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## Antimicrobial Activities of Novel Schiff Base Cu (II) Complex Derived from Glutaraldehyde and L-Histidine

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### Abstract:

Novel metal complex of Cu(II) with Schiff base derived from Glutaraldehyde and L-Histidine have been prepared and characterized by magnetic susceptibility, conductance measurement, elemental analyses, UV-Visible, IR, <sup>1</sup>H-NMR spectral studies, powder XRD and TGA. The magnetic and spectroscopic data indicate an octahedral geometry for the complex. The molar conductance measurement indicates that the complex is non-electrolytic in nature. The electronic absorption spectra of the complex shows intraligand and charge transfer transitions. Room temperature magnetic susceptibility measurement reveals that the nature of the complex is paramagnetic. The XRD data shows that the complex is microcrystalline. The antimicrobial activities of ligand and its complex were screened by Disc Diffusion method.

**Keywords:** Schiff base; Metal complex; Spectral studies; Antimicrobial activity

### 1. Introduction

Schiff bases and their coordination compounds have gained importance recently because of their application in biological, biochemical, analytical, anticancer, antibacterial and antifungal activities. Studies of new kinds of chemotherapeutic Schiff bases have now attracted the attention of biochemists. Metal complexes of Schiff bases and their applications have been widely investigated during the past years<sup>(1)</sup>. Schiff bases are very good complexing agents.

In continuation of the earlier work on Schiff base complexes in this paper, the synthesis, characterization and antibacterial studies of novel Schiff base ligand derived from Glutaraldehyde and L- histidine have been reported. The ligand has both oxygen and nitrogen donor sites. It coordinates with the metal ion in a tetradentate manner.

### 2. Materials and Methods

All the chemicals and solvents used in the present work were of analytical grade. The metal is used as its nitrate salt. The percentage compositions of the elements in the compound were determined using a Vario EL III elemental analyzer at Sophisticated Analytical Instruments facility, CUSAT, Kochi. Ultraviolet spectra were recorded using Shimadzu double beam visible spectrophotometer in the visible region. Conductance of the metal complex was determined in DMSO on SYSTRONICS digital conductivity meter. The room temperature magnetic susceptibility measurement of the complex reported in the present study was made by the Guoy's method using Copper sulphate as calibrant. IR spectra of the Schiff base and its complex in the range of 4000 to 400 cm<sup>-1</sup> were recorded on a Perkin Elmer FT-IR spectrometer MODEL 1600 as KBR discs. <sup>1</sup>H NMR spectra of the complex in DMSO-d<sub>6</sub> were recorded on by employing TMS as internal standard at NIIST Trivandrum. Powder XRD was recorded on a computer controlled X-ray diffractometer system JEOL JDX 8030. Double distilled water was used throughout the experimental work. Antimicrobial studies of the compounds were studied by disc diffusion method.

#### 2.1. Synthesis of Schiff Base

The Schiff base ligand was prepared by reacting Glutaraldehyde and L-histidine in 1:2 molar ratio by refluxing in distilled methanol. The mixture was refluxed for 1 hour. The reaction was examined by TLC with time to time till completion. The solvent was partially evaporated and the yellowish mass product was precipitated by cooling and filtered off, washed with distilled water, dried, recrystallised and finally preserved in a desiccator.

#### 2.2. Synthesis of Schiff Base Complex

Metal (II) nitrate was dissolved in 200 cm<sup>3</sup> of methanol. The filtered solution was added dropwise into 20cm<sup>3</sup> methanol solution of the Schiff base ligand, the resulting mixture was refluxed and stirred for 8 hours. After refluxing, the volume of the solution was reduced to one third and the concentrate was cooled at 0°C. The precipitated complex was filtered off, washed several times with cold ethanol and dried in vacuo over anhydrous CaCl<sub>2</sub>.

### 3. Results and Discussion

Metal (II) salt reacts with Schiff base ligand in 1:1 molar alcoholic medium to afford brownish coloured complex. Cu(II) complex is normally stable at room temperature and hygroscopic in nature. The Schiff base ligand is soluble in common organic solvents like ethanol and methanol. The corresponding complex is soluble in DMSO. The Schiff base and its complex is subjected to

elemental analysis. The results of elemental analysis with molecular formula are presented in Table 1. The results obtained are in good agreement with those calculated for the suggested formulae. The Cu (II) complex is non-electrolytic in nature<sup>(2)</sup> as the molar conductivity measurements in DMSO is  $9.15 \text{ ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$ .

Ligand/Metal Chelate	Empirical Formula	Colour	M:L ratio	Molar Cond. ( $\text{Ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$ )	Elemental analysis % Found (cal)			
					C Found (Cal)	H Found (Cal)	N Found (Cal)	M Found (Cal)
Glu-his ligand	$\text{C}_{17}\text{H}_{22}\text{N}_2\text{O}_4$	Yellow	-	-	52.32 (52.5)	4.23 (4.65)	15.34 (15.48)	-
[Cu(glu)(his).2 $\text{H}_2\text{O}$ ]	$\text{C}_{17}\text{H}_{26}\text{N}_6\text{O}_6\text{Cu}$	Greenish brown	1:1	9.15	42.89 (43.06)	5.50 (5.53)	17.45 (17.73)	13.28 (13.40)

Table 1: Analytical data of Schiff base and its complex

### 3.1. UV-Visible Spectra

The UV-Visible spectra are often very useful in the evaluation of results furnished by other methods of structural investigation<sup>(3)</sup>. The electronic spectral measurements were used for assigning the stereochemistry of metal ions in the complex based on the positions and number of d-d transition peaks<sup>(4)</sup>. The electronic absorption spectra of the Schiff base ligand and its complex was recorded in DMSO solution in the range of 200 to 800 nm regions is given in Fig.1. It is usually recorded as a plot of absorbance (A) versus wavelength ( $\text{cm}^{-1}$ ). The absorption spectrum of free ligand consist of an intense band centered at 285 nm which is assigned to  $\pi - \pi^*$  transition of the C=N chromophore. On complexation, this band was shifted to lower wavelength region at 235 nm suggesting the coordination of azomethine nitrogen with Cu(II) ion. The spectra also shows other transitions in the range of 330 and 345 nm which can be assigned to  $n - \pi^*$  transition. Another transition was found to be in the range of 350 and 370 nm which may be due to charge transfer transition. The spectra also show certain absorption bands at 595 and 625 nm which is in accordance with d-d transitions.

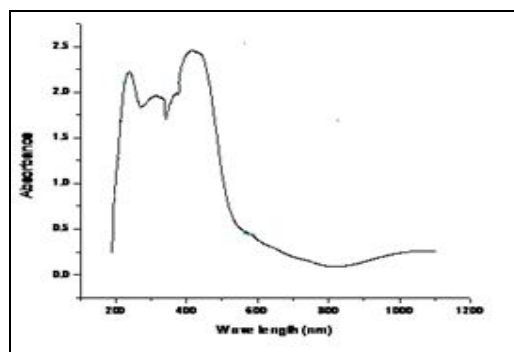


Figure 1: UV Spectrum of Cu (II) Complex

### 3.2. Magnetic Moment

The effective magnetic moment value for the Cu II complex is 1.82 BM which is slightly greater than the spin only value 1.73 BM that was expected for one unpaired electron. This value offers possibility of an octahedral geometry around the metal ion.

### 3.3. Infra Red Spectra

The Schiff base ligand show  $\nu(\text{C}=\text{N})$  azomethine band observed at  $1655 \text{ cm}^{-1}$ . On complexation, this band was shifted to  $1598 \text{ cm}^{-1}$  region<sup>(5)</sup> due to the coordination of azomethine to the Cu (II) ion. New bands observed at  $546 \text{ cm}^{-1}$  and  $486 \text{ cm}^{-1}$  which are not seen in the spectrum of the free ligand can be attributed to the  $\nu(\text{M}-\text{O})$  and  $\nu(\text{M}-\text{N})$  vibrations respectively<sup>(6)</sup>. The spectra of the complex illustrate broad band in  $3044 \text{ cm}^{-1}$  region assigned to the presence of water molecules in the complex.

### 3.4. $^1\text{H}$ NMR Spectrum

$^1\text{H}$  NMR Spectrum of the ligand recorded in DMSO solution shows a multiplet at 2.7, 1.8 ppm due to the methyl protons. The  $^1\text{H}$  NMR Spectra of the Schiff base complex exhibit signals at 8.5 and 7.5 ppm attributed to  $\text{CH}=\text{N}-$  and  $-\text{NH}$  protons respectively. The azomethine proton signal in the spectrum of the corresponding complex is shifted downfield compared to the free ligand, suggesting the deshielding of the azomethine group due to the coordination with the metal ion. There is no appreciable change in all other signals of the complex.

### 3.5. XRD Study

X-ray diffraction pattern of Cu (II) complex shows sharp crystalline peaks. The crystallite size of the complex could be estimated from XRD pattern by the Scherrer's formula,

$D_{\text{XRD}} = 0.9 \lambda / \beta \cos \theta$  Where  $\lambda$  is the wavelength,  $\beta$  is the full width at half maxima and  $\theta$  is the diffraction angle. The XRD shows that Cu(II) complex has the crystallite size of 53 nm and so it is microcrystalline in nature<sup>(7)</sup>

### 3.6. TGA Study

The dynamic TGA with the percentage mass loss at different steps have been recorded. The complex lose its weight in the temperature range 140-240 °c corresponding to two coordinated water molecules with an endothermic peak in DTA curve. After the total loss of water molecules, the decomposition occurs at 650-750 °c that indicate the decomposition of the ligand. The observed residue corresponds to the respective metal oxide. Based on the spectral and analytical characterization studies the expected geometry for the Cu(II) complex is shown in Fig.2.

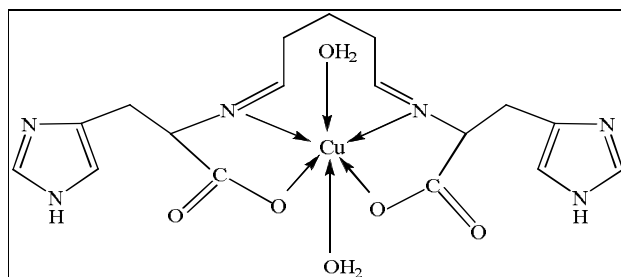


Figure 2: Structure of Cu (II) complex

### 4. Antimicrobial Study

The *In vitro* antimicrobial study of the compounds was tested against the bacteria *Klebsiella sps*, *E.coli*, *Staphylococcus aureus* and fungi *Candida sps*, *Aspergillus niger* and *Aspergillus fumigates* by Disc diffusion method. From the MIC values obtained it was found that the complex exhibit higher activity than the free ligand. This may be ascribed to the increased lipophilic nature of the complex arising due to chelation.

### 5. Conclusion

The elemental analysis, magnetic susceptibility, UV/ Visible and <sup>1</sup>H NMR spectral observations and powder XRD study suggest the octahedral geometry for the Cu (II) complex and exhibit coordination number six. The very low conductance value for the complex indicates that the complex is non-electrolytic in nature. TGA study show the presence of water molecules in the complex. Antimicrobial study indicates the predominant activity of the complex than its corresponding ligand.

### 6. References

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