X-ray diffraction Characterization of crystalline structure regent4(4-Sulphophenyl Azo) Pyrogallol

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Abstract. New azo reagent was synthesized by reaction dizonium salt Sulfadiazine with Pyrogallol. This product was characterized by FTIR, UV.Visible spectrophotometer and (XRD). Purity of the dye was checked by thin layer chromatography (TLC) using solvent system (Toluene-Methanol) (2:3). The melting point of the purified dye was measured in an open capillary tube. The results showed this compound have high levels of antibacterial activity.

Key words: Azo reagent, Sulfadiazine

Introduction

Synthetic dyes play an important role in everyday life.\(^{(1)}\) They are widely used not only in textiles but also in paper, plastics, leather, and other fields.\(^{(2)}\) Azo dyes are synthetic azo compounds that contain azo groups. There are more than 3,000 kinds of azo dyes accounting for approximately 80% of all dyes. in the lead washing and dyeing of dye wastewater. Dye wastewater has the typical characteristics of complex composition, high concentration of pollutants, deep colour and poor biodegradability.\(^{(3,4)}\) It has become an important source of environmental pollution.\(^{(5,6)}\) Azo reagents are the majority use pigment also explanation designed for further than 50 % of whole pigment.\(^{(7,8)}\) Approximately. Azo pigments are characterized by the attendance of azo (-N = N-) in their arrangement, associated by two aromatic systems, separate or the same. Due to its specific physical and substance property and genetic behavior, it has establish wide function in the industries of pharmaceuticals, cosmetics, food and dyeing, textile and analytical chemistry. However, the most common area of utility is the coloring function.\(^{(9)}\) They are the most studied class of organic dyes with major application as dyeing pigment.\(^{(10)}\) In addition studies have also shown that this class of organic compounds showed various medicinal activities such as ant diabetics\(^{(11)}\) antiseptics\(^{(12)}\), antineoplastics\(^{(13)}\) antibacterial\(^{(14, 15)}\) and antitumor\(^{(16)}\). Many of the azo dyes serve as chromogenic...
reagents used for the determination of several metal ions \(^{(17)}\). They have also been reported to show good inhibitory capacity for the corrosion of many materials in both acidic and basic media \(^{(13)}\).

2- Devices and Materials

The chemicals used are of purity. The infrared spectrum of the compound is recorded on a Shimadzu FTIR 8400s spectrophotometer. The UV-vis spectrophotometers are recorded on a Shimadzu UV-1650 PC. X-ray diffract meter XRD-6000 Shimadzu, Japan.

Synthesis of the reagent 4(4-sulphophenylazo)pyrogallol \(^{(18,19)}\)

The above reagent was prepared based on the traditional azo method. 2.72 g (0.01 mol) of 4-sulfadiazine was diazotized by adding 10 ml of concentrated hydrochloric acid (37%) and 0.01 mole of NaNO\(_2\) dissolved in 20 mL of deionized water was added to it and the mixture was left for 30 minutes with constant stirring. Then it was added to a solution of 1.26g (0.01 mol) of pyrogallol dissolved in 30 ml ethanol in addition to 100 ml of sodium hydroxide prepared by dissolving (4 grams in 100 ml of ion-free water) drop by drop with constant stirring. Then add 100 ml of deionized water and let the mixture for the next day. Then the sediment was filtered and washed several times with ion-free water and recrystallized with absolute ethanol, then the sediment was dried using an electric furnace for several hours at 60 °C. Then the product percentage was calculated and it was 34% and the melting point was 165-167 C. The detector (4-BPAP) in the two steps of Diazotization and Coupling shown in scheme (1) below:

![Scheme 1: Preparation of Reagent 4- ((4-sulfo-phenol Azo)) pyrogallol (4-SPAP)](image)

**Results and discussion**:

**Characterized of (4-SPAP)**.
Spectral analysis for 4-SPAP

Infrared spectroscopy was used to diagnose the position of beams in the ligand spectrum (4-SPAP), to describe the inter-atom interactions around the position of the beams. He showed two strong beams at cm$^{-1}$ (3853, 3418) due to the overlap between (-OH) and (-NH) beams, as well as showed a beam belonging to the azo group at cm$^{-1}$ (1516). As Figure (1).

![Infrared spectrum](image1)

**Figure (1):** Infrared spectrum in the of KBr for ligand (4-SPAP).

UV-visible spectrum of synthesized 4-SPAP. The azo functional group (-N=N-) usually absorbs at the wavelength range from (350-370) nm. Figure (2) showed two transitions at 418nm and 399nm. Due to its orange colour, these bands may be due to n-$\pi^*$ of azo group and $\pi^*$-$\pi^*$ transition.

![UV-VIS spectrum](image2)

**Figure (2):** The UV-VIS spectral analysis for 4-SPBP

X-ray diffraction spectra

The structural properties of the prepared surfaces representing the size and crystal structure of the ligand (4-SPAP) in its solid state were studied using X-ray diffraction (XRD) spectra. For this, a single-wavelength light (1.5104A $^\circ$) of Cu-Kα within the angular range was used (0-80) degrees.
Figure (3), Table (1) shows the (XRD) spectrum of the appearance of four peaks of the detector (4-SPBA) and the locations of these peaks are \((2\theta = 13.1713), (2\theta = 15.131), (2\theta = 16.4288), (2\theta = 25.004)\) at the distance. The computed interlayer (d-spacing) corresponding to \((6.72199)\) and \((5.8555), (5.39577), (3.56135)\), and these peaks are wide and the reason is that there are peaks that get wider when the size of the crystals is very small. In the spectrum of X-ray diffraction it refers to the less homogeneous crystalline structures in nature, because the size of the mentioned nanocrystal is rather small \(^{21-22}\).

![Figure (3): XRD spectrum for the reagent (4-SPAP)](image)

Table (1) represents the d-spacing values, diffraction angles, and relative intensity, and average crystal size of the 4-SPBP regent.

| \(2\theta\)   | FWHM | Intensity (I/I\(_0\)) | d-spacing(Å) | Particle size(nm) | Average size (nm) |
|--------------|------|------------------------|--------------|-------------------|-------------------|
| 13.1713      | 0.3936 | 66.69                  | 6.72199      | 3.4305            | 3.02125           |
| 15.131       | 0.492  | 95.34                  | 5.8555       | 2.7208            |                   |
| 16.4288      | 0.3936 | 100                    | 5.39577      | 3.3793            |                   |
| 25.004       | 0.492  | 74.64                  | 3.56135      | 2.5544            |                   |

### Conclusion

A study and preparation of 4-SPAP was obtained from the interaction of sulfadiazine reactive salts of diazonium with pyrogallol. 4-SPAP was characterized by a spectrophotometer (FTIR) and visible ultraviolet light. Xrd explains that these peaks are wide and the reason is that there are peaks that get wider when the size of the crystals is very small.
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