_{15} were applied on polyester fabric in 2% depth by HTHP method. High temperature (130 °C) and high pressure is a convenient method for dyeing polyester fibers in the laboratory. Glycerin-bath high temperature beaker dyeing machine was used. 10 ml DMF was added and dispersion of the dye was produced by dissolving the appropriate amount of dye (2% depth) and then added dropwise with continuous stirring to the dyebath (liquor ratio 20:1) containing 1% Setamol WS (BASF) as anionic dispersing agent. The pH of dye solution was adjusted to 4 to 5 using aqueous acetic acid. Already wetted-out polyester fabric pieces were added. Dyeing was run by rising the dyebath temperature upto 130 °C at a rate of 2 °C /min, maintaining at this temperature for 1 hour, under (24-30 psi) pressure. After completion of process cooling upto 50 °C was done, the dyed fabric pieces were rinsed with cold water and reduction was cleared properly (1 g/l sodium hydroxide, 1 g/l sodium hydrosulphite, 10 minutes and 80 °C). The dyed sample was rinsed with hot and cold water and finally the dyeing air- dried.

Fastness Properties

Light Fastness

The light fastness study was carried out using a xenon light fastness tester having a xenon arc lamp. The dyed sample was assessed by comparing change in colour of specimen with standard dye patterns of specific rating (1 to 8). The higher the rating, the better is the light fastness. The results are given in (Table 2).

Wash Fastness

Fastness to washing was assessed using following method. The washing solution contained soap 5 g/l and anhydrous sodium carbonate 2 g/l, washing temperature was 95 °C, the duration of washing was 30 min, and the material to liquor ratio was 5:1. After washing, the specimen was removed and rinsed with hot water (35 °C) until the rinsed water showed no alkalinity. It was dried in the air. The effect of the change in colour was expressed and defined by reference to grey scale. The results are given in (Table 2).

Sublimation Fastness

The dyed fabric was stitched to undyed cotton fabric on one side and undyed polyester fabric on the other side. The composite dyeing specimens were placed in a heating device (sublimation tester) electrical heating system and kept there for 30 s at 180 °C. The dried fabric was removed and kept in air for 4 hours. The effect of dyed fabrics on the cotton and polyester fabrics was measured and assessed with grey scales. The results are given in (Table 2).

As shown in (Table 2), the light fastness of each dye is rated as 3-5 for polyester, which shows light fastness is moderate to good. The wash fastness of each dye is rated as 3-5 for polyester showing wash fastness is good to excellent. The sublimation fastness of each dye is rated as 3-5 for polyester, which also shows rubbing fastness is good to excellent.

Antimicrobial Activity

Antibacterial and antifungal activities of synthesized compounds were examined in vitro by Kirby-Bauer disc diffusion method. The zone of inhibition (mm) was determined using disc diffusion method according to the standard procedure at concentrations 50 μ g/ml. All the compounds were tested for activity against Staphylococcus aureus, Escherichia coli, Bacillus subtilis, Pseudomonas aeruginosa and Candida albicans. Ciprofloxacin and Flucanazole were employed as the standard (Table 4).

IV. Conclusion

All the synthesized monoazo disperse dyes based on 2-chloro-N-(4-nitrophenyl)acetamide synthesized by conventional methods and their different properties were studied applying them to polyester fabric. The dyes showed good dyeing performance on polyester fibers. The dyes gave generally yellow, orange, brown, red, and cream shades. Table 2 shows moderate to fairly good light fastness. Dyes SP₁, SP₄, SP₅, SP₇, SP₉, SP₁₂, and SP₁₅ showed better light fastness. The wash-fastness of all the dyes was also of very good order. All the samples showed moderate to fairly good antibacterial activity against Escherichia coli, Pseudomonas aeruginosa, Staphylococcus aureus and Bacillus subtilis. All the samples showed moderate to fairly good antifungal activity against Candida albicans. Standard drugs Ciprofloxacin and Flucanazole were used for the comparison purpose. The results are shown in (Table 4).

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Reaction scheme:



Where, R=H, NO₂, CH₃, Cl, F, OCH₃, OH, NHCOCH₃.

Table 1- Physical properties of SP ₁ -SP ₁₅ Dyes									
							Nitroge	en (%)	
Dye	R	Molecular	Molecular	Yield	M.P.	λ_{max}	Found	Calcd.	
No.		formula	weight	%	C	(nm)			
			g/mole						
SP_1	H-	C23H17N7O5S	503	80	146	466	19.35	19.43	
SP ₂	3-NO ₂ -	$C_{23}H_{16}N_8O_7S$	548	79	238	490	19.48	20.43	
SP ₃	4-NO ₂ -	$C_{23}H_{16}N_8O_7S$	548	81	250	508	20.52	20.43	
SP_4	3-CH ₃ -	$C_{24}H_{19}N_7O_5S$	517	74	146	450	18.90	18.95	
SP5	4-CH ₃	C24H19N7O5S	517	76	140	460	18.82	18.95	
SP_6	3-C1-	C23H16ClN7O5S	537	80	188	466	18.34	18.23	
SP ₇	4-Cl-	C23H16CIN7O5S	537	75	172	467	18.12	18.23	
SP ₈	4-F-	$C_{23}H_{16}FN_7O_5S$	521	78	144	454	18.76	18.80	
SP ₉	3-OCH ₃ -	C24H19N7O6S	533	75	150	450	18.46	18.38	
SP_{10}	4-OCH ₃ -	C24H19N7O6S	533	68	154	440	18.30	18.38	
SP11	2,4-(NO ₂) ₂ -	$C_{23}H_{15}N_9O_9S$	593	80	128	533	21.15	21.24	
SP ₁₂	2-OH-	C23H17N7O6S	519	77	154	450	18.80	18.87	
SP13	2-OH,4-NO ₂ -	C23H16N8O8S	564	65	140	460	19.76	19.85	
SP_{14}	2,6-(Cl) ₂ ,4-NO ₂ -	$C_{23}H_{14}Cl_2N_8O_7S$	617	79	142	490	18.27	18.15	
SP ₁₅	4-NHCOCH ₃ -	$C_{25}H_{20}N_8O_6S$	560	63	146	476	20.15	19.99	

Table 1- Physical properties of SP₁-SP₁₅ Dyes

Table 2- Fastness properties of SP₁-SP₁₅ Dyes

Dye Shade		Fastness to		Sublimation fastness at 180 ⁻ C		Exhaustion %	Fixation %	K/S Value	R Value
No.	on Polyester	Light	Washing	Staining of cotton	Staining of Polyester				
SP_1	Yellow	5	4	4	4	71.50	77.5	18.54	9.52
SP ₂	Light yellow	4	5	4	5	75.64	90.1	3.52	27.2
SP ₃	Reddish orange	4	4	4-5	4	72.65	85.5	4.89	23.5
SP_4	Reddish yellow	5	5	4-5	5	77.50	87.1	17.46	10.1
SP ₅	Turmeric yellow	5	4-5	4	4-5	79.80	92.3	21.53	8.5
SP ₆	Yellow	4	5	5	5	74.55	89.2	20.28	8.67
SP ₇	Dark yellow	5	4-5	4-5	5	80.20	88.3	17.95	10.2
SP ₈	Turmeric yellow	4	4	4-5	4	78.70	92.5	14.93	13.5
SP ₉	Light yellow	5	4-5	4	4-5	76.20	91.2	11.55	18.35
SP10	Cream	3-4	4	3-4	4	74.80	89.75	1.38	29.25
SP11	Maroon	4-5	5	4-5	5	77.90	91.70	11.45	13.3
SP12	Cream	5	5	5	4-5	75.75	90.4	1.42	29.02
SP13	Pinkish Orange	4-5	4	4-5	3-4	78.20	92.3	4.43	23.9
SP ₁₄	Reddish pink	4	4-5	4	3	79.33	93.7	3.70	26.24
SP15	Brown	5	5	4-5	4-5	76.65	91.5	3.65	26.80

Light fastness: 1- poor, 2- slight, 3- moderate, 4- fair, 5- good, 6- very good, 7- excellent Wash and sublimation fastness: 1- poor, 2- fair, 3- good, 4- very good, 5- excellent

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Table 3- IR and	¹ H NMR	data of	SP ₁ -SP ₁₅	Dyes

Dye No	IR (KBr) cm ⁻¹	¹ H NMR (DMSO) Chemical shift in \Box ppm
110.	3420 (N-H str.), 3118 (C-H), 1640 (C=O), 1576	3.34 (S. 2HCOCH ₂), 4.20 (S. 1HNH),
SP_1	(N=N), 1520 (NO ₂), 1468 (C-S-C), 760 (C-S)	6.98 (S, 1H, -NH), 7.00-8.70 (M, 13H, Ar-H)
	3410 (N-H), 3110(C-H), 1645 (C=O), 1580 (N=N),	3.40 (S, 2H, -COCH ₂), 4.24 (S, 1H, -NH),
SP ₂	1524 (NO ₂), 1475 (C-S-C), 755 (C-S)	7.00 (S, 1H, -NH), 7.06-8.75 (M, 12H, Ar-H)
	3417 (N-H), 3115 (C-H), 1649 (C=O), 1575 (N=N),	3.38 (S, 2H, -COCH ₂), 4.22 (S, 1H, -NH),
SP ₃	1518 (NO ₂), 1471 (C-S-C), 750 (C-S)	7.10 (S, 1H, -NH), 7.12-8.72 (M, 12H, Ar-H)
	3417 (N-H), 3015 (CH ₃),3115 (C-H), 1651 (C=O),	2.30 (S, 3H, -CH ₃), 3.32 (S, 2H, -COCH ₂),
SP_4	1575 (N=N), 1518 (NO ₂), 1480 (C-S-C), 765 (C-S)	4.20 (S, 1H, -NH), 7.04 (S, 1H, -NH), 7.08-8.70 (M, 12H,
		Ar-H)
	3412 (N-H), 3010 (CH ₃),3113 (C-H), 1655 (C=O),	2.32 (S, 3H, -CH ₃), 3.34 (S, 2H, -COCH ₂),
SP ₅	1570 (N=N), 1518 (NO ₂), 1477 (C-S-C), 760 (C-S)	4.22 (S, 1H, -NH), 7.06 (S, 1H, -NH), 7.10-8.79 (M, 12H,
		Ar-H)
	3416 (N-H), 3113 (C-H), 1658 (C=O), 1578 (N=N),	3.32 (S, 2H, -COCH ₂), 4.26 (S, 1H, -NH),
SP_6	1520 (NO ₂), 1482 (C-S-C), 753 (C-S), 728(C-Cl)	6.9 (S, 1H, -NH), 6.94-8.95 (M, 12H, Ar-H)
	3418 (N-H), 3117 (C-H), 1669 (C=O), 1574 (N=N),	3.36 (S, 2H, -COCH ₂), 4.30 (S, 1H, -NH),
SP ₇	1522 (NO ₂), 1474 (C-S-C), 756 (C-S), 730(C-Cl)	6.96 (S, 1H, -NH), 7.00-8.98 (M, 12H, Ar-H)
	3412 (N-H), 3120 (C-H), 1667 (C=O), 1570 (N=N),	3.30 (S, 2H, -COCH ₂), 4.25 (S, 1H, -NH),
SP ₈	1527 (NO ₂), 1471 (C-S-C), 750 (C-S), 1120 (C-F)	6.98 (S, 1H, -NH), 7.08-8.86 (M, 12H, Ar-H)
	3414 (N-H), 3112 (C-H), 1660 (C=O), 1568 (N=N),	2.36 (S, 3H, -CH ₃), 3.32 (S, 2H, -COCH ₂),

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SP ₉	1530 (NO ₂), 1478 (C-S-C), 755 (C-S),	4.28 (S, 1H, -NH), 7.08 (S, 1H, -NH), 7.10-8.78 (M, 12H,
	1160 (C-OCH ₃)	Ar-H)
	3419 (N-H), 3108 (C-H), 1670 (C=O), 1576 (N=N),	2.40 (S, 3H, -CH ₃), 3.36 (S, 2H, -COCH ₂),
SP_{10}	1532 (NO ₂), 1473 (C-S-C), 759 (C-S), 1150 (C-	4.30 (S, 1H, -NH), 7.10 (S, 1H, -NH), 7.14-8.85 (M, 12H,
	OCH ₃)	Ar-H)
	3412 (N-H), 3116 (C-H), 1644 (C=O), 1572 (N=N),	3.36 (S, 2H, -COCH ₂), 4.28 (S, 1H, -NH),
SP11	1516 (NO ₂), 1460 (C-S-C), 754 (C-S)	7.15 (S, 1H, -NH), 7.2-9.0 (M, 11H, Ar-H)
	3414 (N-H), 3050(О-H), 3110 (С-H), 1642 (С=О),	3.32 (S, 2H, -COCH ₂), 4.26 (S, 1H, -NH),
SP_{12}	1566 (N=N), 1512 (NO ₂), 1456 (C-S-C), 750 (C-S)	7.04 (S, 1H, -NH), 5.6 (S, 1H, -OH), 7.08-8.92
		(M, 12H, Ar-H)
	3410 (N-H), 3054(O-H), 3118 (C-H), 1640 (C=O),	3.34 (S, 2H, -COCH ₂), 4.28 (S, 1H, -NH),
SP_{13}	1562 (N=N), 1514 (NO ₂), 1452 (C-S-C), 752 (C-S)	7.06 (S, 1H, -NH), 5.64 (S, 1H, -OH), 7.20-8.96 (M, 11H,
		Ar-H)
	3408 (N-H), 3116 (C-H), 1660 (C=O), 1578 (N=N),	3.38 (S, 2H, -COCH ₂), 4.22 (S, 1H, -NH),
SP_{14}	1512 (NO ₂), 1472 (C-S-C), 764 (C-S), 726(C-Cl)	7.04 (S, 1H, -NH), 7.10-8.94 (M, 10H, Ar-H)
	3412 (N-H), 3114 (C-H), 1662 (C=O), 1580 (N=N),	2.2 (S, 3H, -CH ₃), 3.30 (S, 2H, -COCH ₂),
SP15	1514 (NO ₂), 1477 (C-S-C), 760 (C-S), 1683 (C-	4.22 (S, 1H, -NH), 7.12 (S, 2H, -NH), 7.10-8.78 (M, 12H,
	NHCOCH ₃)	Ar-H)

Table 4- Antimicrobial screening results of monoazo disperse dyes.

	Zone of inhibition (mm)						
Dye No.		Antifungal activity					
	E. coli	P. aeruginosa	S. aureus	B. subtilis	C. albicans		
SP_1	11	11	10	12	10		
SP_2	10	12	13	14	12		
SP ₃	14	10	19	20	12		
SP_4	9	10	14	15	11		
SP ₅	10	11	10	14	10		
SP_6	12	12	14	16	12		
SP ₇	11	9	12	12	10		
SP_8	13	11	18	17	11		
SP ₉	10	10	11	14	10		
SP_{10}	9	9	12	11	10		
SP_{11}	10	11	13	15	11		
SP_{12}	11	10	13	14	10		
SP_{13}	9	10	15	11	10		
SP_{14}	10	11	14	16	12		
SP15	12	9	12	17	11		
Ciprofloxacin	19	18	24	28	-		
Flucanazole	-	-	-	-	19		