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SYNTHESIS OF NEW DIAZO DYE DERIVED FROM 4,4′-DIAMINO BENZANILIDE WITH CATECHOL AND STUDYING OF ITS PHYSICAL, SPECTRAL AND BIOLOGICAL PROPERTIES

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ABSTRACT

A new di-azo dye was prepared based on the reaction 4`,4-diaminobenzenilide with catechol, and the resulting compound was characterized using UV-Vis, FT-IR, Mass spectroscopy techniques. Also, the biological activity of the prepared compound was also studied and found to be of noteworthy biological activity which promises with amazing results for the prepared compound in various pharmacological applications.

KEYWORDS: di-azo, 4`, 4-diaminobenzenilide, catechol, dyes, biological activity.

1. INTRODUCTION

In the last years, more and more substances of additive and colorant type have been proved their adversity against the human health. Additionally, on the list of allergens inducers of the contact dermatitis in industrial field are continually adding new chemical compounds.[1,2] Despite their existence for over 100 years, direct azo dyes are still one of the most important class of synthetic dyes due to their wide fields of application, ranging from the textile industry to medicine, pharmaceutical industry, cosmetics, food, etc.[3,4] However, a great number of some usual azo direct dyes were prepared from some precursors (especially aromatic diamines) which proved to be genotoxic.[5–7] It was found that these dyes can be either direct acting mutagens or pro mutagens.[8, 9] Hence, the synthesis and the use of such compounds presents a potential occupational and environmental risk, and the search for viable alternative dyes (and precursors) and preparation methods continue to be an important research problem.[10–14] Our studies focus on the possibility of developing a new series of direct azo dyes with good dye and application properties, using harmless precursors. Moreover, a biological study was conducted, in order to evaluate the possibility of using this new diazo dye in the pharmaceutical industry.

2. Experimental

2.1. Materials And Apparatus

All chemicals used in this work were purchased from BDH, Aldrich and Merck companies and were used without further purification.

FT-IR spectra (ν, cm^{-1}) were recorded on a JASCO Spectrum (FT–IR 4100) spectrometer using KBr pellets. UV-Vis spectroscopy were measured by using (Jasco-V630-UV-Vis) at the wavelength range (200–800 nm), using match quartz cells (1 cm) and DMSO as a solvent.

2.2. Synthesis of D1

In the first beaker, add 0.0025 mol (0.5681 gr) of 4`,4-diaminobenzenilide to 0.005 mol of hydrochloric acid solution (37%), the mixture was stirred until full dissolution and cooled to (0-5 ℃). In the second beaker, 0.005 mol of sodium nitrite 98% was added to 15 ml of distilled water, stirring until completely dissolved. We cooled the solution until (0-5 ℃), and then the second beaker is added to the first beaker within an hour while the temperature fixed at (0-5 °C). where the reaction takes place according to the following equation fig.1.:

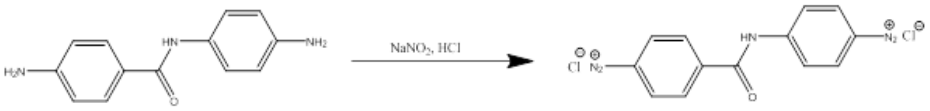

Fig.1: Preparation of Diazo Salt.

In the next step, a mixture (0.005 M of catechol and 0.005 of NaOH) was added to the previous reaction mixture, the following chemical equation describing the reaction fig.2.:

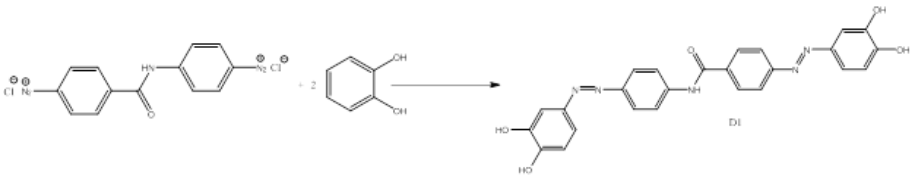

Fig. 2: Preparation of D1.

Finally, the precipitate was filtered and left to dry giving a brown precipitate. The yield of the product was found to be 84.53%, with melting point higher than (360°C).

3. RESULTS AND DISCUSSION

3.1. UV-Vis measurements

D1 compound has been characterized using UV-Vis technique in the DMSO solvent and wave length range (190-600 nm). Fig.3. demonstrate the obtained spectrum.

As it can be seen from the spectrum there are tow important peaks at (197, 462 nm) belongs to the electronic transition (π-π*, n-π*) in the compound.

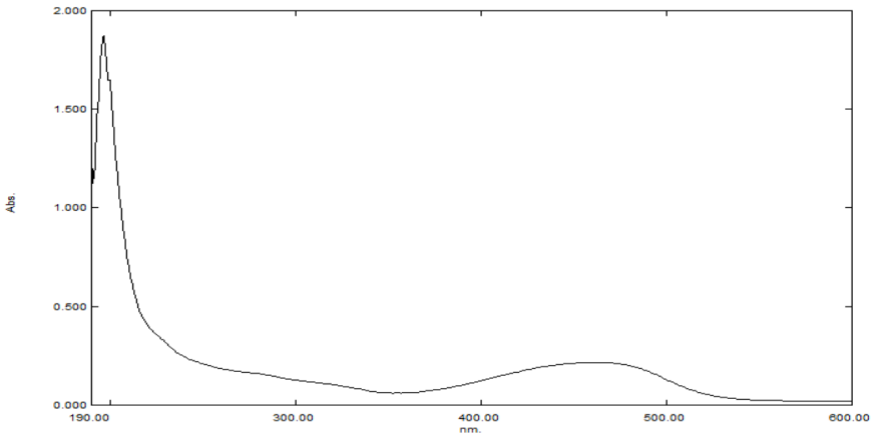

Fig. 3: UV-Vis Spectrum of D1.

3.2. FT-IR characterization

3.2.1. FT-IR of L ligand and its complexes

The FT-IR spectrum of D1 presented in fig.4. Where the IR spectrum showed a remarkable absorption bands at (3444, 3419, 1651, 1570 cm^{-1}) returns for (O-H, N-H, C=O, N=N) stretching.

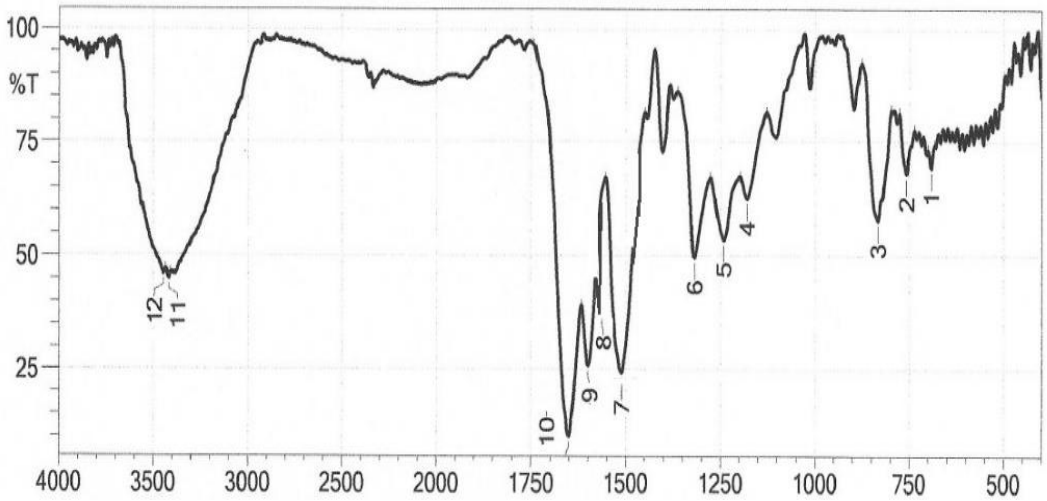

Fig. 4: FT-IR Spectrum of D1.

3.3. Mass spectroscopy characterization

The compound D1 with the formula $C_{25}H_{19}N_5O_5$ (Mw = 469 g/mol) was analyzed using a mass spectrometer to confirm the purity of the resulting compound and determine its molecular weight. Fig.5. shows the mass spectrum of it.

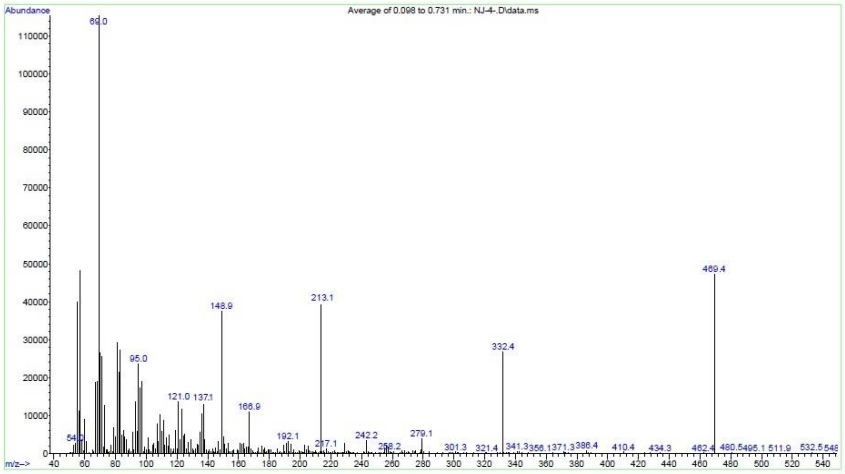

Fig. 5: Mass Spectrum of D1.

We notice a signal at 469 m/z, which proves the purity of the compound. There are also several important fragments at (332,242,213,147,137) m/z. figures (6,7) show the molecular weight and the proposed mechanism of some fragments.

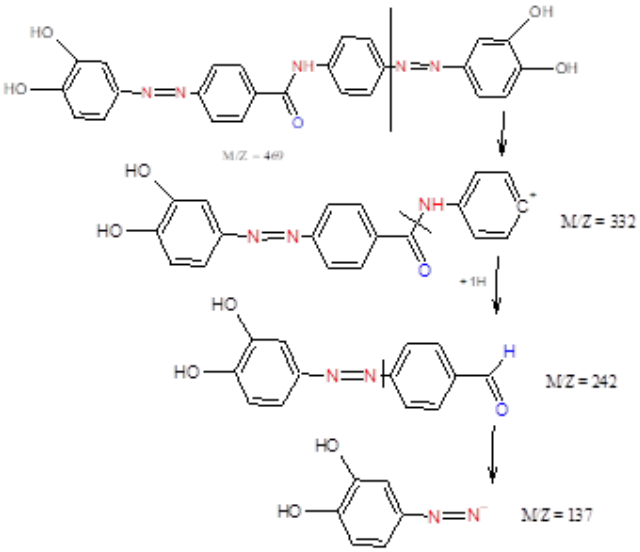

Fig. 6: Proposed Mechanism for D1 Decomposition.

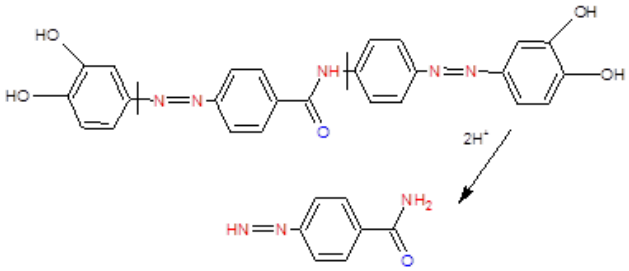

Fig. 7: Proposed Mechanism For D1 Decomposition.

3.4. Antibacterial Activity Study

The antibacterial efficacy of the prepared compounds was tested against Escherichia coli, and Staphylococcus aureus bacteria comparing with Gentamicine (as a reference). Two different concentrations (50 and 100 µg/ml) of the compounds and Gentamicine have been selected for antibacterial assay. In our research, we chose to study E. coli and S. aureus bacteria, because of their wide spread in society so they affect in the daily life of humans, as Escherichia is a common bacterium found in the intestines of humans and warm-blooded animals. It is often used as an indicator for fecal contamination in water and soil.[15] Pathogenic strains of E. coli are often transmitted through contaminated food or water[16], and can be particularly dangerous for young children, elderly individuals, and those with weakened immune systems. This bacterium can cause a range of infections, including intestinal, skin, wound sepsis, septicemia, neonatal septicemia, and urinary tract infections.[17] Studies have shown that some non-steroidal pain relievers, such as diclofenac sodium, can play an inhibitory role in the growth of some bacteria, whether negative or positive, in addition to using it as an anti-inflammatory.[18,19] Escherichia coli is also commonly used in scientific research, as it is easy to grow and manipulate in the laboratory. It has been used as a model organism for studying various biological processes, and has contributed to many important discoveries in microbiology and genetics.[20] While a S. aureus is a major bacterial human pathogen that causes a wide variety of clinical manifestations. Infections are common both in community-acquired as well as hospital-acquired settings and treatment remains challenging to manage due to the emergence of multi-drug resistant strains such as MRSA (Methicillin-Resistant Staphylococcus aureus)[21] S. aureus is found in the environment and is also found in normal human flora, located on the skin and mucous membranes (most often the nasal area) of most healthy individuals. S. aureus does not normally cause infection on healthy skin; however, if it is allowed to enter the bloodstream or internal tissues, these bacteria may cause a variety of potentially serious infections.[22] Transmission is typically from direct contact. However, some infections involve other transmission methods.[23] The results are arranged in the table (1) and presented graphically in the bar graph (fig 8).

Table 1: Biological Test Results of the E.coli and S. Aureus.

	Escherichia coli		Staphylococcus aureus	
	50 (µg/mL)	**100 (µg/mL)**	**50 (µg/mL)**	**100 (µg/mL)**
(D1)	11	13	9	15

Gentamicine	31	32	32	33
DMSO	0	0	0	0

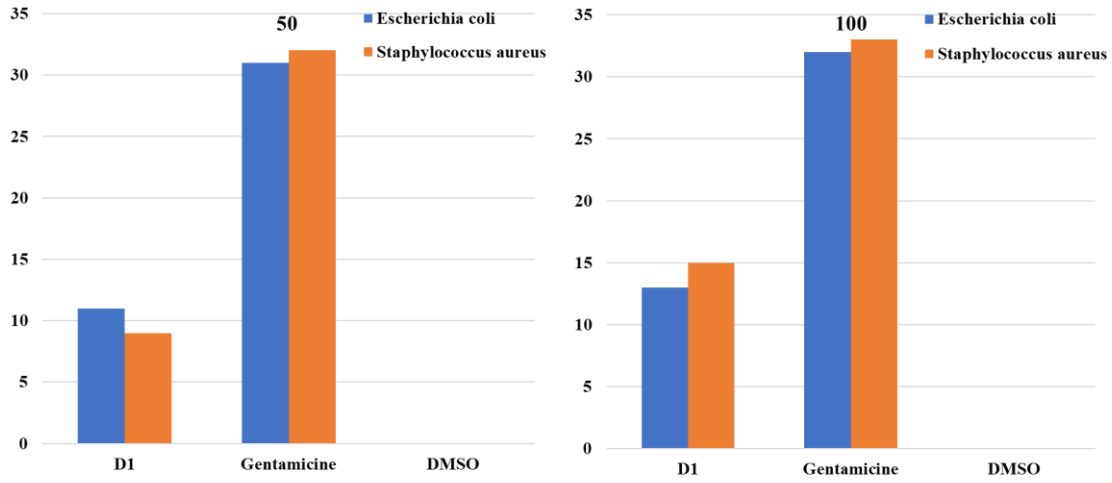

Fig. 8: The graphical presentation of the antibacterial activity against tested bacteria by the D1 at (50µg/mL, A) and (100µg/mL, B).

It is worth noting that the prepared compound has a biological effectiveness.

4. CONCLUSION

In summary, a new di-azo dye was prepared based on the reaction 4`,4-diaminobenzenilide with catechol, and the resulting compound was characterized using UV-Vis, FT-IR, Mass spectroscopy techniques. Also, the biological activity of the prepared compound was also studied and found to be of noteworthy biological activity which promises with amazing results for the prepared compound in various pharmacological applications.

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