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Hetero aryl azo Disperse Dyes and Their Application on Polyester Fabric

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[2-Imine-(1'-hydroxy)-phenyl]benzothiazole (**III**) was prepared and coupled with different diazotized substituted aniline derivatives (**V$_{a-l}$**) and their dyeing performance on polyester fabric was assessed. All the compounds (**VI$_{a-l}$**) shows good substantivity for polyester fabric and gave bright yellow to brown shades with good to excellent light, wash, rubbing and sublimation fastness.

Key Words: Azo disperse dyes, Polyester, Fabric.

INTRODUCTION

Substituted 2-amino benzothiazole have long been established as diazo components giving bright hues of moderate to good fastness on synthetic polyester fabrics[1]. Interest to achieve variety of shades with high tintorial value and overall good dyeing properties is subject of potential interest of researcher still. Rangnekar and Chaudhary[2], Seu[3,4], Dalal and Desai[5], Peters[6], Radulescu[7] and others have synthesized azo disperse dyes from 2-amino benzothiazole, 2-amino thiazole for dyeing polyester fabrics. Several another mono azo dyes derived from diazotized 2-amino benzothiazole ring have been reported[8-11]. Introducing new system by incorporating unsaturation vide preparing Schiff base with amino benzothiazole and it's application on polyester fabrics and it's properties have not been reported. Here we have tried to prepare the Schiff base by interacting 2-amino benzothiazole and salicyaldehyde further the same is used as a coupling component for certain diazotized amines and is evaluated on polyester fabrics after characterization.

EXPERIMENTAL

Preparation of 2-amino benzothiazole (II): 2-Amino benzothiazole were prepared by reported literature[12].

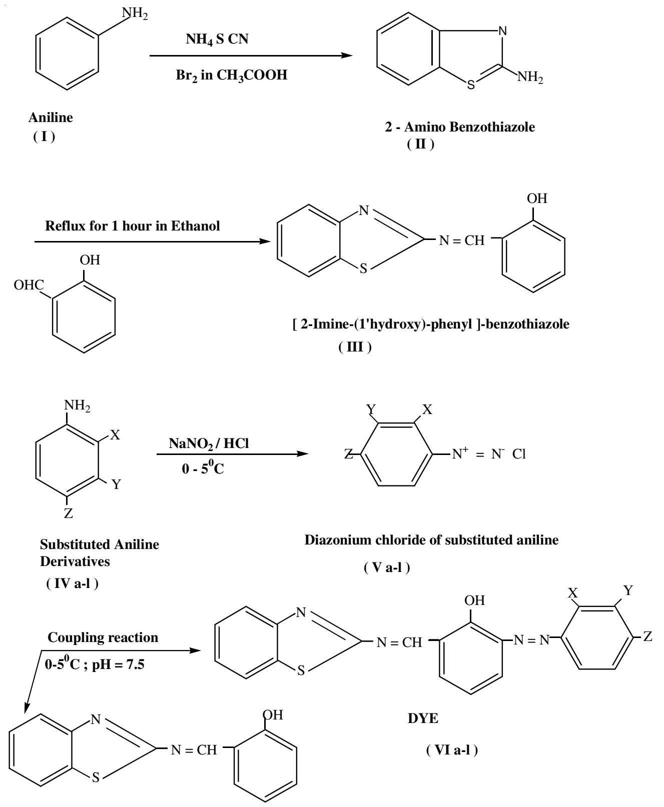
Preparation of [2-imine-(1'-hydroxy)-phenyl]-benzothiazole (III): 2-Amino benzothiazole (1.50 g, 0.01 mol) prepared by reported method and salicyaldehyde (1.12 g, 0.01 mol) were dissolved in ethanol (20 mL) and then refluxed for 1 h. The solvent was removed under reduced pressure and the product was crystallized from ethanol to get yellow crystal. (Yield 89 %, m.p. 202 ºC Found (%): C-66.48; H-3.60; N-11.15; Calculated (%): C-66.40; H-3.55; N-11.08).

Diazotization of compound (IV$_{a-l}$): The substituted aniline derivatives (**IV$_{a-l}$**) were diazotized using known amount of hydrochloric acid and sodium nitrite at 0-5 ºC. The excess amount of nitrous acid was neutralized with urea after spotting out the presence of nitrous acid on starch iodine paper. The mixture is then filtered to get the clear diazonium salt solutions (V$_{a-l}$) which were used for the subsequent coupling reaction.

Preparation of dye (VI$_{a-l}$): [2-Imine-(1'-hydroxy)phenyl]-benzothiazole (**III**) (2.53 g, 0.01 mol) was suspended in water was added to a solution of diazonium chloride of (**V$_{a-l}$**) gradually in 1 h at 0-5 ºC, maintaining pH 6-7 by the addition of sodium carbonate solution (10 % w/v). The stirring was continued for 3 h at the same temperature. The precipitated solid was then filtered, washed with water, dried and recrystallized from DMF-chloroform to give (**VI$_{a-l}$**). (Yield 69-79 %; m.p. > 300 ºC). Compound (**IV$_{a-l}$**) were commercially available.

RESULTS AND DISCUSSION

The infrared spectra of compound (**VI$_{a-l}$**) were recorded on a Perkin-Elmer spectrophotometer (Paragon-1000) using KBr pellets and PMR spectra on Varian spectrophotometer (Model EM-360L) using TMS as internal standard and DMSO-d_6 as a solvent. The absorption spectra were recorded on Perkin-Elmer spectrophotometer (Model U-320) in DMF solution at 25 ºC using 2×10^{-3} M dye concentration. The purity of the compounds was checked by thin layer chromatography using chloroform-methanol (95:5) as solvent and silica gel G as



For compound $\mathbf{IV}_{a\text{-}l}$, $\mathbf{V}_{a\text{-}l}$, $\mathbf{VI}_{a\text{-}l}$

Compd.	X	Y	Z	Compd.	X	Y	Z
a	H	H	H	**g**	H	H	CH_3
b	NO_2	H	H	**h**	Cl	H	H
c	H	NO_2	H	**i**	H	Cl	H
d	H	H	NO_2	**j**	H	H	Cl
e	CH_3	H	H	**k**	NO_2	H	NO_2
f	H	CH_3	H	**l**	OCH_3	H	H

Reaction scheme

absorbent. Polyester fabric (silver mette, 75 denier/36 filament) supplied by Gupta Dyeing & Printing Mill, Surat, Gujrat was washed thoroughly with water and then used for dyeing. The dye bath was prepared by dispersing the dye in a suitable dispersing agent, DMF and leveling agent. The pH was adjusted to 4 with acetic acid. The polyester fabrics were dyed under pressure (HT dyeing) at 130 ºC for 0.5 h, the material to liquor ratio being kept at 1:50. The above dyed fabrics were then treated with a solution of detergent (0.2 g) and sodium carbonate (0.1 g) in water (100 mL) at 80 ºC for 0.5 h. The treated fabrics after washing thoroughly 3-4 times with water were passed through hot air to fix the dye molecule properly into the fabric space and finally ironed.

The characterization data of ($\mathbf{VI}_{a\text{-}l}$) are given in Table-1. It is observed that the absorption maxima of compounds ($\mathbf{VI}_{a\text{-}l}$) vary from 425 to 475 nm. The compound (VI_k) shows absorption at longer wavelength (475 nm) which is due to the presence of two electron donating nitro groups in these compounds. The relatively lower absorption maxima of ($\mathbf{VI}_{a\text{-}f}$, $\mathbf{VI}_{h\text{-}j}$, $\mathbf{VI}_l$) vary from 435 to 455 nm, showing good colour over the polyester fabrics.

Despite the presence of the variety of electron donor and acceptor groups, the absorption maxima of dyes are relatively insensitive to the substituent changes. Taking dyes of structure ($\mathbf{VI}_{a\text{-}l}$), it can be assumed that electron delocalization structures enhancing electron availability on the imino nitrogen atom. However it does not enhance the colour depthening of the dyes and also not leading it to a higher bathochromic shift.

The structures of compounds ($\mathbf{VI}_{a\text{-}l}$) were confirmed by their IR, NMR spectra's (some of them were shown in Tables 1 and 2) and elemental analysis shown in Table-3. The IR spectra of compounds $\mathbf{VI}_a$, $\mathbf{VI}_g$, $\mathbf{VI}_j$ showed characteristic sharp band at different absorption band which is shown below in the Table-1.

TABLE-1
IR ABSORPTION BANDS (cm^{-1}) OF COMPOUNDS $\mathbf{VI}_a$, $\mathbf{VI}_g$, $\mathbf{VI}_j$

$\mathbf{VI}_a$	$\mathbf{VI}_g$	$\mathbf{VI}_j$	Assignment	Functional group
1320	1325	1320	C–N stretching	Amines (Ar-tertiary)
3260	3230	3215	O–H stretching, H bonded	Phenolic –OH g
1620	1665	1640	C = N stretching	Benzothiazole and
1410	1400	1415	C–N stretching	Imines
1500	1500	1505	C = N stretching	
1590	1610	1590	N = N stretching	Azo compound
2900	3000	2960 2875	Aliphatic C–H stretching	Normal paraffin
1450	1440	1435	C–H bending	$-CH_3$ group
1370	1360	1375		
3035	3025	3040	Aromatic C–H stretching	Benzene ring
1070	1065	1065	C–H bending	Benzene ring
–	–	750	C–Cl stretching	Halogen

TABLE-2
PMR SPECTRA SIGNALS OF COMPOUND $\mathbf{VI}_l$

Chemical shifts (δppm)	Multiplicities	Relative number of protons	Assignment
3.82	s	3	$Ar\text{-}OCH_3$
5.90	br, s	1	Ar-OH
7.20	d of d	4	H-5'', 6'',5'
7.35	t	5	H-4'',5'',4'
7.99	d	2	H-5',6', H-4,7
8.50	d	1	Ar–CH=N

The exhaustion and fastness properties of compound ($\mathbf{VI}_{a\text{-}l}$) are given in Table-4. It is observed that this compound show good to moderate substantives for polyester fabrics and exhaust well to give dyeing with good to excellent light fastness as well as rubbing and sublimation fastness.

The dyes ($\mathbf{VI}_{a\text{-}l}$) synthesized were possessing good overall characteristics which includes the fastness to shades on polyester fabrics however the additional functional groups and the imine formation used as a coupling component is not found to be so effective for the colour variations.

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TABLE-3
CHARACTERIZATION DATA (VI_{a-l})

Compd.	Yield (%)	m.p. (ºC)	λ_{max} (nm)	R_f value	Elemental analysis (%): Found (calcd.)		
					C	H	N
VI_a	73	275	450	0.90	67.07 (67.03)	3.90 (3.91)	15.65 (15.64)
VI_b	69	> 300	450	0.84	59.51 (59.55)	3.25 (3.22)	17.33 (17.36)
VI_c	69	> 300	455	0.86	59.55 (59.55)	3.25 (3.22)	17.36 (17.36)
VI_d	72	> 300	455	0.88	65.30 (65.28)	4.17 (4.14)	18.10 (18.13)
VI_e	75	> 300	440	0.89	65.25 (65.28)	4.17 (4.14)	18.15 (18.13)
VI_f	74	> 300	440	0.85	65.29 (65.28)	4.19 (4.14)	18.17 (18.13)
VI_g	78	> 300	440	0.81	59.00 (59.04)	3.22 (3.19)	17.25 (17.22)
VI_h	76	> 300	425	0.74	59.05 (59.04)	3.20 (3.19)	17.22 (17.22)
VI_I	79	> 300	435	0.72	59.07 (59.04)	3.18 (3.19)	17.27 (17.22)
VI_i	77	> 300	440	0.84	51.95 (51.94)	2.55 (2.59)	21.25 (21.21)
VI_k	71	> 300	455	0.82	62.70 (62.68)	4.00 (3.98)	17.40 (17.41)
VI_l	69	> 300	475	0.86	67.03 (67.03)	3.90 (3.91)	15.65 (15.64)

Melting points are uncorrected.

TABLE-4
SHADE, EXHAUSTION AND FASTNESS PROPERTIES (VI_{a-l})

Compd.	Colour on polyester	Exhaustion (%)	Fastness					
			Light	Washing	Rubbing		Sublimation	
					Dry	Wet	160 ºC	180 ºC
VI_a	Yellow	72.3	6	5	4-5	5	5	5
VI_b	Yellow	78.0	6	4	5	5	5	4
VI_c	Yellow-brown	79.0	5	5	5	5	4	5
VI_d	Yellow-brown	75.3	5-6	5	5	4-5	5	4-5
VI_e	Khakhi-yellow	67.3	6-7	5	5	5	5	5
VI_f	Dull-yellow	65.8	5	5	5	5	5	5
VI_g	Dull-yellow	63.4	5-6	5	5	4-5	5	5
VI_h	Yellow	60.2	5	5	5	5	4	4
VI_I	Brown-yellow	58.0	5	4-5	5	5	5	5
VI_i	Brown-yellow	60.7	6	5	4-5	5	5	5
VI_k	Yellow-brown	62.8	5	5	5	5	5	5
VI_l	Golden yellow	76.8	6	5	5	5	4-5	4-5

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