



Novel Azo Disperse Dyes Countaining Benzthiazole and Phenacyl

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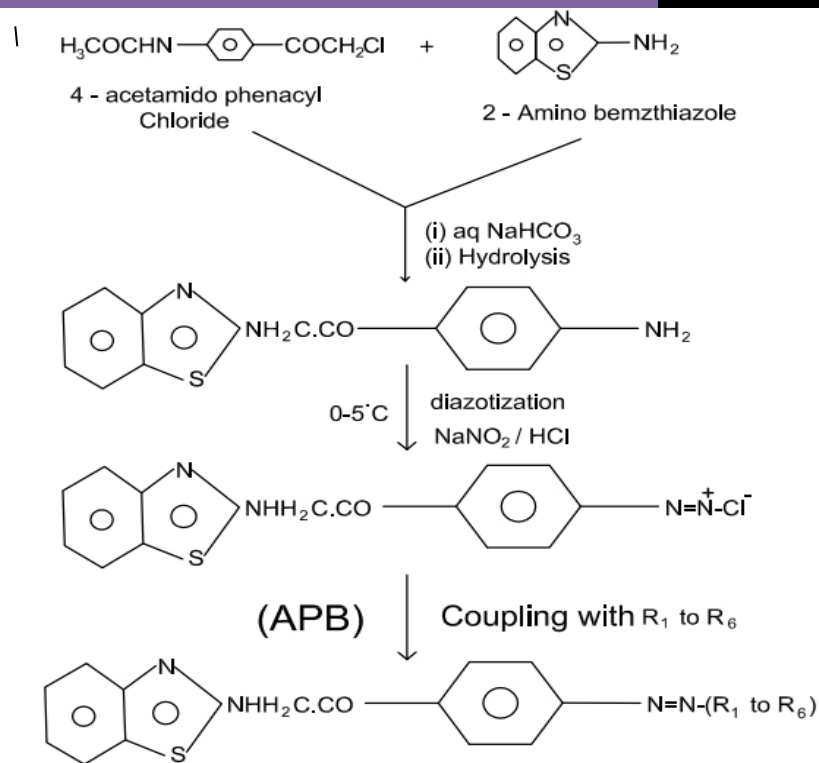
Abstract:

The aspect on novel azo disperse dyes countaining benzthiazole and phenacyl moieties has been undertaken. The novel aromatic amine countaining benzthiazole and phenacyl groups has been prepared. This was diazotised and coupled with different coupling components. All the resulting novel disperse dyes have been characterized by Elemental analysis IR, NMR, Visible spectroscopy. The produced disperse dyes were employed for dyeing of commercial nylon and polyester fibres and the fastness properties of dyes were evaluated.

Keywords: *Recycled plastic, Antioxidants*

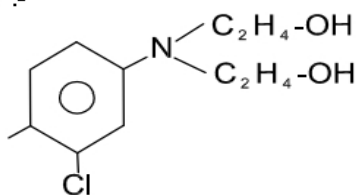
1. Introduction

In the field of dyes, numerous mono and bis azo dyes contribute major industrial implementation. These dyes are mostly based on aromatic amines, benzidine derivatives and naphthalene based couplers. [1]. Recently most of the benzidine based dyes have been banned all over the world. Due to their carcinogenic and toxic nature. [2]. The introduction of phenacyl group (PhCOCH₂-) and benzthiazole group into dye moiety may afford the dyes with better dyeing properties up to some extent. Till now, no such dye synthesis has been reported in literature. So it is interesting to explore the synthesis containing phenacyl group. Hence, the present paper comprises synthesis, characterization and fastness properties of phenacyl group containing disperse azo dyes. The whole work is furnished into **scheme-1**.

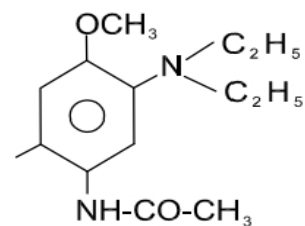


Where :- (R₁ - R₆)

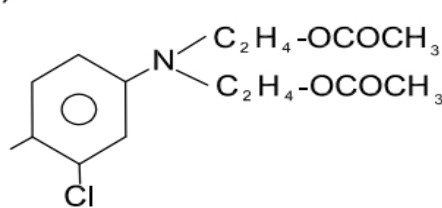
(I) R₁ :-



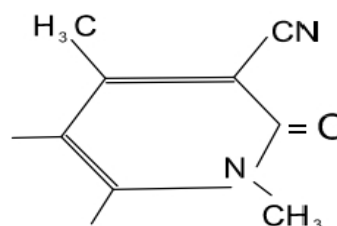
(IV) R₄ :-



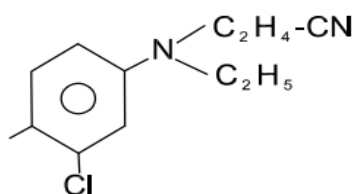
(II) R₂ :-



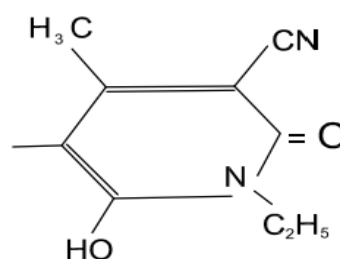
(V) R₅ :-



(III) R₃ :-



(VI) R₆ :-



2. Experimental

1. Materials

4-(N-acetamido phenyl)-2-Chloroethanone (i.e 4-acetamido phenacyl chloride) was prepared according to the method reported in literature

All the coupling component listed in Table-1 obtained from Jupiter Dyes, Vapi. Undyed nylon and polyesters fibres were obtained from Kiran Threads Ltd, Vapi. All the other were used of Laboratory grade.

3. Preparation of 1-(4-Aminophenyl)-2-(N-2'-benzthazolyl amino)

Ethanone: To a mixture of 2-Amino benzthiazol (0.05 mole) in 40 ml. Anhydrous acetone few drops of pyridine and sodium bicarbonate as acid acceptor in 250ml flask, 4-acetamido benzene phenacyl chloride (0.05 mole) was added gradually at 20°C.

The reaction mixture stirred and kept a side overnight keeping temperature below 20°C. 1-(4-acetamido phenyl)-2-(N-2'-benzthazolyl amino) ethanone is filtered off-washing with cold acetone yield crystals of product. The melting point was 112-3°C (Uncorrected).

It was hydrolysed by alcoholic HCL under reflux for 4-5 hours and the resultant cool solution was neutralized by aqueous NH₃ to yield 1-(4-aminophenyl)-2-(N-2'-benzthazolyl amino) ethanone. The melting point of the product is 99-100°C (Uncorrected).

4. Diazotization 1-(4-amino phenyl)-2-(N-2'-benzthazolyl amino) ethanone

(0.01 mole) was 1-(4-amino phenyl)-2-(N-2'-benzthazolyl amino) ethanone dissolved in Hydrochloride acid (0.03 mole) by vigorous stirring. The solution was cooled to 0-5°C in ice bath. A solution of sodium nitrite (0.01 mole) previously cooled to 0°C was added drop wise to above solution maintaining the temperature below 5°C. The reaction mixture is stirred until the positive test of nitrous acid on starch iodide paper. Excess of nitrous acid was destroyed by adding the required amount of **sulphamic acid**. The resultant diazo component was used for subsequent coupling reaction. Different couplers (R₁-R₆) were taken as their sodium salts. The solution of acid couplers in their respective medium was cooled and stirred. To this well stirred solution, the above mentioned diazo solution was added while maintaining pH at 6-7 by addition of sodium carbonate solution (10% w/v) maintaining the pH of the resulting solution. The solution was stirred continuously for these hours keeping temperature below 5°C. After completion of reaction, the solid material was filtered off and washed with water.^{4,5} The disperse dye was purified from DMF- chloroform system. The melting points of all the dyes are Uncorrected.

5. Measurements

C, H, N, S contents of all samples were estimated by C, H, N, S, P analyser, Carlo Erba, Italy. The IR spectra of all the samples were scanned in KBr on Nicolet Impact 400-D. The NMR spectra of all the samples were taken in CDCl₃ on HITACHI R-1500, 60 MHz instrument. The dyeing assessment of all the dye samples was carried out on nylon and polyester by the method reported in literature.^{6,7} Visible spectra of all the dyes are taken in methanol.

6. Results and Discussion

The novel amine 1-(4-amino phenyl)-2-(thiazolyl amino) ethanone having the structure shown in **Scheme-1**. Confirmed by elemental and spectral analysis. The C, H, N contents and IR, NMR spectral features of all this amine are shown in **Table-1**. Consistent with the predicted structure. The azo disperse dyes obtained from this compound are shown in **Scheme-1**. The detailed analysis of all the dyes are presented in **Table-2**. The C, H, N contents of all the dye samples are consistent with the predicted structures (**Scheme-1**). The IR spectra of all the dyes comprises the important bands due to initial diazo component. The bands due to -CH₂ and CO of COCH₂ dimethyl amine (CH₃)₂ N groups

are at their respective positions. Only the discernible difference in the spectra of dye samples is that the new band at 1625 and 1450 Cm^{-1} is observed due to azo $-\text{N}=\text{N}-$ groups. The other bands due to presence of coupling component are at their respective position. The observed bands in the spectra for each dye are shown in **Table-1**. The NMR spectral data of CDCl_3 soluble dye samples are shown in **Table-1** are confirming the structure of dyes. The UV visible spectra of all the dye samples was recorded in methanol. It is apparent that the Wave length of maximum absorption is relatable to the azo groups in the compounds and it is observed with in the region of 380-490 nm. Variation in λ_{max} being attributed to structural vibration in the dye samples. All the dyes were dyed on polyester and Nylon fibers at 2% dye bath of shade and gave yellow to dark brown shades implied in **Table-3**. The dye bath exhaustion of dyes on both fibres was (about 20-30%). Thus fixation of dye an fibres is very good (70 to 80%). The light fastness of the dyes is shown in **Table-3**. The light fastness on fibres varied from good to very good. All the dyes have higher rating. The washing of the dyes is also very good compared with commercial dyes. The produced dyes have good exhaustion on dye pattern and better washing and light-fastness.

7. Conclusion

Unsymmetrical acid azo dyes have been prepared using 1-(4-aminophenyl)-2-(N-2'-benzthiazolyl amino) ethanone. The dyes thus obtained gave dyeing having deeper shades and Excellent light fastness and wash fastness properties. Results of the work indicate that 1-(4-amino phenyl)-2-(N-2'-benzthiazolyl amino) ethanone is a potential diazo component in the synthesis of acid dyes.

Table-1: Analysis of 1-(4-amino phenyl)-2-(N-2'-benzthiazolyl amino ethenone

Elemental analysis

$\text{C}_{15}\text{H}_{13}\text{N}_3\text{SO}$ (283)

	%C	%H	%N	%S
Calc.	63.6	4.59	14.84	11.30
Found	63.4	4.50	14.80	11.20

IR (KBr) Cm^{-1}

ν of NH_2 : 3400, 3300

ν co of $-\text{COCH}_2$: 1700

ν CH_2 of $-\text{COCH}_2$: 2872, 2950

ν aromatic: 3030, 1500, 1600

$^1\text{H-NMR}$ (CDCl_3)

Singlet δ 7.2 ppm 4H (aromatic) \Rightarrow Singlet δ 7.3 ppm 4H (aromatic of Benz thiazolyl)

Singlet δ 2.1 ppm 2H ($-\text{CH}_2-$)

Singlet δ 2.8 ppm 1H (NH) \Rightarrow Singlet δ 2.4 ppm (NH_2)

Table 2: Characterization of Novel Azo Disperse Dyes

Dye Sample	MP $^{\circ}\text{C}$ (Uncorrected)	Mol. Wt. (gm/mole)	% C		%H		%N		%S	
			Found	Cal.	Found	Cal	Found	Cal	Found	Cal
D-R1	135-6	496.5	60.42	60.20	5.03	4.90	11.27	11.20	6.44	6.40
D-R2	162-3	592.5	60.75	60.60	4.89	4.70	9.45	9.30	5.40	5.30
D-R3	167-8	489.5	63.73	63.5	4.90	4.70	14.30	14.20	6.53	6.40
D-R4	195-6	517	64.99	64.80	5.99	5.70	13.53	13.40	6.19	6.10
D-R5	205-6	445	62.02	62.0	4.26	4.1	15.73	15.60	7.19	7.1
D-R6	225-4	459	62.74	62.60	4.57	4.40	15.25	15.18	6.97	6.93

Table 3: Characterization and Dyeing assessment of Azo Disperse Dyes

Dye	λ max (nm)	Shade	Dyeing of Properties			
			Dyeing of Polyester		Dyeing of Nylon	
			LF	WF	LF	WF
D-R1	347	Light Yellow	4-5	4	4	4
D-R2	415	Yellow	4-5	4	4	3
D-R3	420	Yellow	4	3	4	5
D-R4	455	Light Yellow	6	5	3	4
D-R5	345	Light Green	4-5	4	4	5
D-R6	342	Green	5	4-5	4	4

LF= Light Fastness

WF= Washing Fastness

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