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Preparation, spectroscopic characterization and biological activity of a new azo dye ligand

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Abstract: A new azodye ligand was synthesized by the reaction of 2-hydroxyacetophenone and 2-aminobenzoic acid. The prepared ligand was characterized by using CHN elemental analysis, infrared, proton nuclear magnetic resonance and mass spectroscopies. The CHN elemental analysis data showed a good agreement between experimental and theoretical values. The infrared spectral results displayed the main groups of the ligand which are –C=O, -OH, -N=N-, -COOH beside the phenyl rings. The proton nuclear magnetic resonance spectral results exhibited all the signals which belong to the principle groups of the azo dye ligand. The mass spectral data showed the base peak which confirms the original molecular weight of the synthesized ligand. The biological activity of the 2-hydroxyacetophenone, 2-aminobenzoic acid and azodye ligand was screened on some pathogenic bacteria.

Key words: 2-hydroxyacetophenone, 2-aminobenzoic acid, Azodye ligand

Introduction: Acetophenone compound is a bidentate ligand and it has a good ability to form several complexes with transition and non transition metal ions. [(1)] It used as polymerization catalyst for the manufacture of olefins, intermediate for pharmaceuticals and as a drug to induce sleep.[(2)] Chelation behavior of 2-hydroxyacetophenone N(4)- disubstituted thiosemicarbazones Mn(IV) complexes were investigated by X-ray structure, e.p.r and cyclic voltammetric studies.[(3)]

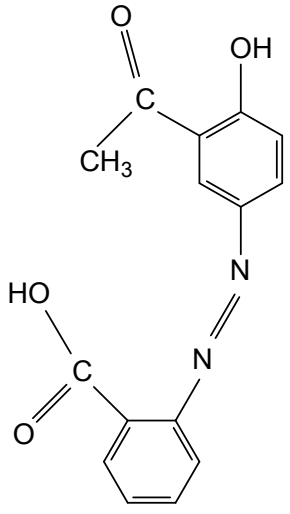
The aim of this paper is to prepare and characterize an azodye derivative of an acetophenone compound, and also to study its biological activity on some pathogenic bacteria.

Materials

All chemicals used in this study [2-aminobenzoic acid, sodium nitrite, sodium hydroxide, 2-hydroxyacetophenone, absolute ethanol, Dimethylsulfoxide and double distilled water] are pure and obtained from BDH or Aldrich.

Synthesis of azodye ligand

The ligand was synthesized by adding slowly with stirring sodium hydroxide (4.00 g); 0.1 mol.) in 100 cm^3 of absolute ethanol at 60 ^oC , 2-aminobenzoic acid (1.37g; 0.01 mol.) in 20 cm^3 of conc hydrochloric acid and sodium nitrite (0.76g) in 20 cm^3 of double distilled water at 0 ^oC to 2-hydroxyacetophenone (1.36 cm^3) in 50 cm^3 of ethanol. The obtained reaction mixture was stirred for 2 hrs. The isolated brown crystals were filtered off, washed by water and recrystallized from ethanol (M.p.;185 $^\circ C$) and finally kept in a desiccator over silica gel.



Azodye ligand ($C_{15}H_{12}N_2O_4$)

Biological assay

The two species of bacteria were streaked on nutrient agar (Oxford, England) plates, so that the streaking covered the surface of the plane, The acetophenone, 2-aminobenzoic acid and azodye compound were applied on the streaked nutrient a gar plate as powder, taking an area not more than 6mm (size of an antibiotic paper disc) and leaving enough distances between them. The plates were then inverted and incubated at 37 ^{0}C for 24 h. The inhibition zones were then increased in millimeters and recorded. The two strains of bacteria (*Bacillus cereus* and *Staphylococcus aureus*) were tested by Mueller-Hinton agar plates. The acetophenone, 2-aminobenzoic acid and azodye compound were applied on the plate by disc paper 6mm, the plates were then inverted and incubated at 37 ^{0}C for 24 h.

Physical Measurements

The prepared azodye ligand was subjected to CHN elemental analyses using 2400 elemental analyzer at Micro-Analytical Center, Cairo University, Giza, Egypt. Infrared spectra were obtained by KBr disc technique by using IFS-25DPUS/IR Spectrometer (Bruker). Electronic absorption spectra were measured in nujol mull using a Perk in-Elmer-Lambda β-Spectrophotometer. The proton NMR spectra were recorded using a Varian Gemini 400 MHz Spectrometer using d^6-DMSO solvent Mass spectrum was carried out using Q1000 EX GC-MS Schimadzu spectrometer at 70 ev and AM energy using a direct insertion probe at temperature 90-100 ^{0}C

Results and Discussion

The reaction of 2-aminobenzoic acid with 2-hydroxyacetophenone yields only one product which is the azodye of the scientific name of 2-[(*E*)-2-(3-acetyl-4-hydroxyphenyl)-1-diazenyl]benzoic acid. The CHN elemental analyses (% C, calc., 63.38, found 63.13, %H, calc., 4.22, found, 4.70, %N, calc., 9.86, found, 9.10) are in a good agreement with each other.

Infrared spectrum of the azodye ligand

Infrared spectral data of the ligand Fig. (1) show several bands at 3379, 2709, 1697, 1490 and 1651cm^{-1} due to -OH, -CH_3, -C=O, -C=N and –N=N- groups respectively. [4-6]

Proton nuclear magnetic resonance spectrum of azodye ligand

^{1}H-nmr spectral data of the ligand was recorded in d^6-DMSO (Figure 2) show the proper signals at 2.253, 6.935 –7.977 and 14.3 ppm, due to CH_3, phenyl protons and OH groups respectively.[7]

Mass fragmentation of the azodye ligand

The mass spectrum of the ligand confirms the proper fragmentations, whereas, the base peak at m/e$^+$= 284 (Fig. 3, scheme 1) is corresponding to the original azodye ligand.

Ultraviolet spectrum of the azodye ligand

The ultraviolet spectral data show bands at 220, 250 and 270 nm (45454, 40000 and 37037 cm^{-1}) corresponding to intraligand transitions, Fig. 4.

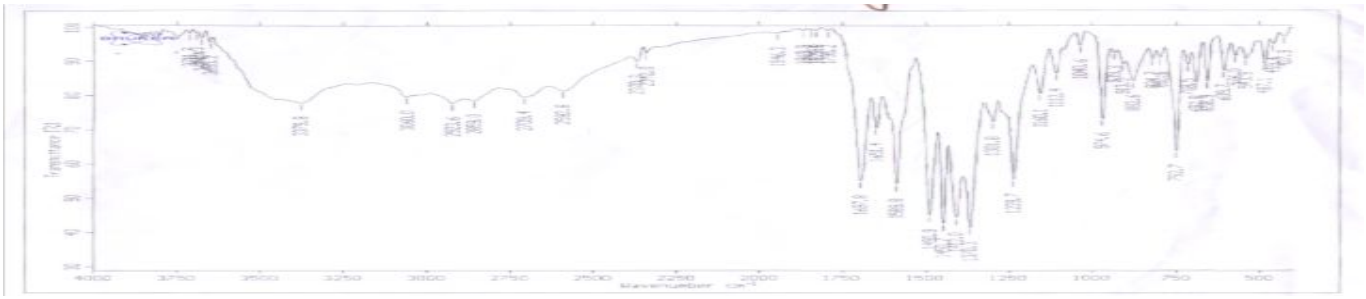


Fig.(1): IR spectrum of the ligand.

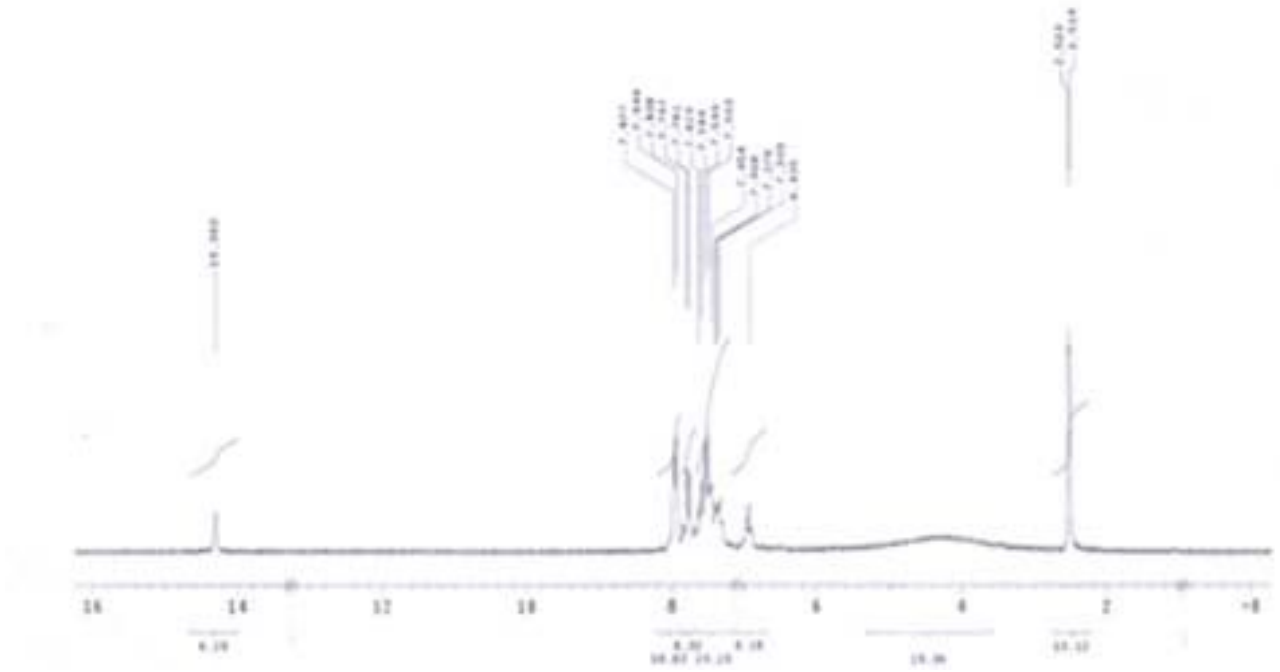


Fig. (2): ^{1}H-NMR spectrum of the ligand.

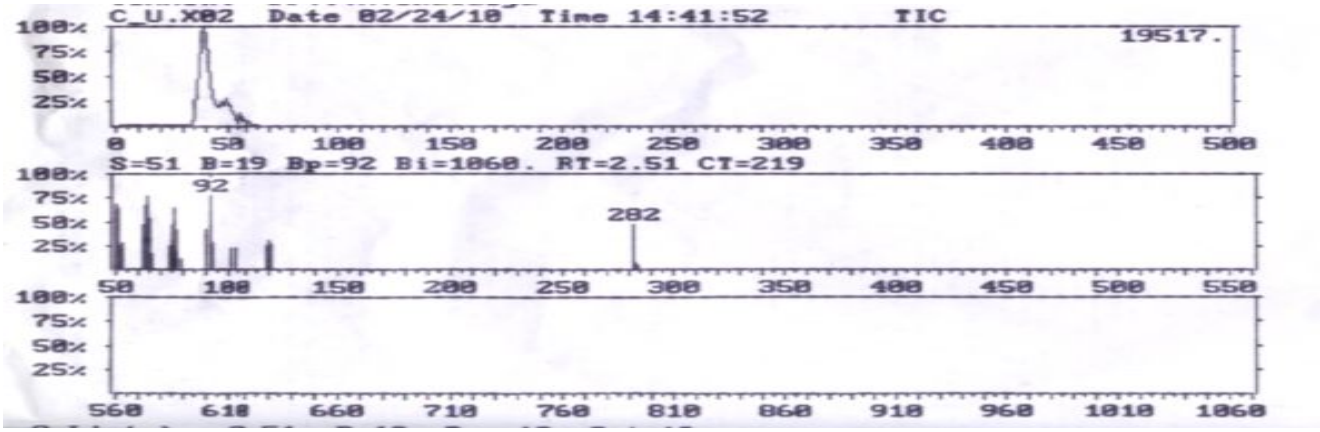


Fig.3: Mass spectrum of compound.

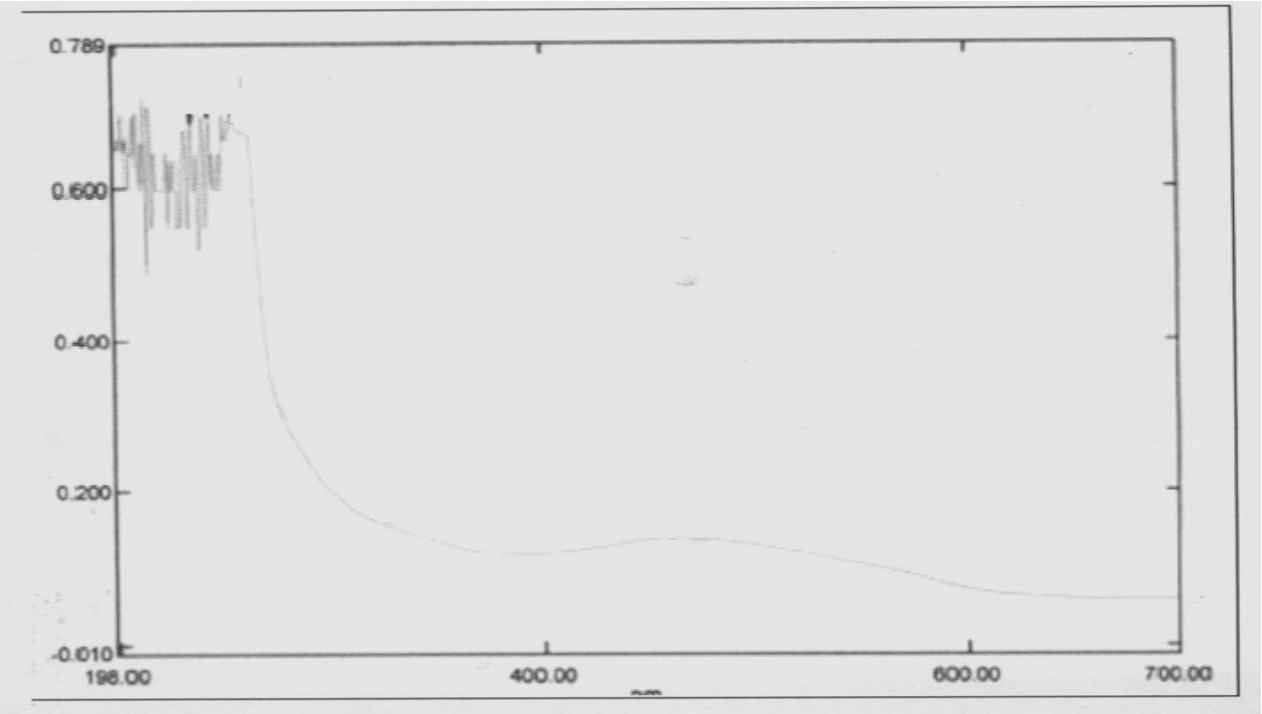


Fig.4: Electronic spectrum of the ligand.

Biological activity

The biological activity showed that 2-hydroxyacetophenone compound is effective on both pathogenic bacterial species tested (*Bacillus cereus* and *Staphylococcus aureus*). Whereas, 2-aminobenzoic acid compound has not effect at all and the prepared azodye has a less effect than 2-hydroxyacetophenone compound. The results show that some compounds tested in this study exhibited antimicrobial activity against bacteria strains tested. The 2-hydroxyacetophenone compound was stronger against bacteria strains. Whereas, 2-aminobenzoic acid compound did not show any inhibitory activity against bacteria strains tested and the azodye was less inhibitory activity than 2-hydroxyacetophenone compound.

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