Preparation and characterization of a novel ligand (5-MeTAQ) with some metal complexes of Cd(II), Zn(II), Hg(II)

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Abstract. This research includes preparation of heterocyclic compounds derivatives containing different rings through the use of 2-Amino-5-methyl thiazole as a starting material and their reaction with 8-hydroxy quinoline with some metal complexes Cd(II), Hg(II), and Zn(II) of the spectral characterized compounds were by melting point, FT-IR, UV-Vis, SEM and EDX

Keywords: Thiazolyl ligand (5-MeTAQ), Preparation of complexes Cd(II), Hg(II), Zn(II), characterization of compounds

1-introduction

Heterocycles form by far the major of classical divisions of organic chemistry and are of immense use biologically and industrially. It is well known that the heterocycles are present in all kinds of organic compounds of interest in electronics(1) biology, optics, pharmacology, material sciences and so on. Heterocyclic nucleus imparts an important function in medicinal chemistry and serves as a key template for the development of Thiazole or 1,3-thiazole is a heterogeneous ring containing both sulfur and nitrogen. Thiazol itself is a pale yellow liquid with pyridine - such as odor and molecular formula, (2) Thiazol also refers to a wide range of derivatives. Thiazol is a vitamin B1. The simplest member of the thiazole family is thiazole itself, a rarely prepared liquid with the molecular formula C3H3NS. Other thiazole compounds include rhodanine and the dye rhodanine red derived from it, and the yellow dye primuline. Synthetic drugs belonging to the thiazole family include sulfathiazole sulfasuxidine, and thiazolsulfone (Promizole) 2-Mercaptobenzothiazole (Mertax) is a thiazole derivative used for accelerating the vulcanization of rubberthiazole is found in many natural and synthetic products with a wide range of pharmacological activities (3), such as antiviral, anticancer antibacterial, antifungal, anticonvulsant (4), antiparkinsonian and anti-inflammatory activities that can be well illustrated by the large number of drugs in the market containing this function group. Due to its importance, the aim of this review is to highlight and the preparation of complexes of Cd(II), Hg(II), and Zn(II) with that ligand (5-MeTAQ). Finally the characterization of the
these complexes were by melting point , FT.IR, UV-Vis, SEM and EDX of the thiazole natural products

2- Experiment

2.1- Chemicals and method

All chemicals used are the 2-amino-5-methyl thiazole 8-hydroxy guinole ,NaOH ,HCl and NaNO2 produced by (Sigma ,Fluka and Aldrich) company , In addition to use of ethanol, and DMSO as solvent azo dye ligand

(5-MeTAQ) characterized by analytical data. the FT-IR spectra of azo dye ligand (5-MeTAQ) recorded in KBr medium using Shimadzu 8400 FT_IR spectrophotometer in wave length at rang (4000-400) cm⁻¹ the electronic spectra of ligand were recorded on a Shimadzu double beam UV-Vis spectrophotometer the range of (200-1100)nm in absolute ethanol solution , energy dispersive-x-ray (EDX) ,SEM images were taken on micrograph kyky 3200 of azo dye ligand

2.2- Preparation of azo dye ligand(5-MeTAQ)

Preparation of ligand with some 1,2g (0.01mol) of 2-amino -5-methyl thiazole in a mixture of 5ml hydrochloric acid 30 ml distilled water and (5)ml ethanol the mixture with continuous stirring and keep temperature above (5 °c) then the mixture added to ( 0.9)g Sodium nitrite dissolved to (30)ml distillated water added drop wise at (0-5°c ) continuous stirring for 25min the added of diazunium salt solution with continuous added drop wise with cooling at (0-5°c ) into 0.9g (0.012mol) of 8-hydroxy guinole was dissolved in mixture( 50)ml ethanol and (10) ml Sodium hydroxide . for coupling after had been stirring for two hour at (0-5°c) to pH=6.0 the precipitate

2.3-Preparation of Complexes

The preparation of metal complexes were prepared by dissolving ( 0.682) g (0.002 mol ) from ligand dissolving in ethanol (30ml) and added drop wise with stirring to (0.001mol) of 1:2 (Metal : ligand) molar ratio of Cd (II)and Hg(II),Zn(II) Chlorides salts dissolving in buffer Solution (ammonium acetate) at optimal pH for each metal ions

3- Result and Discussion

3.1- Infrared spectra

Infrared spectral of the prepared azo dye ligand (5-MeTAQ) ,IR spectrum showed absorption band in the region (3332) cm⁻¹ due to stretching vibration (O-H) thiazole group ,disappear of
(1612,1531,1457,1234) cm$^{-1}$ of (C=O),(C=N),(N=N),(C-S). (5,6)

Figure .(1) IR spectrum of Ligand (5-MeTAQ)

3.2- Electronic spectral

The electronic absorption of azo dye ligand (5-MeTAQ) and some metal complexes Cd (II),Zn (II) and Hg(II) of the ethanol solution (0.0001M) and at room temperature ,the electronic spectrum is characterized by three absorption is band in UV-Vis these bands are appearing at the ligand 240nm(41408) cm$^{-1}$ ,361 nm(40384) cm$^{-1}$ can be attributed transition at n $\rightarrow$ $\pi^*$ and band 465nm(19290) cm$^{-1}$ can be attributed transition at n $\rightarrow$ $\pi^*$ this band showed at red shift on coordination with metal ions Cd (II) shows three bands at 546nm(21275) cm$^{-1}$ ,394nm(11548) cm$^{-1}$ 234nm(11327) cm$^{-1}$ , the electronic spectra Zn(II)shows tree bands at 526nm(21146) cm$^{-1}$ ,431nm (21457) cm$^{-1}$ ,243nm(11328) cm$^{-1}$ ,the electronic spectra Hg (II)shows tree bands at 489nm(21446) cm$^{-1}$ ,362 nm (21452) cm$^{-1}$ ,241nm(11028) cm$^{-1}$ ,the UV-Vis spectra ligand and some metal complexes,(7,8)

Figure.(2)UV-Vis of Ligand (5-MeTAQ)
3.3- Metal: Ligand Ratio

The composition of metal complexes were determined by the method of molar ratio at pH =7.0 and optimum concentration at wavelength of maximum absorption (λmax). The solution of prepared metal complexes increase the intensity of the colors as approach my point of intersection ratio and the color continues constant at passing this point which indicates the metal complex formation ,The azo dye ligand (5-MeTAQ) was found to form 1:2 [M:L] chelates,(9) As in Figure (4).

Figure.(3) UV-Vis of Complexes of Cd(II),Hg(II), and Zn(II) with ligand (5-MeTAQ)

Figure.(4) Molar ratio method (M:L)  
5-MeTAQ) -metal chelates (  
M=Cd (II),Hg (II) and Zn(II))
3.4- The Study of the effect(pH)

If the optimal pH of each ion is selected with ligand (5-MeTAQ) depending on the solutions that give high absorption at (1x10^-4)M for all prepared complexes, the graph curves are drawn as in (pH) and absorption of metallic ions mixing with ligand. The optimum pH of each metal ion on its curve was determined from the curves. We conclude that the solubility of the solutions is at its lowest (pH = 3.3.5). This intensity increases with increasing (pH), up to a maximum of the optimal acidic function, indicating that the complexes have been completed at this pH (10,11) As in Figure (5) and Table(1).

![Graph showing pH and absorption relationship](image)

**Figure.(5) Effect of pH on the absorbance of (5-MeTAQ)- metal chelats;ligand**

| Ligand     | Metal ions | Optimal pH | Optimal molar con. x10^-4M | Metal : ligand | Molar Absorptivity x10^4 L. mol^-1 .cm^-1 | Amax)(nm) |
|------------|------------|------------|---------------------------|----------------|------------------------------------------|----------|
| (5-MeTAQ)  | Hg(II)     | 5.5        | 1.50                      | 1:2            | 14.23                                    | 486      |
|            | Cd(II)     | 6          | 1.25                      | 1:2            | 12.46                                    | 546      |
|            | Zn(II)     | 7          | 1.75                      | 1:2            | 16.35                                    | 526      |

Table(1): The optimal pH values ,optimal concentration ,mole ratios and wavelengths(λmax) with molar absorptivity( ) of metal ions

3.5- Energy-dispersive-X-ray spectroscopy (EDX)

This technique is used to determine the chemical elements in the sample and determine the weights for each element of the sample. For each element, there are a number of allowed transitions between atomic orbits. These transitions are described as quantum transitions, Kα, Kβ, Lα. and weights of elements in Ligand (5-MeTAQ) were shown in the following,(12) as in Figure (6).
3.6- SEM Analysis

Scanning electron microscopy (SEM) was employed to observe the morphology and particle size of the ligand (5-MeTAQ). The surface image is obtained with the SEM technique provides general information regarding the microstructure, surface morphology, particle size, chemical composition, and porous structures of the surfaces, in addition to the distribution of these particles. The SEM image of ligand have been illustrated in figure(7). SEM image shows the ligand (5-MeTAQ) that has a homogeneous particle shape surface with fewer aggregations and a particle size,(13,14)
Figure.(8) the proposed structural of ligand 2-[5-(5-methyl thiazoly) azo] -8-hydroxy guniole (5-MeTAQ)

4- Conclusion

The Preparation and the Characterization azo dye ligand (5-MeTAQ) derived which from thiazole and its some metal complexes with Cd (II),Hg (II) and Zn(II) metal chlorides , the spectroscopy of ligand by analytical data, FT-IR, electronic spectra and EDX, and SEM of the study pH effect and mole ratio of metal complexes.

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