International Journal of Science and Research (IJSR)
ISSN: 2319-7064
Impact Factor 2024: 7.101

Synthesis and Characterization of Copper (II) Complex of 2-(1-(benzyl imino) propyl) 4-bromo phenol

Shaila Wagh

Department of Chemistry, Adarsh Education Society's Arts, Commerce and Science College, Hingoli, (M.S.) India 431513 Email: Email: *sswagh16[at]rediffmail.com*

Abstract: ***A new Schiff base ligand, 2-(1-(benzyl imino) propyl) 4-bromo phenol has been synthesized by condensation of 5'-bromo-2'-hydroxy propiophenone with benzylamine and its Cu (II) complex has been synthesized in the alcoholic medium. The newly prepared Schiff base ligand and its Cu (II) complex have been characterized by various physicochemical techniques such as elemental analysis, magnetic moment, molar conductance method, UV-vis, infrared and ^1H NMR spectral technique and thermogravimetric studies. Elemental analysis and spectral studies indicated six coordinated complex, IR and TGA studies confirmed the presence of the coordinated water molecules in the Cu (II) complex.***

Keywords: Schiff base, Metal complex, electronic spectra, IR spectra

1. Introduction

Schiff base ligands are found to be the most sensitive and selective ligands for the formation of metal complexes in coordination chemistry. Schiff bases are compounds with an azomethine group produced by the condensation of a primary amine with an active carbonyl group of aldehyde or ketones. These are useful chelating ligands for a varied range of metal ions. These Schiff base metal complexes have found several applications in various fields like catalytic,[1-5] industrial and biological[6-9]and analytical.[10] Schiff base metal complexes possess powerful anticancer[11-14] activity. Similarly, these complexes are found to have antifungal,[15-16] antimicrobial[17-20] and antiviral activities.

In this work, we have reported the synthesis and characterization of a novel Schiff base of 2-(1-(benzyl imino) propyl) 4-bromo phenol derived from the condensation of 5'-bromo-2'-hydrdoxy propiophenone with benzylamine and its complex with Cu (II).

2. Experimental

2.1 Material and Methods

All analytical-grade chemicals were used without further purification. 2' hydroxy propiophenones have been purchased from Sigma Aldrich. The literature approach was used to synthesize bromo-substituted 2' hydroxy propiophenones. The melting points were determined using an open capillary tube and are uncorrected. UV spectra were recorded on W-1800 series spectrometer, ^1H NMR spectra were recorded on Brucker Avance Neo 500MHz spectrophotometer in DMSO solvent, FTIR spectra were recorded on Shimadzu IR spectrometer. The molar conductance (10^{-3}M in DMF) was measured using a Systronic CM304 conductivity bridge. Thermal analysis was carried out by using Shimadzu (TGA-50 H) from room temperature to 900^0C at a heating rate of 10^0C per minute.

2.2 Synthesis

I) Preparation of Ligand

Ligand was synthesized by refluxing the mixture of 5'-bromo-2'-hydroxy propiophenone (0.01M) and benzylamine (0.01M) in ethanol solvent for 3-4 hours along with catalytic amount of glacial acetic acid.The progress of the reaction mixture was monitored by thin layer chromatography. After completion of the reaction, the reaction mixture was cooled at room temperature and 25 ml of ice-cold water was added to it. The yellow solid separates. The separated solid was filtered, washed with cold water and recrystallized by ethanol to obtain pure crystals.

II) Synthesis of metal complexes

The Cu (II) metal complex was prepared by refluxing ethanolic solutions of Cu (II) salt (0.02 M) with an ethanolic solution of Schiff base (0.01M) for 3-4 hrs. The pH of the solution was adjusted by adding an alcoholic solution of ammonia. A coloured product appeared on standing and cooling the above solution. The precipitated complex was filtered, washed with ethanol and dried over fused $CaCl_2$.

2.3 Microbial Assay

The synthesised Cu (II) metal complex was screened against nine microbial strains which include two bacteria *S. aureus, E. coli and* fungus *C. albicans.*

2.3.1 In vitro microbial activity

The microbial activity of newly synthesized compounds was analysed by disc diffusion method. The Mueller Hinton plate was inoculated with the bacterial suspension, and the sterile discs were impregnated with the test compounds. The assay was inoculated at 37 °C for 24 hours and the zone of inhibition was measured. Ofloxacin was used as the standard antibacterial drug for gram-positive bacteria, ciprofloxacin was used for gram-negative bacteria and fluconazole was used for fungi.

3. Result and Discussion

The ligand and copper (II) complex are found to be stable at room temperature and are nonhygroscopic.

3.1 Molar conductance measurement and magnetic moment:-

The molar conductance of the complex in DMSO lies in the range of 20 $\Omega^{-1}cm^2mol^{-1}$ which indicates that non-electrolyte nature of Cu (II)complex.The magnetic moment observed for the copper (II) complex is 1.95 B.M which is consistent with the distorted octahedral stereochemistry of the complex.[21]

3.2 Infra red spectra

The spectrum of the ligand showed a band at 3028 cm^{-1} for phenolic -OH stretch which disappeared in Cu(II) complex indicating the coordination of phenolic oxygen with copper.[22] The stretch at 1573 cm^{-1} attributed to C=N stretch in ligand is shifted to lower frequencies in the spectra of the complex (1512 cm^{-1}) indicating the involvement of –C=N nitrogen in coordination to the metal ion.[23] A band at 1240 cm^{-1} is assigned to νC-O stretching frequency which appeared at 1339 cm^{-1} in the spectra of metal. The infrared spectrum of the complex display new bands at 487 and 532cm^{-1} attributed to νM-O and νM-N vibrations respectively.[24] The appearance of νM-O and νM-N vibrations supports the involvement of nitrogen and oxygen atom in complexation with metal ion under investigation.

3.4 Electronic spectra

The electronic spectra of Cu (II) complexes showed a band in the range 15385 cm^{-1} assigned to $^2Eg \rightarrow 2T_{2g}$ and a charge transfer transition in the range 27473cm^{-1}.[25] The ligand field parameters viz ligand field splitting energy (10Dq) and LFSE were found at 15385 and 43.92 resp. All of these elements contribute to the formation of the distorted octahedral complex. The magnetic moment of the Cu (II) complex (1.95 B.M.) further confirmed the distorted cathedral geometry. Molar conductance 18.9 $Ohm^{-1}cm^2mol^{-1}$.

3.5 ^{1}H NMR spectra

^{1}H NMR spectrum of the ligand in DMSO d_6 displayed singlet at 16,86 ppm which is attributed to the hydroxyl proton of ligand[26] which was found to be disappeared in the complex.

3.6 Thermogravimetric Analysis

Thermal studies help in determining the existence of water molecules in complexes and also to study the decomposition pattern. Thermal studies of the complex were conducted in the range of 50-900^{0}c by controlling the heating rate of 15^{0}c/min. The TGA curve of Cu (II) complex of ligand was found to be stable almost up to 230^{0}c indicating the absence of lattice water.[27] At about 310^{0}c, the TG curve of the Cu (II) complex of the ligand showed weight loss. The weight loss corresponds to the two coordinated water molecules. (actual 4.89%, cal. 4.95%). The loss of water molecules was confirmed by the exothermic peak obtained in DTA. The other exothermic peak was observed at 358.8^{0}c which corresponds to the loss of some organic moiety. Following this, the complexes displayed a sharp drop in weight loss suggesting that the organic moiety of the complex is eliminated and then decomposition continues up to 900^{0}c showing the formation of stable copper oxide.

3.7 Biological Evaluation

The Schiff base ligand and its Cu (II) complex were screened for antimicrobial activity against *S. aureus, E. coli and C. albicans.* According to the data, copper (II) complex showed higher antimicrobial activity than the ligand.

Ligand/ Complex	*S. aureus* Zone of inhibition (mm)	*E. coli* Zone of inhibition (mm)	*C albicans* Zone of inhibition (mm)
Ligand	17	14	NR
Cu (II) complex	26	18	10

4. Conclusion

The syntheses, characterization, and molecular structure of new Cu (II) complex of the Schiff base ligand have been reported in this paper and characterized by magnetic and spectral data. Cu (II) indicated distorted octahedral geometry. The molar conductance values of the complex imply the non-electrolytic structure, thermal analysis indicates the presence of two coordinated molecules. Antibacterial and antifungal activity indicated that Cu (II) complex possess more activity than the corresponding ligand.

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