



## Synthesis, characterization and spectral study of chelating azo dyes containing salicylic acid ligand

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### ABSTRACT

We have synthesized five sulfanilamide derivatives. Then all sulfanilamide derivatives [1a-1e] diazotization with NaNO<sub>2</sub> and HCl at 0-5°C. Then the azo dye was synthesized by the coupling of diazonium salt of sulfanilamide derivatives (2a-2e) with Salicylic Acid ligand. After the synthesis, compounds were characterized by chemical as well as instrumental methods, like Melting point, elemental analysis, UV-visible spectroscopy and IR spectral studies.

**Keywords:** Sulfanilamide derivatives, Salicylic acid ligand, Azo dye, UV-visible and 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 ring 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 [1-5] and studied dyeing properties [6-7]. Azo dyes have been widely used in various fields and technologies like textiles, leather, plastics, paper, laser liquid crystalline displays and ink jet printers [8-10]. They are also used in food [11], drug, cosmetic and photochemical production [12]. It was thought interesting to explore the field of [3a-3e] acid azo dyes were prepared based on sulfanilamide derivatives and Salicylic Acid 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<sub>2</sub>, 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.

Where,

R<sub>1</sub>

Ethyl

Phenyl

Phenyl

2,4-dichloro phenyl

2,6-dichloro-4-nitro phenyl

R<sub>2</sub>

Ethyl

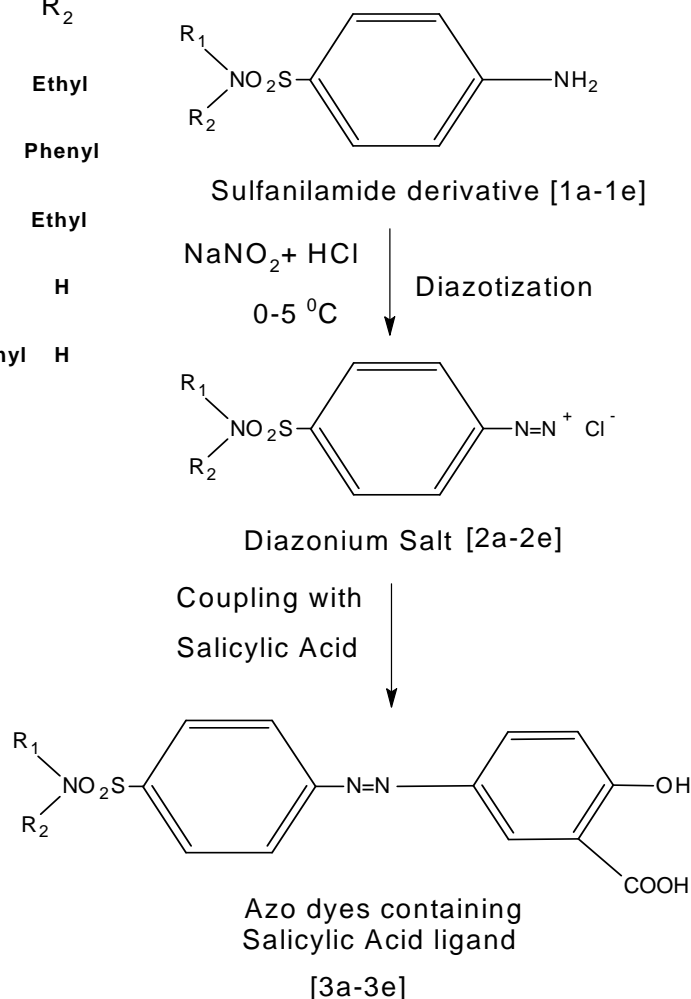
Phenyl

Ethyl

H

H

### Reaction Scheme-I



#### Synthesis of acid azo dyes :

Diazotization of different sulfanilamide derivatives [1a-1e] was dissolved in HCl with stirring and the solution was cooled to 0-5°C in an ice bath. A solution of sodium nitrite 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).

Salicylic acid 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 [2a-2e] of was added slowly so that temperature 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 [3a-3e] acid azo dyes were prepared.

### RESULTS AND DISCUSSION

#### Physical properties of dyes :

All five azo dyes the physical data and the elemental of C, H, N confirmed by Table: I. The yield of the dye was 60% to 70%.

**Infrared Spectra:**

The observed bands in the IR spectra for each dye are shown in Table-II. Examination of the IR spectra of all the azo dyes reveals 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 comprise a broad band extended from 2500  $\text{cm}^{-1}$  to 3200  $\text{cm}^{-1}$  mainly raised due to -OH group. The bands at 1500, 1200 and 1050  $\text{cm}^{-1}$  appeared in the double bond region are due to aromatic stretching vibrations. The strong bands 1605 and 1632  $\text{cm}^{-1}$  appeared in the spectra are considered due to presence of azo (-N=N) group. Apart from these a weak band was observed around 2930  $\text{cm}^{-1}$  and 2932  $\text{cm}^{-1}$  which are attributed to the -CH<sub>2</sub> stretching vibrations. In the spectra of Salicylic Acid containing dyes, bands around 1680 and 1685  $\text{cm}^{-1}$  of due to Salicylic Acid moiety are observed [9].

**Table I Characterization of Dyes containing Salicylic Acid 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	
<b>3a</b>	C <sub>17</sub> H <sub>19</sub> N <sub>3</sub> O <sub>5</sub> S	377	60	54.11	54.0	5.03	5.0	11.14	11.0	8.48	8.4	-	-	1.0
<b>3b</b>	C <sub>25</sub> H <sub>19</sub> N <sub>3</sub> O <sub>5</sub> S	473	70	63.4	63.2	4.01	3.9	8.88	8.8	6.76	6.6	-	-	1.1
<b>3c</b>	C <sub>21</sub> H <sub>19</sub> N <sub>3</sub> O <sub>5</sub> S	425	70	59.29	59.33	4.47	4.4	9.88	9.8	7.53	7.4	-	-	1.1
<b>3d</b>	C <sub>19</sub> H <sub>13</sub> Cl <sub>2</sub> N <sub>3</sub> O <sub>5</sub> S	466	70	48.92	48.8	2.78	2.7	9.01	8.9	6.86	6.7	15.23	15.1	0.98
<b>3e</b>	C <sub>19</sub> H <sub>12</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>7</sub> S	511	66	44.62	44.6	2.34	2.3	10.96	10.8	6.26	6.2	13.89	13.8	1.0

**Table II IR Spectral data of chelating dyes containing Salicylic acid**

Dye No.	Azo Group	Aromatic Nucleus	-OH Group	Alkane	8-hydroxy quinoline
<b>3a</b>	1600 1631	3030 1500, 1600	3200-2500 (b)	2930 1370	1680
<b>3b</b>	1610 1630	3032 1500, 1600	3200-2500 (b)	2930 2370	1681
<b>3c</b>	1600 1630	3031 1500, 1610	3200-2500	2932 1370	1683
<b>3d</b>	1605 1632	3031 1500, 1610	3200-2500	2930 1370	1680
<b>3e</b>	1605 1632	3030 1500, 1600	3200-2500	2930 1370	1685

**CONCLUSION**

The azo dyes were successfully synthesized by standard methods of diazotization and coupling of diazo solution of sulfanilamide derivatives with Salicylic acid. The results on the elemental analysis and spectral studies of each dye were consistent and hence confirm the predicted structure. The present study prepared acid azo dyes showed wide range of shades. They showed good dyeing performance on wool and nylon fiber and the light fastness values of the acid azo dyes are more consistent.

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