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Synthesis and characterization of Chelating azo dyes Containing 8-Hydroxy Quinoline ligand

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Abstract
First we synthesized five sulfanilamide derivatives. Then the azo dye was synthesized by the coupling of diazoniumsalt of sulfanilamide derivatives with 8 hydroxy quinoline ligand. So D1-D5 Chelating azo dyes were prepared.After syntheses compounds were characterized by chemical as well as instrumental methods. like Melting point, elemental analysis, UV-visible spectroscopy and IR spectral studies.
Key words: sulfanilamide derivatives, 8-hydroxy quinoline *A*zo dye, UV-visible, IR Spectroscopy

Introduction
Azo compounds, with two phenyl rings separated by an azo (-N=N-) bond are versatile molecules and have received much attention in research areas both fundamental and application. The strong electronic absorption maximum can be tailored by ring substitution to fall anywhere from the ultraviolet to red visible regions, allowing chemical fine-tuning of color. This combined with the fact that these azo groups are relatively robust and chemically stable, has prompted extensive study of dyes and colorants.

The azo dyes have the general structure R-N=N-R', where R and R' are alkyl, aryl or heterocyclic radicals. Most of them are prepared by the condensation of azo compounds with hydroxyl, aldehydes or ketones. Several bidentate azo dyes in which the phenolic –OH group and azo nitrogen are present in such a way that they form six membered rings with metal ions. Azo dyes have been investigated by many workers as chelating agent and their metal chelates and complexes have been extensively used in dyeing industry [1-4] and studied dyeing properties [5-6]. Azo dyes have been widely used in various fields and technologies like textiles,leather, plastics,paper,laser liquid crystalline displays and ink jet printers [7-8]. It was thought interesting to explore the field of chelating azo dyes Containing 8-hydroxyl quinoline ligand.The proposed synthetic route is shown in Scheme-I.

Materials 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 ethanol, 8-Hydroxy quinoline, $NaNO_2$, HCl, sodium acetate were purchased from S.D.fine chemicals (india). Melting points were taken by open capillary tube and are uncorrected. The UV-Visible spectra were recorded in Shimadzu A-20 Spectrophotometer, The IR Spectra of all the chelating azo dyes are measured in KBr pellets was scanned on perkin Elmer spectrophotometer and C,H,N of all azo dyes were estimated by the means of a T.F.Flash elemental analyzer.

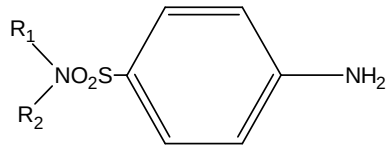
Experimental:
Synthesis of chelating azo dyes:
Diazotization of different five sulfanilamide derivatives (A) (0.01 mole) was dissolved in hydrochloric acid (0.03 mole) with stirring and the solution was cooled to 0-5° C in an ice bath. A solution of sodium nitrite (0.01 mole) in 5 ml water cooled to 0° C was then added and the reaction mixture was then stirred until the positive test of nitrous acid on starch iodide (e.e. blue colour on starch iodide paper).

8-Hydroxy quinoline was dissolved in hydrochloric acid and the solution was then cooled to 0-5° C. To this well stirred solution the above diazonium salt solution of was added slowly so that tempreture did not rise above 5° C while maintaining the pH 4-5 by the action of sodium acetate solution. The mixture was then stirred for 3 hrs. at 0-5° C. After completion of the reaction the solid material was filtered, washed and dried it. So D1-D5 Chelating azo dyes were prepared.

Rection Scheme-I

Where

R1	R2
Methyl	Methyl
Ethyl	Ethyl
Phenyl	Phenyl
Phenyl	Ethyl
2,4-dichloro phenyl	H
2,6 -dichloro-4-nitro phenyl	H

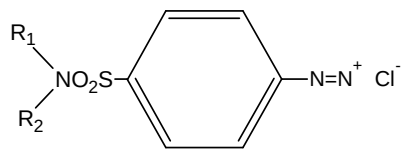


Sulfanilamide derivative

$NaNO_2$ + HCl

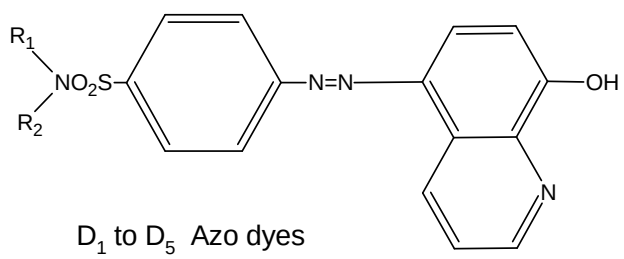
0-5 ^{0}C

Diazotization



Coupling with

8- hydroxy quinoline



D_1 to D_5 Azo dyes containing 8-HQ ligand

Results and Discussion

The observed bands in the IR spectra for each dye are shown in Table-I. Examination of the IR spectra of all the azo dyes revals that all the spectra are identical in the important features due to the presence of aromatic nucleus azo group, and hydroxyl group. Most of the spectra comrises a broad band extended from 3600 cm^{-1} to 3400 cm^{-1} mainly arsied due to -OH group. The bands at 1500,1200 and 1050 cm^{-1} appeared in the double bond region are due to aromatic stretching vibrations. The strong bands 1625, and 1575 cm^{-1} appeared in the spectra are considered due to presence of azo (-N=N) group. Apart from these a weak band observed around 2920 cm^{-1} and 2950 cm^{-1} which are attributed to the -CH_2 stretching vibrations. In the spectra of 8-hydroxy quinoline containing dyes, bands around 1630,1575 and 1500 cm^{-1} of due to 8-hydroxyquinoline moieity are observed [9]. The elemental of C,H,N confirmed by Table: II. The visible absorption spectroscopic properties of the all dyes were recorded in DMF. Absorption maximam (λmax), Intensites (log ε), dyeing assessment of azo dyes shown in Table-III, The absorption maximam (λmax) of all the dyes falls in the range 385-482 nm in DMF. The values of the logarithm of molar extinction coefficient (log ε) of all the dyes were in the range of 0.437-7.41, consistent with their medium absorption intensity.

Table: I Characterization of Dyes containing 8- Hydroxy Quinoline ligand

Dye No.	Mol. Formula	Mol. Wt.	% Yield	Elemental analysis										No. of COOH Group
				C%		H%		N%		S%		Cl%		
				Cald	Found	Cald	Found	Cald	Found	Cald	Found	Cald	Found	
D-1	$C_{19}H_{20}N_4O_3S$	384	70	59.37	59.2	5.2	5.1	14.5	14.3	8.33	8.2	-	-	0.9
D-2	$C_{27}H_{20}N_4O_3S$	480	75	67.5	67.5	4.16	4.1	11.66	11.5	6.66	6.5	-	-	1.1
D-3	$C_{23}H_{20}N_4O_3S$	432	70	63.88	63.6	4.62	4.5	12.96	12.8	7.4	7.3	-	-	1.1
D-4	$C_{21}H_{14}Cl_2N_4O_3S$	473	75	53.2	53.2	2.95	2.85	11.83	11.7	6.76	6.6	15.01	14.9	0.95
D-5	$C_{21}H_{13}Cl_2N_5O_5S$	518	70	48.64	48.5	2.5	2.4	13.51	13.4	6.17	6.1	13.7	13.6	1.1

Table : II IR Spectral data of chelating dyes containing 8-hydroxyquinoline

Dye No.	Azo Group	Aromatic Nucleous	-OH Group	Alkane	8-hydroxy quinoline
D-1	1600 1632	3030 (w) 1500, 1600(s)	3200-2600 (b)	2930 1370	1630 1575 1500
D-2	1605 1630	3032 1500, 1600(s)	3400-2000	2930(w) 2370(w)	1630 1577 1505
D-3	1603	3031 1500, 1610(s)	3400-2600	1980(w) 1370	1630 1575 1500
D-4	1600	3031 1500, 1610(s)	3400-2600	1930 1370	1630 1575 1500
D-5	1600	3030 1600, 1170(s)	3400-2600	1930 1370	1630 1580 1510

Table: III Visible spectral characteristics of chelating azo Dyes containing 8-hydroxyquinoline

Dye NO.	λ maxnm	loge
D-1	480	0.588
D-2	457	0.919
D-3	400	0.515
D-4	385	7.41
D-5	482	0.437

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