



Review Article

ISSN : 0975-7384  
CODEN(USA) : JCPRC5

## Synthesis, Spectroscopic and Antimicrobial Studies of Cu (II) and Fe (III) Complexes of Heterocyclic Schiff Base Ligand

SY Deshmukh<sup>\*1</sup>, NS Padole<sup>2</sup>, MP Wadekar<sup>3</sup>, MA Chaudhari<sup>4</sup>

<sup>1</sup>Department of Chemistry, Vidharbha Institute of Science and Humanities, Amravati, India

<sup>2</sup>Department of Chemistry, Vinayak Vidhyan Mahavidyalaya, Nandgoan Khandeshwar, India

<sup>3</sup>Applied Chemistry Division, Govt. Vidharbha Institute of Science and Humanities, Amravati, India

<sup>4</sup>Department of Chemistry, Govt. Vidharbha Institute of Science and Humanities, Amravati, India

### ABSTRACT

In the presence study synthesis, characterization and antimicrobial studies of newly synthesized Cu (II) and Fe (III) complexes from heterocyclic Schiff base ligand. The new Schiff base ligand was synthesized by condensing chalcone 1-(2-hydroxy-3-nitro-5-methyl acetophenone)-3-(2-hydroxy phenyl) with phenyl hydrazine hydrochloride in the ethanolic medium. From this ligand new transition metal complexes of Cu (II) and Fe (III) with a Schiff base ligand 3-(2-hydroxy-3-nitro-5-methyl phenyl)-5-(2-hydroxy phenyl)-1-phenyl- $\Delta$ 2-pyrazoline were synthesized and characterized on the basis of elemental analysis, <sup>1</sup>H NMR, IR, Mass, Electronic spectra and Molar conductance data. IR spectral data shows that ligand acts as monobasic ONdonor towards both Cu (II) and FE (III) ions. The antimicrobial activities of these complexes were screened against *S.aures*, *S.pyrogenes*, *E. coli* and *S.typhe* pathogens which are heretofore too unknown.

**Key words:** Metal complexes; Schiff Base; Antimicrobial activity

### INTRODUCTION

Over the years, scientific research has expanded our knowledge of metal complexes synthesized from Schiff base ligand plays an influential role for the development of coordination chemistry. Since it has appreciable role in the pharmaceutical fields such as antimicrobial, antifungal, antibacterial, anticancer, biochemical and biological activities. Heterocyclic coordination compounds can also effectively used as catalyst for oxidation, reduction and hydrolysis reaction. Heterocyclic Schiff bases generally are a Nitrogen and Sulphur analogue of aldehyde and ketone having good ability to form a metal complex. For the synthesis of complexes, the Schiff base are used which are generally synthesized by refluxing carbonyl compound with primary amine. From the literature survey it looks that heterocyclic Schiff base has been enormously used as biological active complexing agent. Last decades improvement in the synthetic procedure of metal complexes and biological activity of transition metal complexes containing O, S and O,N donor atom. The present study focus on the synthesis of metal complexes of Cu(II) and Fe(III) ion from heterocyclic Schiff base which has been proven using molar conductance and spectroscopic characterization.

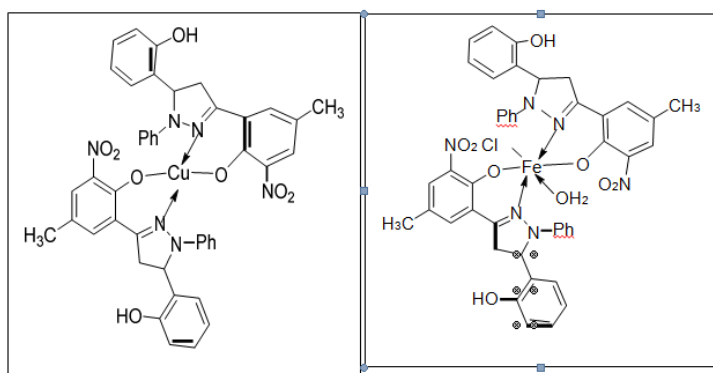
### Experimental

All the chemicals and solvents used in the study were obtained commercially and of analytical grade. It included PhenylHydrazineHydrochloride, CuNO<sub>2</sub>.6H<sub>2</sub>O, FeCl<sub>3</sub>.6H<sub>2</sub>O, ethanol, methanol and dimethyl formamide. The IR spectra of ligand and complexes were recorded on Perkin-Elmer spectrometer in Nuzol in range 400-4000cm<sup>-1</sup>. PMR spectra of ligand was recorded on Bruker 400F spectrometer using DMSO-d<sub>6</sub>as solvent and TMS as internal standard. The FAB-mass spectra of ligand and

complexes were recorded with thermoscientific TSQ 8000 gas chromatograph-mass spectrometer. The antimicrobial activity of metal complexes of substituted pyrazoline were determine by using disc diffusion method.

### Synthesis of Schiff Base ligand

The mixture of 1-(2-Hydroxy-3-nitro-5-methyl acetophenone)-3-(2-hydroxyphenyl) chalcone (0.01M) and Phenyl Hydrazine Hydrochloride (0.02M) was added to the hot ethanolic solution reflux up to 2Hr and progress of reaction was checked by thin layer chromatography using methane hexane system. The reaction mixture was decomposed into ice water and solid obtained washed by petroleum ether. Yield 78%, Melting point 155°C. Scheme for the synthesis of Schiff base ligand as shown in figure1. PMR spectrum was carried out in DMSO-d<sub>6</sub> and TMS used as a internal solvent.



**Figure 1: Probable structure of Cu (III) complex Probable structure of Fe (III) complex**

### Synthesis of Metal Complexes

Both metal complexes were prepared by adding hot ethanolic solution of metal salt of CuNO<sub>2</sub>.6H<sub>2</sub>O (II) and FeCl<sub>3</sub>. 6H<sub>2</sub>O(III) to a hot ethanolic solution of ligand in equimolar proportion i.e., 1:1 ration. The reaction mixture was refluxed for 15 to 16 hours, residue obtained and kept it for drying. Crystallization of complexes was done with methanol.

### RESULT AND DISCUSSION

Both the metal complexes are colored solid and soluble in DMSO and DMF but insoluble in common inorganic solvent such as methanol and ethanol. The analytical data shows good accord with the intended structure of Schiff base ligand as well as metal complexes. It is also showing 1:1 ratio for metal to ligand for both Cu(II) and Fe(III) ions. Th lower values of conductance shows that both the complexes are non-electrolyte. The physical and analytical data of ligand and its complexes shown in table 1.

**Table1: Physical and analytical data of ligand and its metal complexes.**

Compounds	Mol. Formula	Colour	F.W.	M.P.	C%	H%	N%	M%
					Found/Calculated			
LH	C <sub>22</sub> H <sub>19</sub> N <sub>3</sub> O <sub>4</sub>	Yellowish White	389	155 °C	67.86	4.88	10.79	---
[Cu(L) <sub>2</sub> . H <sub>2</sub> O]	C <sub>44</sub> H <sub>38</sub> CuN <sub>6</sub> O <sub>9</sub>	Blue	857.54	>300 °C	61.5	4.43	9.79	7.40
[Fe(L) <sub>2</sub> Cl.H <sub>2</sub> O]. H <sub>2</sub> O	C <sub>44</sub> H <sub>38</sub> ClFeN <sub>6</sub> O <sub>10</sub>	Green	904.36	>300 °C	58.38	4.20	9.28	6.06

### Infrared Spectra

Fourier transform Infrared spectra gives valuable information regarding the nature of bonding mode and the functional group of ligand and also gives information of attachment of ligand to the metal. Hence to identify the binding mode of ligand with metal complexes IR spectrum of ligand has been compared with IR spectrum of metal complexes in the range between 400-4000 $\text{cm}^{-1}$ . The IR spectrum of Schiff base ligand shows broad band at 3333 $\text{cm}^{-1}$  of OH and the broadening of band due to intramolecular hydrogen bonding between -OH and azomethine group. The frequency of -OH in metal complexes shifted towards lower frequency at 3318 $\text{cm}^{-1}$  it may shows that involvement of one of the -OH group. It also proves by the phenolic C-O stretching frequency of metal complexes which is obtained at 1373  $\text{cm}^{-1}$  and 1310  $\text{cm}^{-1}$  for Cu (II) and Fe (III) ions, it is greater than phenolic C-O band in ligand. The peak of azomethine group -CH=N at 1832 $\text{cm}^{-1}$  in the spectrum of ligand which is shifted towards lower frequency in both Cu (II) and Fe(III) complexes at 1794  $\text{cm}^{-1}$  and 1788  $\text{cm}^{-1}$  respectively indicates that azomethine Nitrogen coordinate to metal ions. Electron density during coordination decreases and hence IR stretching frequency also decreases. The additional peak which was absent in ligand but present in metal complexes are in the range of 424-478  $\text{cm}^{-1}$  and 498-554  $\text{cm}^{-1}$ , assigned to stretching frequency of  $\nu$  (M-N) and  $\nu$  (M-O) bands

### Mass Spectra

The molecular ion peak for Schiff base ligand has been successfully determined by mass spectroscopy. The mass spectral data show good agreement for both Cu (II) and Fe (III) complex is dimeric.

### Electronic Spectra

The electronic spectra of metal complexes were recorded in DMSO in the range 200-800nm. The electronic spectrum of Cu complexes shows transition band at 382nm and at 330nm ascribed to  $1A_{1g}-1E_g$  and ligand to metal charge transfer. This transition indicates square planer geometry for Cu (II) complex (12). For Fe (III) complex bands appears at 510,426,372 and 360nm for transition  $6A_{1g}-4T_{1g}$ ,  $6A_{1g}-4T_{1g}(G)$ ,  $6A_{1g}-4E_g(G)$  and fourth band may be due to charge transfer spectra. All these transitions indicate the octahedral geometry for Fe (III) complex

**Table2: Infrared spectral data of ligand and its metal complexes.**

Complex	V (O-H)	V (C-OH)	V (C=N)	V (M-N)	V (M-O)
LH	3333 $\text{cm}^{-1}$	1265 $\text{cm}^{-1}$	1832 $\text{cm}^{-1}$	----	----
Cu	3318 $\text{cm}^{-1}$	1373 $\text{cm}^{-1}$	1794 $\text{cm}^{-1}$	424 $\text{cm}^{-1}$	498 $\text{cm}^{-1}$
Fe	3309 $\text{cm}^{-1}$	1310 $\text{cm}^{-1}$	1788 $\text{cm}^{-1}$	478 $\text{cm}^{-1}$	554 $\text{cm}^{-1}$

**Table 3: Electronic absorption spectrum data of complexes.**

Compounds	Solvent	Absorption (nm)	Band assignment	Geometry
[Cu(L) <sub>2</sub> ]	DMSO	382	$1A_{1g}-1E_g$	Square planer
		330	LMCT	
[Fe(L) <sub>2</sub> ]	DMSO	510	$6A_{1g}-4T_{1g}$	Octahedral
		426	$6A_{1g}-4T_{1g}(G)$	
		372	$6A_{1g}-4E_g(G)$	
		360	LMCT	

**Table 4: Antimicrobial activity of complexes**

Complexes	Gram +ve		Gram -ve	
	<i>S. aureus</i>	<i>S. pyrogenes</i>	<i>E. coli</i>	<i>S.typh</i>
[Cu(L) <sub>2</sub> ]	19 mm	25 mm	16 mm	19 mm
[Fe(L) <sub>2</sub> ]	22 mm	19 mm	16 mm	20 mm

**Antimicrobial Activity**

Any chemical substance inhibiting the growth of micro-organism is known as antimicrobial agent (15). Inorganic compounds play an important role in organism; hence the inorganic chemist has solely begun to recognized the similarities between compound they worked with and biologically important compound containing metal ions. These two trends have merged into active research i.e., bioinorganic chemistry (16-19). The antimicrobial activity of both compounds were screened by using disc diffusion method in DMF, against gram positive and gram-negative bacteria such as *S. aureus*, *S. pyrogenes*, *E. coli* and *S. typh*

**Disc Diffusion Method**

The antimicrobial activities of both metal complexes of substituted pyrazoline were determined by using disc diffusion method briefly 100µl of suspension containing colony forming units of bacteria cells (Gram positive and Gram negative) were spread on petri plates containing nutrients Muller Hinton auger (NA) and potato dextrose auger(PDA) medium (50ml media per plate) respective medium. The paper disc 6mm diameter were separately impregnated with 15 µl of extract component of compound under study and placed on the auger which had previously been inoculated with the selected test microorganism. The disc without sample were used as negative control. Plates were kept at 40C for 1 hour. The plates were incubated at 370C for 24 hours for bacteria stain. The antimicrobial activity was assessed by measuring the diameter of growth inhibition zone in mm for the test of organism comparing to the controls. The result is cited in table number 4. All the four organisms studied are human pathogens, from the result it is cleared that both the synthesized metal complexes show remarkable and considerable antimicrobial activity. Complex of Cu (II) ions shows excellent activity against *S. pyrogenes* and good activity against *S. aureus* and *S. typh*. *S. aureus* and *S. typh*. excellent activity for the complex of Fe ion, it also shows good activity against *S. pyrogenes*. From the data it is concluded that these complexes show remarkable antimicrobial activity.

**CONCLUSION**

The present article includes the synthesis and characterization of new Schiff base ligand and its Cu (II), and Fe (III) complexes. The IR, 1NMR, Mass and UV techniques were used for spectral data interpretation of compounds. The spectral data specify that the flexidentated nature of ligand was observed toward different metal ion. Its co-ordinates to both Cu (II) and Fe (III) metal ion through azomethine nitrogen and hydroxyl hydrogen and act as monobasic bidentated and metal ligand ratio 1:2. The remarkable and noticeable antimicrobial activity shown by both metal complexes. The antimicrobial activity test and proposed structure of Cu (II) and Fe (III) complexes.

**ACKNOWLEDGEMENT**

The authors are thankful to The Dctor and Head, Department of Chemistry, Govt. Vidharbha Institute of Science and Humanities, Amravati for providing necessary laboratory facility, Directors of RSCI, Chandigarh, for recording IR and 1H NMR spectra, CDRI, Lucknow, for elemental analysis.

**REFERENCES**

- [1] Joshi KR, Rojivadiya AJ, Pandya JH, et al. Int J Inorganic Chem. 2014;17 ;2014..
- [2] Nair, MS, Arish D, Joseyphus RS, et al. J Saudi Chem Soc. 2012; 16(1), 83-88.
- [3] Abd El-Halim HF, Mohamed GG, Anwar MN, et al. Appl Organometallic Chem. 2018; 32(1), e3899.
- [4] El-Gammal OA, El-Reash GA, Bedier RA, et al. Appl Organometallic Chem. 2019; 33(10), e5141.
- [5] Mahmoud WH, Deghadi RG, Mohamed GG, et al. Appl Organometallic Chem. 2016; 30(4):221-230.

