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FORMATION OF SOME NOVEL DISPERSE AZO DYES: SYNTHESIS, CHARACTERISATION AND PRINTING PROPERTIES

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ABSTRACT

Formation of some novel disperse azo dyes synthesis by the coupling component of diazonium salt with 1-(4-N-acetyl amino) 2-methyl phenyl-2-chloro ethanone to give the corresponding various azo disperse dyes (D13-D18). These dyes were applied to polyester and nylon fabric and their fastness properties were evaluated. Dyes were characterized by IR, elemental analysis, UV-Visible spectral studies.

Key words: Disperse azo dyes, UV-Visible spectra, Fastness properties.

INTRODUCTION

Disperse dyes are organic colors having less water solubility, these are applied in colloidal aqueous dispersions to hydrophobic textile fibers in which the dyes literally dissolve and produce desired coloration. The development of disperse dyes is due to significant increase in the world production of polyester fibers[1] as compared to other fibers. Over 90% of disperse dyes usage is for the coloration of polyester.

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)[2] 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[3-6]. Hence, in continuation of earlier work[7-9], the present work shows the formation of some novel disperse azo dyes synthesis by the coupling component of diazonium salt with 1-(4-N-acetyl amino) 2-methyl phenyl-2-chloro ethanone to give the corresponding various azo disperse dyes (D13-D18).

EXPERIMENTAL

Material and methods

All the chemicals used were of analytical reagent grade and were used without further purification, All the product were synthesized and characterized by their spectral analysis, All Chemicals and solvents like acetone, ethanol, $NaNO_2$, sodium acetate were purchased from S. D. fine Chemicals (India).

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Melting points were taken by open capillary tube and are uncorrected. The UV-Visible spectra were recorded in DMF using Shimadzu A-200 Spectrophotometer and C, H, N of all disperse dyes were estimated by the means of a carlo Erba elemental analyzer (Italy) The characteristic data of different molecules were studied their light, wash fastness properties[10,11] and further study applied on polyester fibers[12] and nylon fibers.

EXPERIMENTAL

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

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

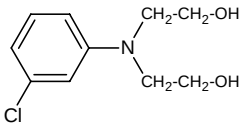
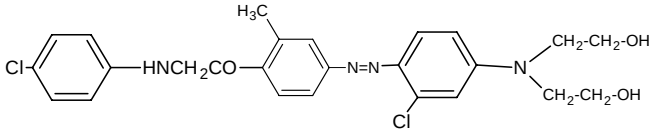
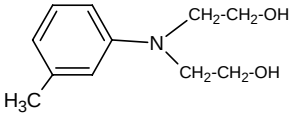
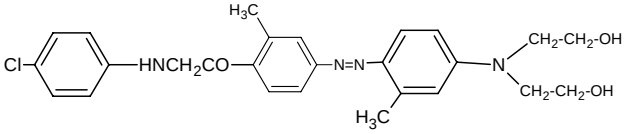
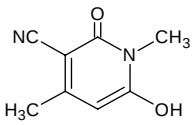
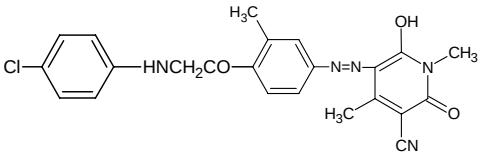
Formation of some novel disperse azo dyes

1-(4-amino-2-methyl phenyl)-2-(N-4-chloro phenyl amino) ethanone (A). (0.01 mole) was dissolved in HCl (0.03 mole) with stirring and the solution was cooled to 0-5^{o}C in an ice-bath. A solution of sodium nitrite (0.01 mole) in 5 mL water cooled to 0^{o}C then was added. The excess nitrous acid was neutralized with sulfamic acid/urea (1.0 g) and the mixture was filtered to get the clear diazonium salt solution. Diazotization of various aromatic amine was performed by a reported method[13,14]. Then D13-D18 coupling component was dissolved in HCl (15 mL) and then solution cooled 0-5^{o}C. To this well stirred solution the above diazonium salt solution was added slowly so that temperature did not rise above 0-5^{o}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^{o}C. After completion of the reaction the solid material was filtered, washed and dried it, so D13-D18 azo disperse dyes were prepared.

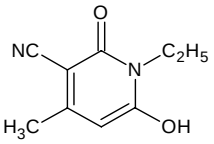
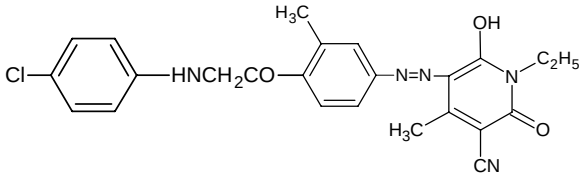
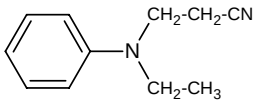
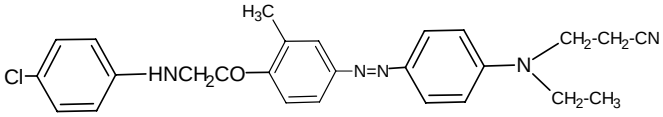
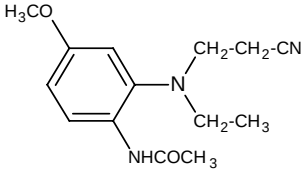
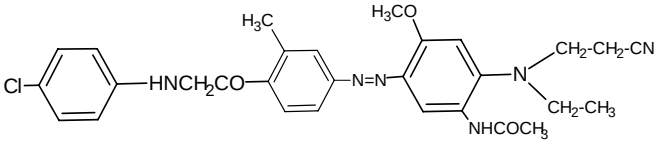
RESULTS AND DISCUSSION

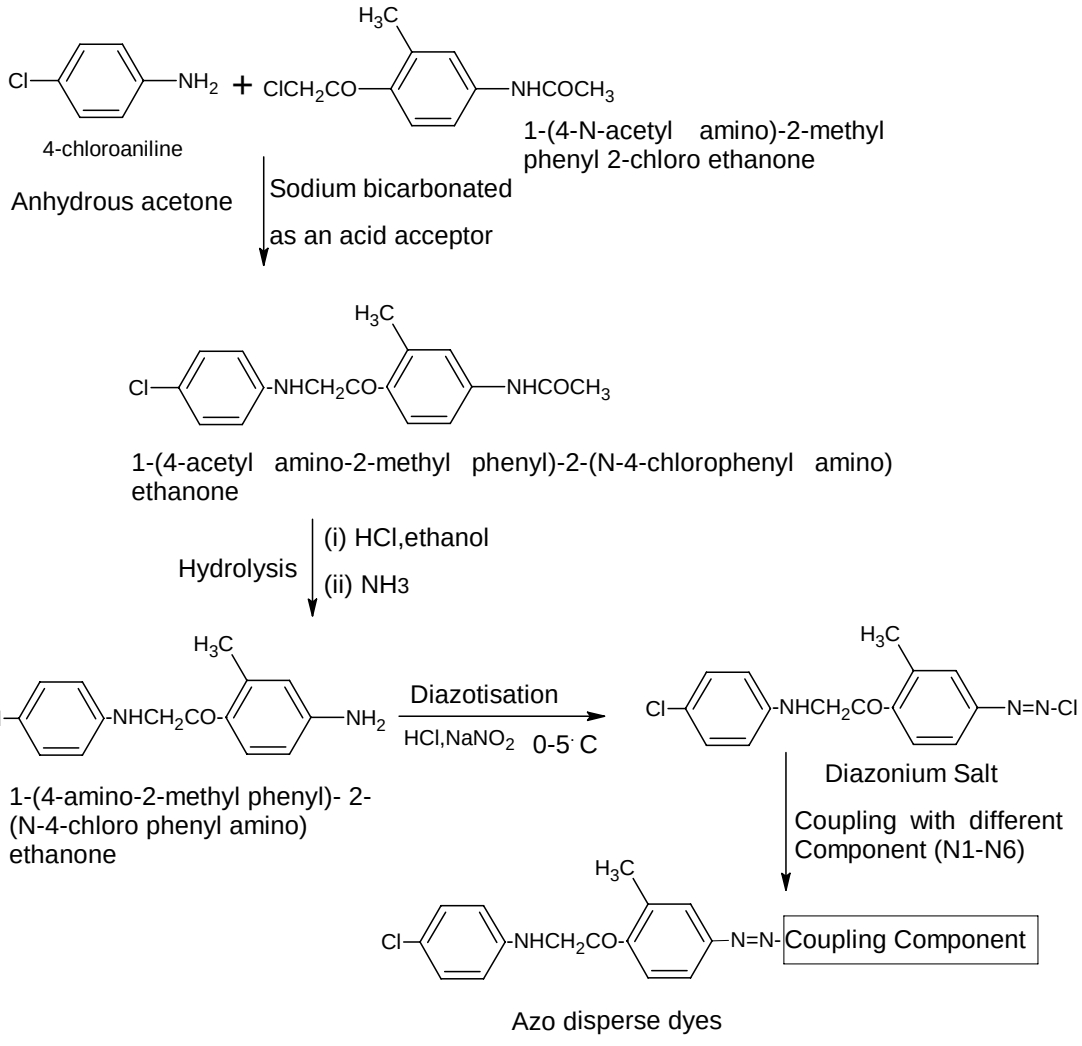
The azo disperse dyes obtained from this compounds are shown in Scheme 1. The Structure of the coulping component and corresponding of novel azo disperse dyes in Table 1.

Table 1: Structure of the coulping component and corresponding of novel azo disperse dyes

Dye No.	Coulping component	Disperse dyes
D-13		
D-14		
D-15		

Cont…

Dye No.	Coulping component	Disperse dyes
D-16		
D-17		
D-18		



Scheme 1

The observed bands in the IR spectra for each dye are shown in Table 2, IR spectra of all six series of disperse azo dyes contain aromatic nuclei, azo group and hydroxyl group. The band appeared from 1050 to 1355 cm^{-1} due to primary –OH group, The strong band at 1575 to 1640 cm^{-1} appeared in the spectra are considered for the presence of azo (-N = N-) group. The band of 1720 to 1735 cm^{-1} might be responsible to –CO of $–COCH_2$ group. In the spectra of dyes obtained from 4-chloro aniline derivatives side chain attached to the tertiary nitrogen. The weak bands observed 2890-and 2950 cm^{-1} which is attributed to the $-CH_2$ stretching vibration.

Table 2: 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 (CH_2OCOCH_3) $COCH_2$ Stretching cm^{-1}	N=N Stretching cm^{-1}	1,2,4-tri Sustituted cm^{-1}	Tritiary amine cm^{-1}	Primary-alcohol cm^{-1}
D-13	2950, 1580 1480	–	–	–	1590	3150, 1580 1490	1230, 1280 1350	1050, 1100 1260, 1300
D-14	2930, 1615 1475	2850, 1475, 1330, 1350	–	–	1600 1610	3110, 1615, 1465	1230, 1350 3430	1060, 1355 1100
D-15	2950, 2975 1480	2820, 1445, 1360	2210 2240	1720	1630	1640, 1520	1320, 1350 3450, 1520	1050, 1100 1350, 1320
D-16	3000, 2950 1610, 1520	2845, 1480, 1350	2230	1735	1630 1640	3000, 1640, 1475, 1525	1230, 1275 3200, 1525	1050, 1100 1260, 1330
D-17	2920, 2990 1600, 1480	–	2225	–	1600	1590, 1640 1490, 1525	1240, 1310 3420, 1520	1050, 1100 1240, 1320
D-18	1150 530, 640	–	–	1720	1575 1620	3100, 1560 1640, 1475	2950, 2990 1580, 1620	1050. 1100 1250, 1290
All these spectra comparised the bands around 2890 and 2950 cm^{-1} presented of $-CH_2$ of $-COCH_2$ group								

The IR spectra of all dyes comprise the important bands due to initial diazo component. The bends due to $–CH_3$ stretching 2820-2850 and 1330-1360 cm^{-1} and -CO of $-COCH_2$ dimethyl aniline ($-CH_3$). The other bands due to presence of coupling component are their respective positions. The elemental of C, H, N confirmed by Table 3.

Table 3: Characterization of novel azo disperse dyes

Dye No.	Molecular formula	Mol. wt (g/mole)	Melting Point °C	% Yield	% C		% H		% N	
					Found	Cal.	Found	Cal.	Found	Cal.
D-13	$C_{25}H_{26}N_4O_3Cl_2$	502	170	73	59.66	59.76	5.10	5.17	11.10	11.15
D-14	$C_{26}H_{29}N_4O_3Cl$	480.5	207	75	64.90	64.93	6.00	6.03	9.90	9.98
D-15	$C_{23}H_{20}N_4O_3Cl$	435.5	214	72	63.30	63.37	4.50	4.59	11.00	11.2
D-16	$C_{24}H_{22}N_4O_3Cl$	464.5	185	77	61.90	62.00	4.68	4.73	10.30	10.33
D-17	$C_{26}H_{26}N_5OCl$	460.5	235	67	67.72	67.75	5.61	5.64	3.40	3.47
D-18	$C_{29}H_{31}N_6O_3Cl$	548.5	178	64	63.60	63.62	5.60	5.65	15.20	15.31

The visible absorption spectroscopic properties of the all dyes were recorded in DMF. Absorption maximam (λ_{max}), Intensites (log ε), dyeing assessment of disperse azo dyes on polyester and nylon fabrics are shown in Table 4, The absorption maximam (λ_{max}) of all the dyes falls in the range 418-516 nm in DMF. The values of the logarithm of molar extinction coefficient (log ε) of all the dyes were in the range 4.008-4.239, consistent with their medium absorption intensity. The disperse azo dyes were applied at a 2% dye bath on nylon and polyester fabrics and give various shade implied in Table 4.

Table 4: Absorption maximam (λ_{max}), intensites (log ε), dyeing assessment of disperse azo dyes on polyester and nylon fabrics

Dye	λ_{max} (nm)	Log ε	Shade	Dyeing properties			
				Dyeing on polyester		Dyeing on nylon	
				Light	Wash	Light	Wash
D-13	464.0	4.239	Orange	5	4	5	4
D-14	504.0	4.132	Pale rose	4-5	4	4-5	4
D-15	452.0	4.027	Pista	4	3	4	3
D-16	458.0	4.047	Orange	5	5	5	5
D-17	516.0	4.067	Off white	5	4	5	4
D-18	418.0	4.008	Light green	5-6	5	5-6	5

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

Fastness properties

The light fastness of all the dyes rating 3-6 for nylon and polyester fabrics, which shows light fastness good to very good for nylon and polyester. The wash fastness of all the dyes rating 3-5 for nylon and polyester fabrics, shows wash fastness and of all the dyes very good to excellent (Table 4).

CONCLUSION

Formation of some novel disperse azo dyes have been prepared using based on 1-(4-N-acetyl amino) 2-methyl phenyl-2-chloro ethanone. The present study revealed that prepared azo disperse dyes showed wide range of shades. They showed good dyeing performance on polyester and nylon fiber shows. All the

dyes wash fastness very good to excellent and the light fastness values of the azo disperse dyes are good to very good.

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