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RESEARCH ARTICLE

THE STUDY PREPARATION AND IDENTIFICATION NEW DYE OF AZO DYES.

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Key words:-

4-aminobenzenesulfonic acid, α -naphthol, azo dye.

Abstract

A new azo dye prepared by reaction between 4-aminobenzenesulfonic acid and α -naphthol in several steps. UV spectroscopy was used to determine the maximum wavelength (300 nm) and compare the absorption spectra between primary material and result (new azo dye). Melting point was measured for primary materials and prepared azo dye which was the highest. The FT.IR spectroscopy was measured for new azo dye and studied of the bending and stretching bonds where the spectrum conformed to the suggested form of dye.

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Introduction:-

Azo compounds are an important class of organic colorants which consist of at least a conjugated chromophore azo ($-N=N-$) group in association with one or more aromatic or heterocyclic system^(1,2). They are capable of providing high intensity color and have reasonably good technical properties, including light and weather fastness and resistance to solvents and water⁽³⁾. It has been known for many years that azo compounds are the most widely used class of dyes due to their versatility in various fields such as the dyeing of textile fiber, the coloring of different materials, coloured plastics, biological-medical studies and advanced applications in organic synthesis⁽⁴⁻¹¹⁾. This paper reports the synthesis, Identification of the new azo dye.

Experimental Part:-

Synthesis of Azo Dye:-

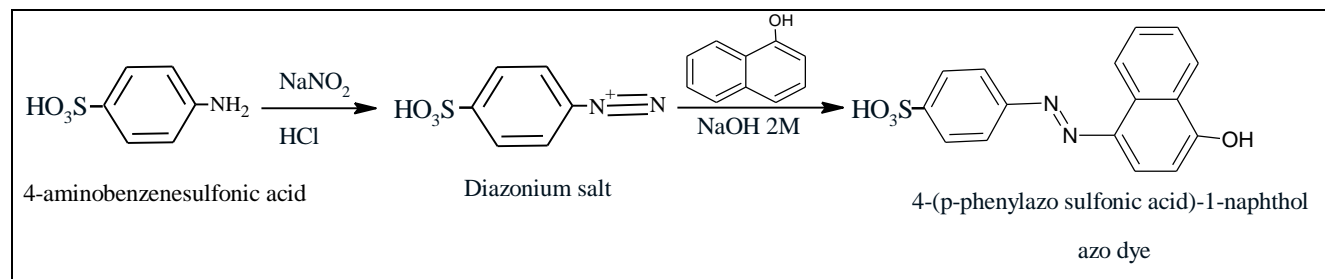
1. Formation of the diazonium salt in test tube (A) add 10 mL of HCl (1M) to test tube contain a 0.25 mg 4-aminobenzenesulfonic acid, cool the solution in an ice-water bath at 5 C° and stirring.
2. In test tube (B) prepared a solution of 2g of sodium nitrite in 10 mL of water, cool the solution in an ice-water bath at 5 C° and stirring.
3. In test tube (C) prepared a solution of 0.5 g of α -naphthol in 2 mL of NaOH (2M), cool the solution in an ice-water bath at 5 C° and stir.
4. By dropper pipette added the solution in test tube (B) to test tube (A) and mixed the two solution to gather.
5. added 2 ml of diazonium salt solution that prepared in 4 step to test tube (C) and stirring with spatula through the add. at 5 C° for 5 minutes until the solution color changes to brown.
6. Melting point was measured for primary materials the m.p (238 C°) and prepared azo dye the m.p (decompose 147 C°)
7. UV spectroscopy was used to determine the maximum wavelength (300 nm) and compare the absorption spectra between primary material and result (new azo dye).
8. The FT.IR spectroscopy was measured for primary material and result (new azo dye).

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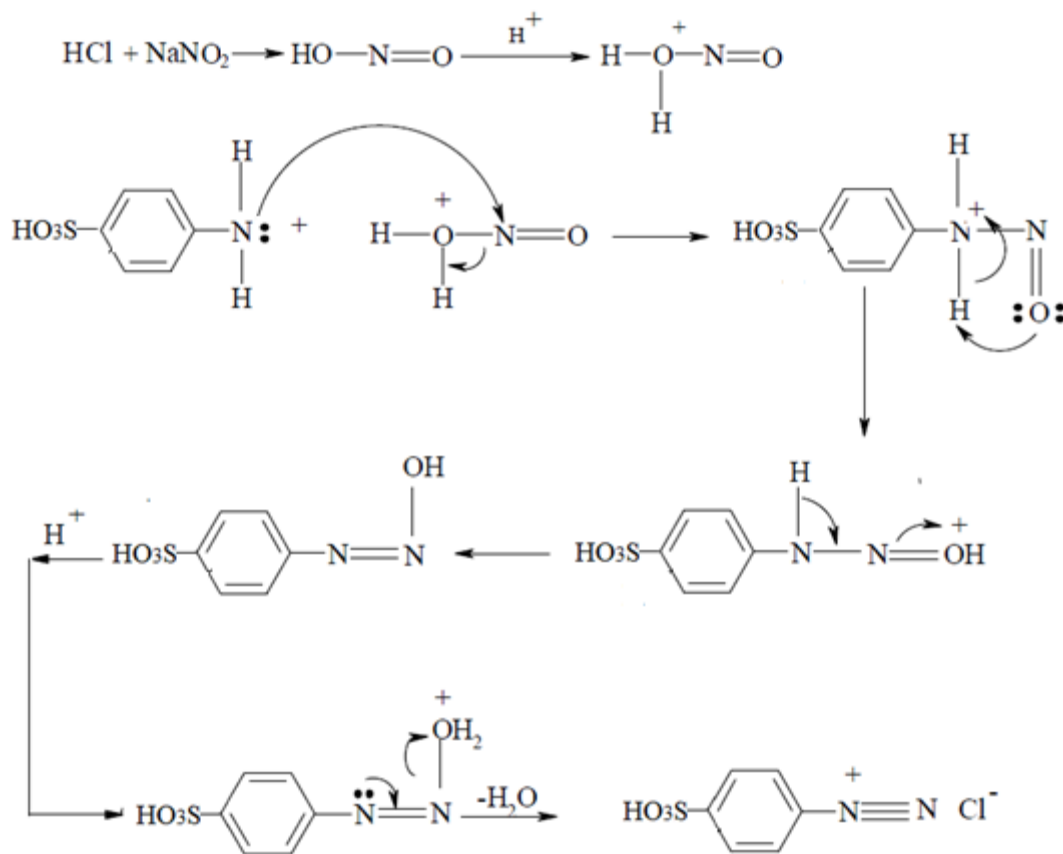
Results and Discussion:-

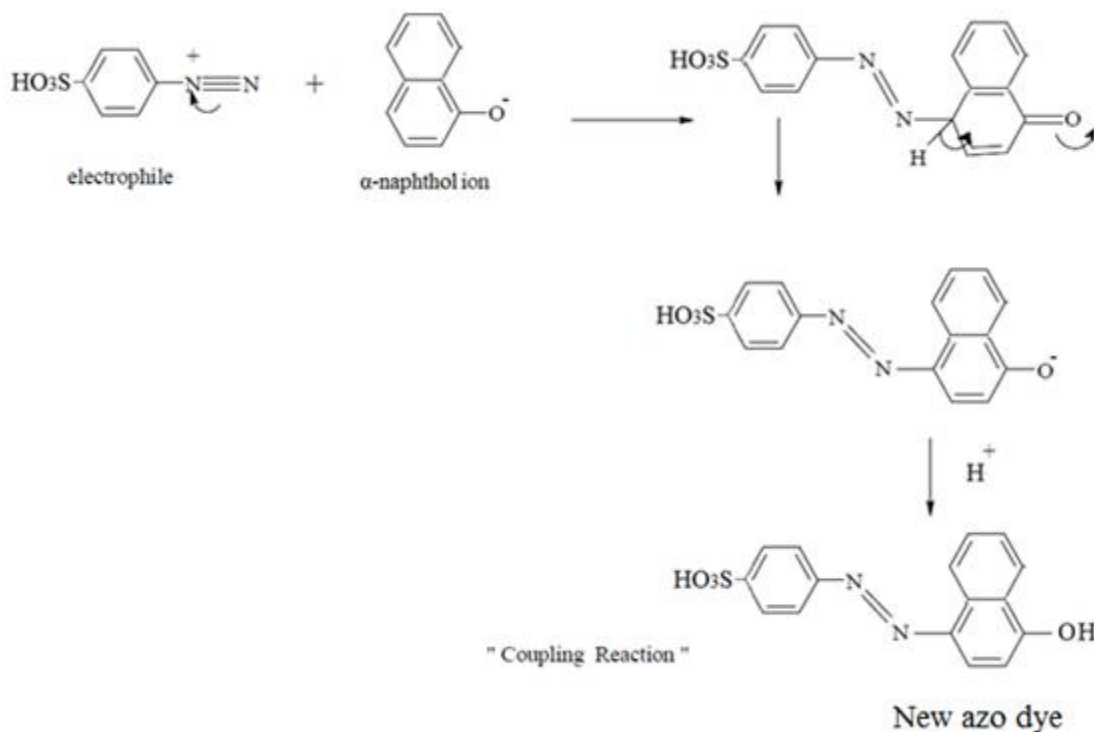
In this research used organic primary amine to synthesized azo dye. Azo dyes, which were developed, are one of the most common dye materials. They contain the basic structure of Ar-N=N-Ar . Their color is due to the high level of conjugation that extends through N-N double bond to the aryl unit. Azo dyes are synthesized via the following reaction. A aromatic primary amine Ar-NH_2 (4-aminobenzenesulfonic acid) is converted to a diazonium salt and this is reacted with another aryl unit (α -naphthol) is shown in scheme [1].



Scheme [1]

The aromatic ring can be substituted with different functional groups (auxochromes) and these substituents, due to their conjugation with the azo system, will affect the color of the dye. Azo dyes are synthesized from reaction between a primary amine (4-aminobenzenesulfonic acid) is converted to a diazonium salt and another aryl unit (α -naphthol) according to the following mechanism is shown in scheme [2].

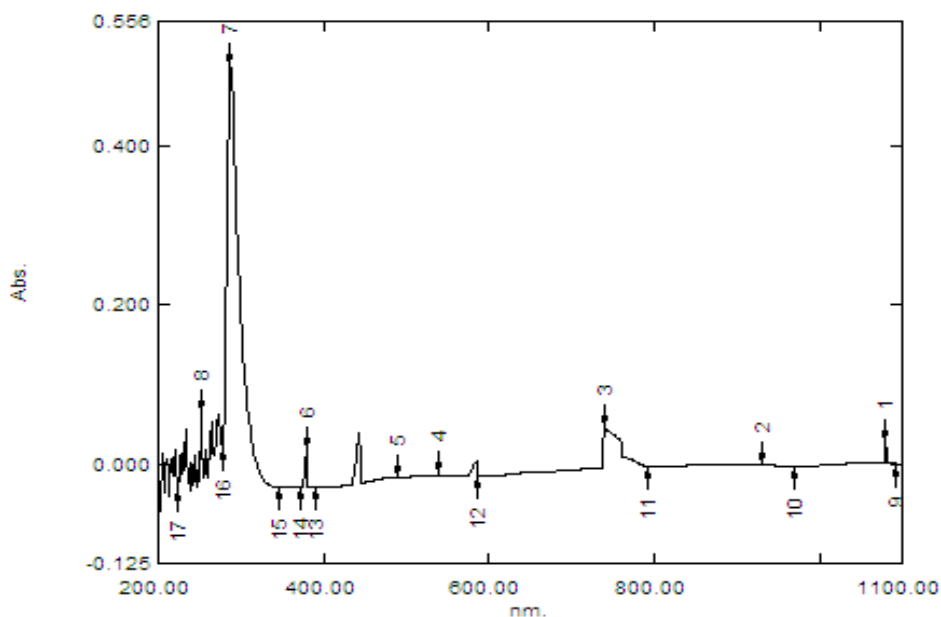




Scheme 2:-

UV-VIS spectroscopy:-

In our present work, we have analyzed our material by UV-Vis spectroscopy Model,1500pc. In general UV spectrum of PABS (4-aminobenzenesulfonic acid) consists of two major absorption peaks. One at 280–300 nm is assigned to the π - π^* transition which is related to the extent of conjugation length. The second absorption band for excited transition was observed at 501–565 nm. UV spectroscopy was used to determine the and compare the absorption spectra between primary material and result (new azo dye) at concentration 0.001M and wavelength from 200-1100 nm as in the Fig.[1, 2].

**Fig.1:-** The relation between the absorption and wave length for 4-amino benzenesulfonic acid.

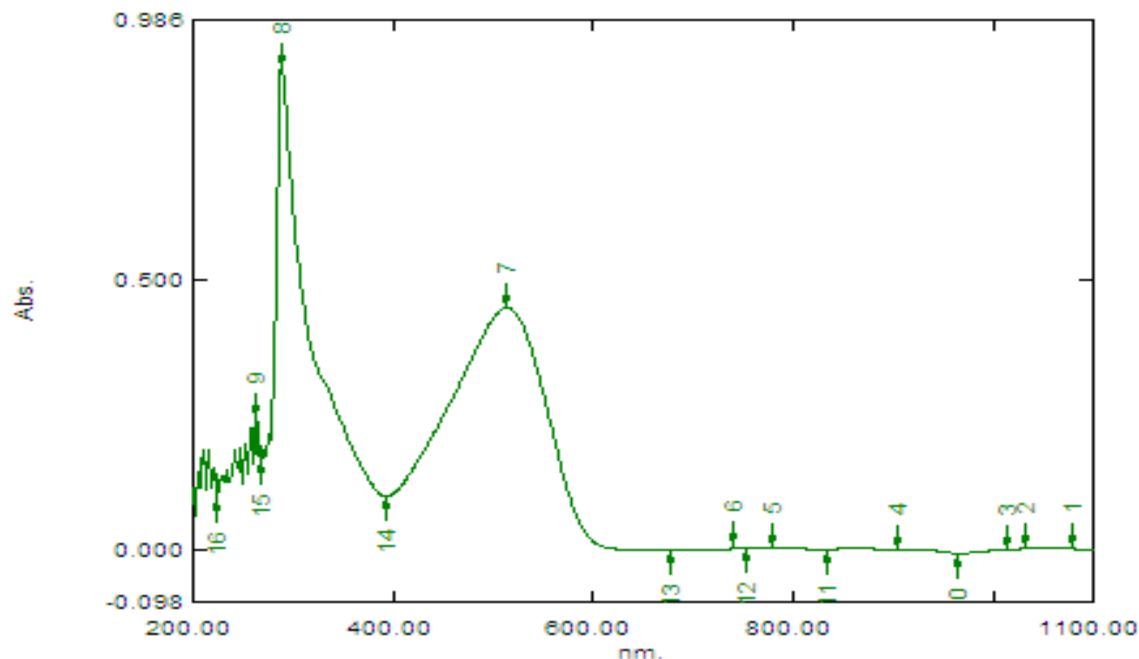


Fig. 2:- The relation between the absorption and wave length for prepared new azo dye.

Transition from $\sigma - \sigma^*$ indicates the presence of a group **C-H** at a wavelength 125 nm.

Transition from $\pi - \pi^*$ indicates the presence of a double bond **N=N** group at a wavelength 200-700 nm.

Transition from $n - \sigma^*$ indicates the presence of a (**O, N, S**) groups at a wavelength least of 260 nm.

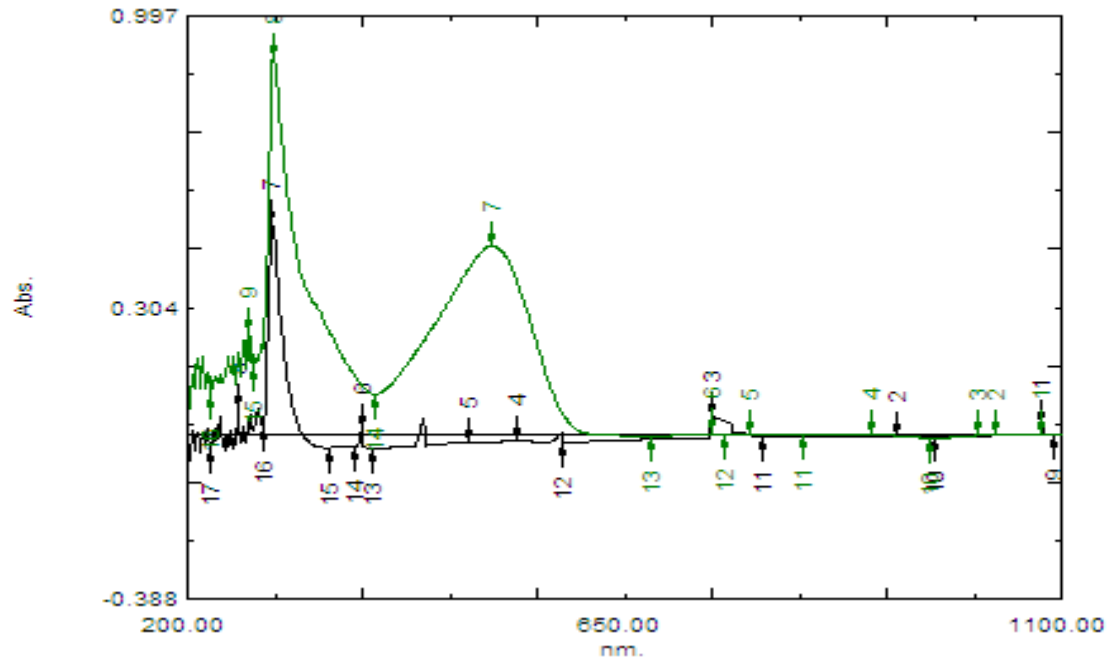


Fig. 3:- The relation between the absorption and wave length for 4-amino benzenesulfonic acid and prepared new azo dye to gather.

FT-IR spectroscopy:-

FT-IR spectra were recorded on FT-IR 8400S, Shimadzu-Spectrophotometer and using KBr discs to identify the chemical structure of the (4-amino benzenesulfonic acid) and new azo dye as in the Fig. [4, 5]. The synthesis azo

dye was identified with FT.IR, Fig. [5] showed disappearance of the two absorption bands at 3383 cm^{-1} and 3307 cm^{-1} were due to the stretching vibrations of $(-\text{NH}_2)$ group of 4-aminobenzenesulfonic acid Fig. [4] appearance of the azo dye showed of absorption band at 3437 cm^{-1} of the stretching vibration of the $(-\text{OH})$ groups⁽¹²⁾. FT.IR spectrum also showed the appearance of absorption band at 1450 cm^{-1} for $(\text{N}=\text{N})$ group⁽¹³⁾. The stretching vibration of the $(\text{C}-\text{H})$ aromatic appear at 2968 cm^{-1} , The absorption bands at 879 cm^{-1} were due to the bending vibration of $(\text{C}-\text{H})$ aromatic out of plane⁽¹³⁾. The other data of functional groups were shown in the following Table [1].

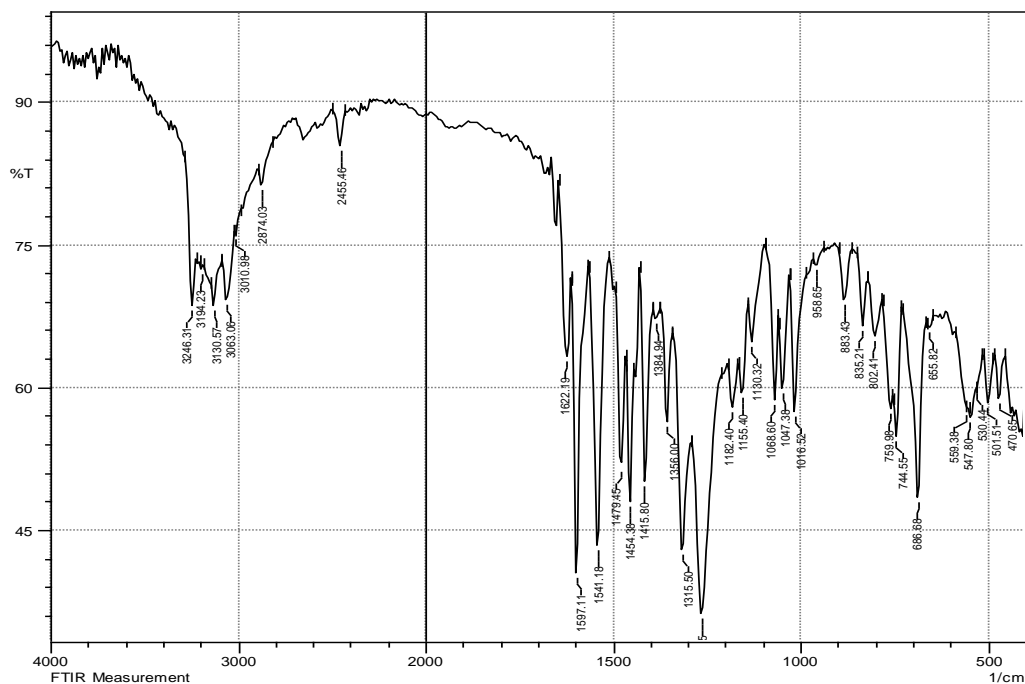


Fig 4:- F.T.I.R Spectrum of 4-aminobenzenesulfonic acid.

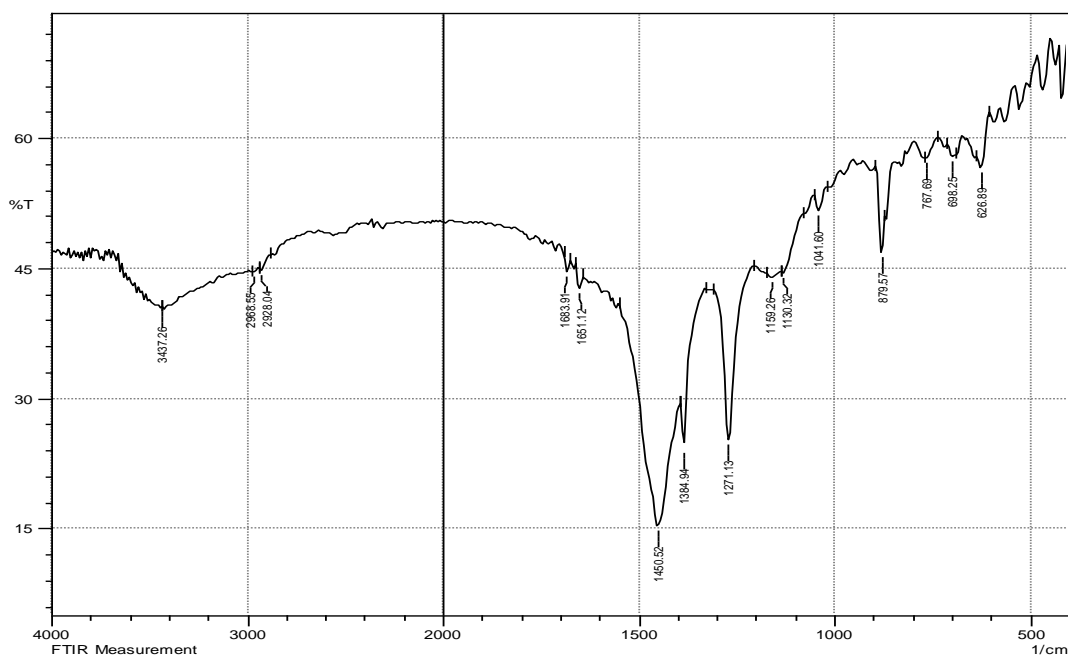


Fig. 5:- F.T.I.R Spectrum of azo dye.

Table 1:- FT.IR data of new azo dye and 4-aminobenzenesulfonic acid [A].

COMP. NO.	ν (O-H) cm^{-1}	Amine ν (N-H) cm^{-1}	Aromatic ν (C-H) cm^{-1}	ν (N=N) cm^{-1}	Aromatic ν (C=C) cm^{-1}	ν (S=O) cm^{-1}	ν (C-S) cm^{-1}	Bend ν (N-H) cm^{-1}
[A]	—	-3246 3194	3063	—	1454-1479	1315	1275	-1597 1541
Azo dye	3437	—	2968	1450	1651-1683	1384	1271	—

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