STUDIES ON SYNTHESIS CHARACTERIZATION OF SOME AZO DYES FOR DYEING POLYESTER FABRIC PART 2

[1]ANKITKUMAR D. PANCHAL, [2]HITESHKUMAR J. PATEL

Department of Chemistry, Shri R.K. Parikh Arts and Science College Petlad, Dist.-Anand, Gujarat (India)

Abstract: A series of new heteroaryl azo dyes were synthesized by coupling of diazonium salts of various phenyl amine with newly synthesized imidazole - thiazole combined compounds. Namely N-((1H-benzo[d]imidazol-2-yl)methyl)-4-(4-bromophenyl)thiazol-2-amine. It was prepared by simple condensation reaction of 2-chloromethyl benzamidiazol with 4-(4-bromophenyl)-1,3-thiazol-2-amine. It was characterized duly various aromatic amines were diazotized and the resultant diazonium salts were coupled with above compound. The so-called result dyes N-(1H-benzimidazol-2-ylmethyl)-4-(4-bromophenyl)-5-[(E)-phenyldiazenyl]-1,3-thiazol-2-amine (Dye-A) obtained were characterized by elemental content, azo group determination and spectral studies. The dyeing performance of these dyes where assessed on polyester and nylon fabric. The dyes patterns were of various shades of red with good depth, brightness and leveling properties. The dyed fabric showed fairly good to very good light fastness and very good to excellent fastness to washing, perspiration and rubbing. The dye bath exhaustion and fixation on the polyester fabric was found to be very good.

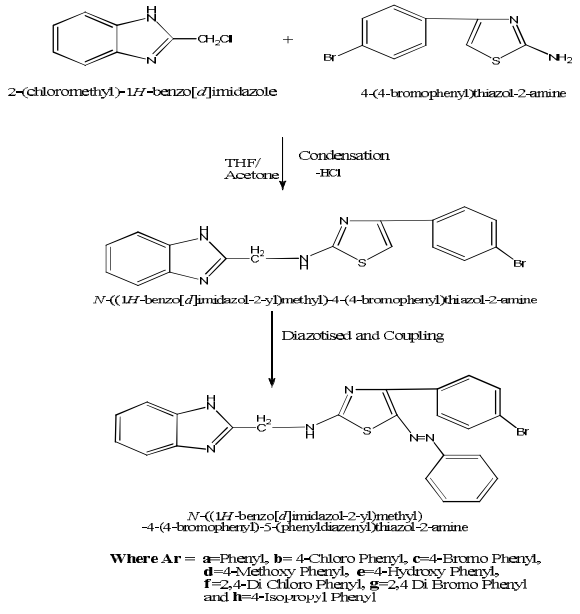
Keywords: 4-(4-bromophenyl)-1,3-thiazol-2-amine, Benzimidazole, Dyeing, Polyester and nylon.

I. INTRODUCTION

Azo dyes with thiazol components have been invented to produce shades ranging from red to greenish blue on synthetic fibre Colour Index described various dyes wherein thiazole nucleus occurs [1].

In this regard, azo dyes based on heterocyclic nucleus have been developed, and the resultant dyes have higher tinctorial strength and give brighter dyeings than those derived from simple diazo components. e.g amino-thiazoles, isothiazole, thiophene, and pyrazole compounds afford a pronounced bathochromic effect compared to benzenoid derivatives [2–9]. Hence the present author thought to study containingImidazol-thiazol merged molecule. In continues of these work [10,11], so, the present paper comprises the study of novel heteroaryl azo dyes scanned in the scheme.

II. REACTION SCHEME



III. MATERIAL AND METHOD MATERIAL

All the required reagents and solvents were of the commercial quality and purchased from local dealers.

General Information

4-(4-nitrophenyl)thiazol-2-amine and 2-(chloromethyl)-1-H benzo[d]imididazole were prepared by methods reported [12,13]. All other chemical used of pure grade. Melting points were determined by a Kofler apparatusand UV-visible spectroscopy. Infrared spectra were recorded using Shimadzu (8400) FT-IR Spectrometer.

Synthesis of imidazole - thiazole combined compound

This was prepared by refluxing the mixture of 2-(chloromethyl)-1-Hbenzo[d] imididazole (0.9gm 0.006mole) with 4-(4-bromophenyl)thiazol-2-amine (1.056gm 0.006 mole) in the presence of K_2CO_3 (1 mole) for 5 hour. The resultant product was washed by water-ethanol (50:50) mixture and finally by ethanol as air-dried. The product was dissolved in DMF and reprecipitate by solvent ether. The product is named as *N-((1H-benzo[d]imidazol-2-yl) methyl)-4-(4-bromophenyl) thiazol-2-amine.*

Coupling of diazotited arylamine with N-((1H-benzo[d]imidazol-2-yl) methyl)-4-(4-nitrophenyl) thiazol-2-amine combined compound

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 (4ml) 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.

N-((1H-benzo[d]imidazol-2-yl) methyl)-4-(4-bromophenyl) thiazol-2-amine. (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.

The physical properties of the synthesized dyes are shown in table 1 and IR spectra are recorded in table 2.

Dyeing of Polyester

The dye baths were prepared from the dye (2% O.W.F) with a dispersol-levelling agent (1gm/liter) and 5% toluene as carrier to a final liquor of 50:1, w/w. The pH value of the bath was adjusted to 4-5 with acetic acid (10%). The polyester fabrics, previously wetted, were placed into the liquor at 25°C-30°C. The temperature was raised to 100°C at the rate of 2°C/min, and dyeing continued for 60 min. After cooling, the dyed fabrics were reduction cleared in sodium hydroxide (2gm/liter), dispersing agent (1.5gm/liter) and sodium dithionite (2gm/liter) at 60°C for 30mins and then washed and dried. Percentage exhaustion was calculated to determine the dye absorption unto the fabric [14].

Dyeing of Nylon

The procedure carried out for dyeing of the polyester fabric was repeated here but this time without a carrier.

Fastness Tests

The various fastness tests such as washing, light, perspiration and rubbing was carried out. The wash fastness test was carried out using the ISO number 3 ethod, the light and perspiration fastness was assessed in accordance with BS:10061978 while the rubbing fastness test was carried out using crock meter (Atlas) in accordance with AATCC-1961. The %exhaustion and %fixation was carried out according to known method [15].

Table-1: Physical properties of synthesized dyes

No.	Molecular weight (gm)	Melting point °C	% yield	Colour on Polyester	Colour on Nylon
A	489	195	69	Reddish maroon	Dark Brown
B	523	201	68	Dark Orang	Reddish Brown
C	568	196	65	Dark Orang	Brown
D	519	204	68	Light Red	Dark Red
E	505	198	67	Red	Light Red
F	558	212	64	Light Red	Light Orange
G	647	221	62	Light Orange	Light Red
H	521	206	67	Orange	Red

Infrared spectra of the Dyes

Table-2: IR Spectra features of synthesized Dyes cm^{-1}

Dyes NO.	Aromatic C-H Stretch	NH stretch	C=C	N=N	-C-N Stretch	N=C=S	-C-Br
A	2925.23	3445.45	1617.27	1454.41	1215.11	2236.10	1058.5
B	2948.47	3436.14	1624.23	1457.13	1215.21	2245.44	1058.54
C	2931.34	3435.37	1629.23	1455.22	1218.12	2242.15	1052.31
D	2938.43	3465.23	1622.16	1457.14	1217.17	2245.17	1059.61
E	2949.16	3447.15	1632.21	1457.25	1219.17	2242.74	1055.25
F	2935.14	3435.29	1627.51	1451.24	1215.37	2241.91	1050.41
G	2838.36	3457.29	1621.11	1447.42	1218.16	2256.24	1054.32
H	2947.12	3443.15	1626.25	1452.21	1211.14	2236.14	1057.41

Visible Absorption spectra of Dyes

The visible absorption spectra of all the synthesized compounds were recorded in different solvent system and their data are shown in Table 3. The solvatochromic properties of these dyes were systematically investigated in different solvents with different polarities [16-19].

Table-3: Visible Absorption spectral band of Synthesized Dye

Dyes No	emaxin acetone $mole^{-}cm1\times 10^{4}$	Acetone λmax(nm) a	DMF λmax (nm) b	Ethanol λmax(nm)c	Ethanol+HCl λmax(nm)d	Change in λmax(nm) d-c
A	2.362	513	513	502	512	+10
B	2.244	524	518	516	521	+5
C	2.238	511	517	494	501	+7
D	1.651	412	407	412	412	+0
E	1.323	417	409	413	423	+10
F	2.422	617	594	537	552	+15
G	2.775	607	616	613	590	-23
H	2.413	567	577	527	532	+5

Dyeing and Fastness properties

The dyes were applied to polyester, nylon and polyester/cotton blend using the methods described above. The dyes gave very good levelness and fiber penetration on polyester nylon and polyester/cotton blend. The exhaustion was good up to 95% and fixation values up to 93%. The dyes gave very-good to moderate wash, perspiration and rubbing fastness while the light fastness result was between good to fair. The results show that nylon gave higher exhaustion values than polyester for most of the dyes this can be attributed to the amorphous nature of the nylon fabric and hence easy penetration of the dye molecules than the polyester fabric which is highly crystalline. It can also be seen from table 5 that the

Results and Discussion

The present study revealed that prepared thiazole-imidazole junction derivative showed wide range of shade. This thiazole-imidazole junction derivative was obtained using conventional methods and the synthesis is extremely convenient and relatively inexpensive. These dyes provide bright shades of red, orange, brown and an outstanding property of these dyes is their high affinity and intensity of colour with only a small molecule of the dyes. The nature of the substituents in the diazo component has little or no effect in the visible absorption spectra and shade of the dyeing. Furthermore, these dyes show good exhaustion, fixation, and uniform dyeing properties in addition to the good fastness properties. Some of the dye have good rating for fastness properties on both nylon and polyester fabrics. These dyes can hence be used commercially.

Exhaustion and fixation data, Washing and Light Fastness Result, Perspiration Fastness Result, and Rubbing Fastness Results are shown in the Table number 4,5,6 and 7 respectively.

TABLE 4 % Exhaustion and fixation of Synthesized Dyes

Dye no	Polyester		Nylon	
	Dry	wet	dry	Wet
A	4	4	3-4	4
B	4	4-5	3-4	4
C	3-5	4-5	4	4
D	5	4	4	4
E	5	4-5	4-5	4
F	4-5	4-5	4-5	4-5
G	4	4-5	3-4	4
H	5	4-5	3-4	4

Table 5 Washing and Light Fastness Result

Dye no.	% Exhaustion Polyester	%Exhaustion Nylon	%fixation Polyester	%fixation Nylon
A	70.25	70.53	80.21	94.12
B	71.57	72.25	87.15	90.69
C	73.38	73.7	85.93	90.03
D	71.95	70.10	78.01	92.40
E	70.70	69.65	77.62	87.51
F	77.0	75.08	75.56	92.81
G	73.0	73.50	84.83	93.37
H	70.98	72.88	79.50	87.23

Table 6 Perspiration Fastness Result

Dye no.	Polyester		Nylon	
	Acid	Alkaline	Acid	Alkaline
A	2-3	4	4	4
B	4	4-5	4	3-4
C	2	4	4	3-4
D	3-4	2-3	3-4	3-4
E	3-4	2-3	3-4	4
F	3-4	2-3	3-4	3-4
G	3	4-5	3-4	4
H	3	4	3-4	4

Table 7 Rubbing Fastness Results

Dye no.	Polyester		Nylon	
	Wash	light	Wash	Light
A	4-5	6	4	3
B	4-5	4-5	3-4	3
C	4-5	4-5	4	3
D	4-5	5	4	4
E	4-5	4-5	4	3-4
F	4-5	4-5	3-4	3
G	4-5	4-5	3-4	3
H	4-5	5-6	3-4	3-4

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