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Synthesis, Characterization of some novel disperse azo dyes based on 4-(N-acetyl amino) 2-methyl phenacyl chloride.

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ABSTRACT

Some novel azo disperse dyes prepared 4-(N-acetyl amino) 2-methyl phenacyl chloride was reacted with aniline. Then resultant product were hydrolysis to give yield 1-(4-amino-2-methyl phenyl)2-(phenacyl) ethanone. Then diazotized by the standard procedure and coupling with various components. Thus a series of azo disperse dyes has been prepared. After syntheses compounds were characterized by chemical as well as instrumental methods. like melting point, elemental analysis and IR spectral studies.

Keywords: Disperse dyes, Hydrolysis, diazotized, coupling components, IR

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INTRODUCTION

Disperse dyes are coloured organic compound which are not completely insoluble in water and suitable for colouring hydrophobic fibers. The first member of the group of disperse dyes were introduced in 1924 by Baddiley and Sheperdon of the British Dye Stuffs Corporation (Duranol dyes) and by Ellis of the British Celanese Company (SRA dyes) [1] for dyeing it.

Traditionally, azo dyes are the most important class of commercial dyes, occupying more than half of the dye chemistry, which contain phenols as intermediates. [2-5] Hence, in continuation of earlier work, [6,7] the present communication comprises the synthesis, characterization of some novel disperse azo dyes based on 4-(N-acetyl amino) 2-methyl phenacyl chloride.

MATERIALS AND METHODS

All reagents were of analytical reagent grade and were used without further purification. All the products were synthesized and characterized by their spectral analysis. All chemicals and solvents like acetone, Methanol, Aniline, NaNO_2 , sodium acetate were purchased from S.D. Fine Chemicals (India).

Melting points were taken in open capillary tube. IR spectra (KBr) were recorded on PerkinElmer I.R. Spectrophotometer and C, H, N of all disperse dyes were estimated by the means of a Carlo Erba elemental analyser (Italy).

Experimental

Synthesis of 1-(4-amino-2-methyl phenyl)2-(phenacyl) ethanone

A mixture of 40 ml of anhydrous acetone and 1 ml of pyridine and aniline in 250 ml flask then added drop wise to a mixture of (0.05M) 4-(N-acetyl amino) 2-methyl phenacyl chloride (AMPC) and sodium bicarbonate as an acid acceptor. The reaction mixture is set aside overnight. Then resultant product was hydrolysed by refluxing with 75 ml of ethanol containing 15 ml HCl for 4-5 hours. The cool solution was diluted with water and was made just alkaline with conc. NH_3 solution ($d=0.880$) to give yield 1-(4-amino-2-methyl phenyl)2-(phenacyl) ethanone (A).

Synthesis of azo disperse dyes

1-(4-amino-2-methyl phenyl)2-(phenacyl) ethanone (0.01 mole) (A) was dissolved in HCl (0.03 mole) with stirring and the solution was cooled to $0-5^\circ\text{C}$ in an ice-bath. A solution of sodium nitrite (0.01 mole) in 5 ml water cooled to 0°C then was added. The excess nitrous acid was neutralized with sulfamic acid/urea (1.0 gms) and the mixture was filtered to get the clear diazonium salt solution. Diazotization of various aromatic amines was performed by a reported method [8-9]. D1-D6 coupling component was dissolved in HCl (15 ml) and then solution cooled

0-5°C To this well stirred solution the above diazonium salt solution was added slowly so that temperature did not rise above 0-5 °C while maintaining the pH 4-5 by the action of sodium acetate solution(10% w/v) the mixture was stirred for 3 hrs. at 0-5°C. After completion of the reaction the solid material was filtered, washed and dried it. So D1-D6 azo disperse dyes were prepared.

RESULTS AND DISCUSSION

The azo disperse dyes obtained from these compounds are shown in Scheme-I. The elemental analysis of C,H,N confirmed by Table: I. The observed bands in the IR spectra for each dye are shown in Table-II.

IR spectra of all six series of disperse azo dyes contain aromatic nuclei, azo group and hydroxyl group. The band appeared from 1050 to 1350 cm^{-1} due to primary -OH group, The bands at 1500, 1200 and 1050 cm^{-1} appeared in the double bond region due to aromatic stretching. The strong band at 1600 to 1630 cm^{-1} appeared in the spectra are considered for the presence of azo (-N=N-) group. The band of 1700 to 1735 cm^{-1} might be responsible to -CO of -COCH₂ group. In the spectra of dyes obtained from aniline derivatives bearing a β -hydroxy ethyl side chain attached to the tertiary nitrogen. The weak bands observed 2920 which is attributed to the -CH₂ stretching vibration.

The IR spectra of all dyes comprise the important bands due to initial diazo component. The bands due to -CH₃ and -CO of -COCH₂ dimethyl amine(-CH₃) . N group are at their respective positions. Only the discernible difference in the spectra of dye samples is that the new bands at 1625 and 1350 cm^{-1} are observed due to azo -N=N- groups. The other bands due to presence of coupling component are at their respective positions.

CONCLUSION

Unsymmetrical azo disperse dyes have been prepared using based on 4-(N-acetyl amino) 2-methyl phenacyl chloride. The dyes thus obtained give dyeing having deeper shade and excellent light fastness and wash fastness properties. However, disperse dye can be used by even young children to make design paper, which can then be transferred to polyester fabrics or other synthetics.

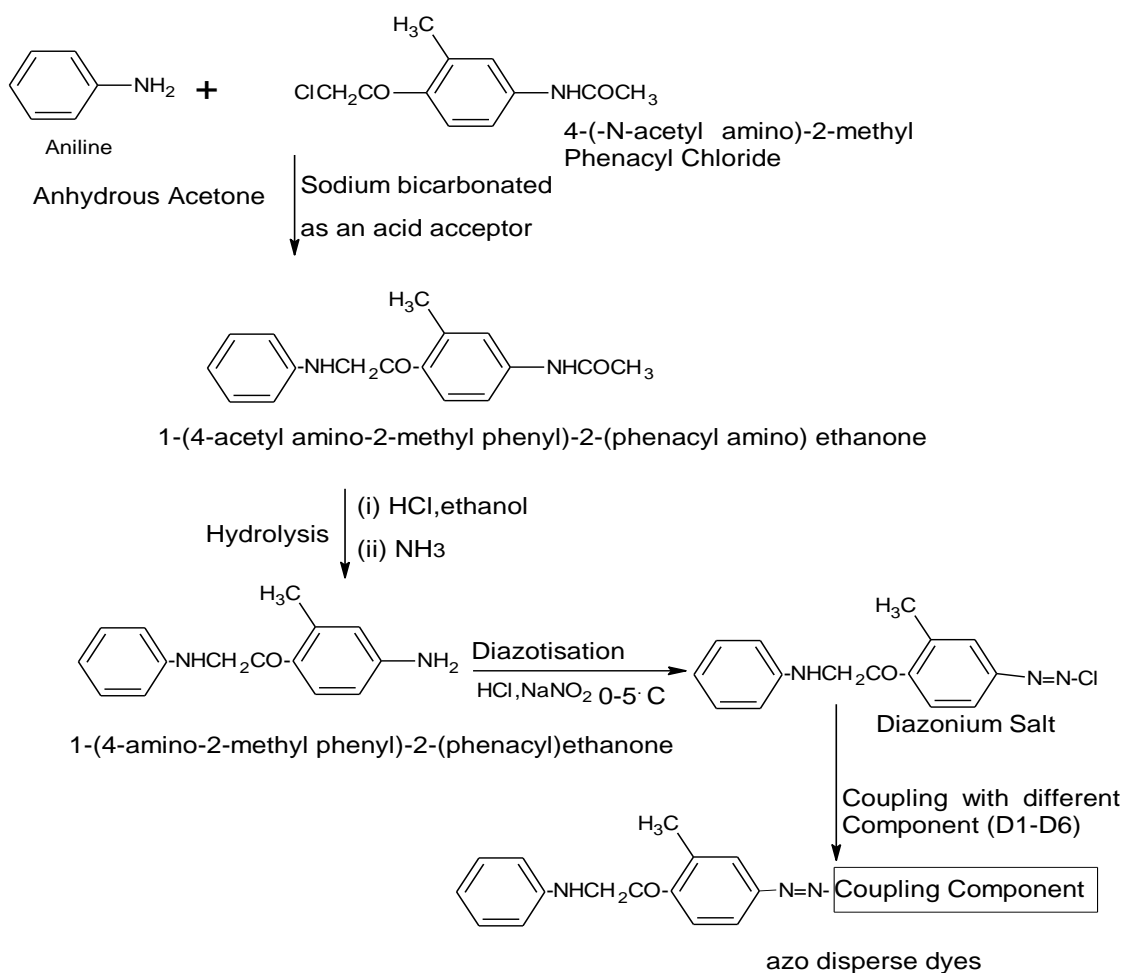
Table : I Characterization of novel azo disperse dyes

Dye No	Molecular Formula	Mol.Wt (gm/mole)	Melting Point °C	% C		% H		% N	
				Found	Cal	Found	Cal	Found	Cal
D-1	C ₂₅ H ₂₇ N ₄ O ₃ Cl	466.5	230	63.4	63.64	5.3	5.52	12.2	12.32
D-2	C ₂₆ H ₃₀ N ₄ O ₃	446	225	69.88	69.95	6.70	6.72	12.40	12.55
D-3	C ₂₂ H ₂₁ N ₅ O ₃	403	235	60.20	60.50	5.20	5.21	11.90	17.36
D-4	C ₂₂ H ₂₁ N ₅ O ₃	403	208	65.20	65.50	5.20	5.21	11.90	17.36
D-5	C ₂₆ H ₂₉ N ₅ O	425	245	73.40	73.41	6.30	6.35	16.41	16.41
D-6	C ₂₉ H ₃₂ N ₆ O ₃	512	255	67.90	67.96	6.22	6.25	16.38	16.40

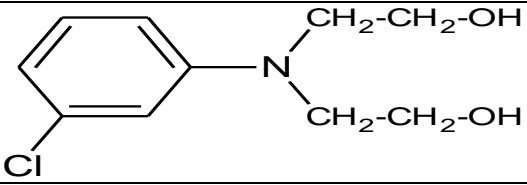
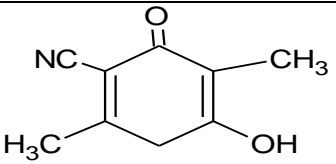
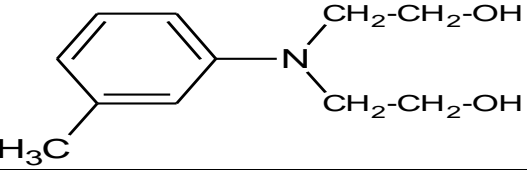
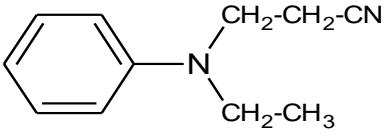
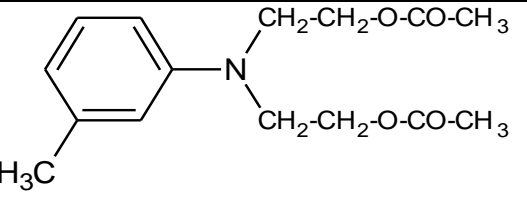
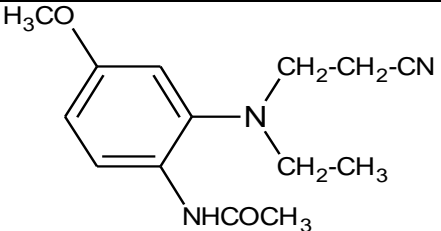
Table : II Position of selected bands in IR spectra of Disperse Azo dyes :

Dye No.	C-H Stretching of aromatic cm^{-1}	CH_3 Stretching cm^{-1}	CN Stretching cm^{-1}	C=O ($\text{CH}_2\text{OCOCH}_3$) COCH_2 Stretching cm^{-1}	N=N Stretching cm^{-1}	1,2,4-Tri Sutedited cm^{-1}	Tertiary amine cm^{-1}	Primary alcohol cm^{-1}
D-1	3030, 1600,1480	-----	-----	-----	1600 1630	1590,1640 1475,1510	1240,1280 3420	1050,1130 1270,1550
D-2	3030, 1480,1490	2840,1480, 1320	-----	-----	1600 1630	3100,1600, 1475	1250,1360 3420	1070,1360 1100
D-3	3030, 1520,1610	2830,1470, 1350,1380	-----	1720	1600 1630	3100,1600 1650,1475	1250.1280 3360,3450	1050,1100 1280,1360
D-4	2960 -----	2820,1470, 1350	2240	1735	1600 1610	1620,1475 1510	1250,1340 3420,1600	1050,1100 1300
D-5	3030, 1500	-----	2220	-----	1620	1620 1515	1245, 3415	1050,1100 1310
D-6	3030, 1520,1610	-----	-----	1700,1720	1625	3100,1600 1650.1475	1250,1280 3350,3430	1050.1100 1270,1350

Reaction scheme-I



Where,

D-1		D-4	
D-2		D-5	
D-3		D-6	

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