

Synthesis, Characterization and Antimicrobial Activity of Metal Complexes prepared from Schiff Base Ligand

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ABSTRACT

Coordination compounds of some transition metal ions Co(II) of synthesized ligand 4-chloro-2-(((4-chlorophenyl) imino)methyl) phenol. These Schiff base ligand were characterized by elemental analysis, FTIR, UV-Vis, ¹H-NMR, ¹³CNMR, Mass spectral analysis, magnetic susceptibility and molar conductance, whereas, Metal complexes is analyzed by IR, UV-Vis, Magnetic susceptibility, Molar Conductance, elemental analysis. Schiff base ligand and its metal (II) complexes were tested in vitro to evaluate their bactericidal activity against bacteria *S. aureus* and *B. subtilis* and antifungal activity against *A. Niger* and *F. Oxysporum* using the Kirby-Bauer disc diffusion method. antibacterial evaluation results revealed that the metal (II) complexes exhibited higher antibacterial activity than the free Schiff base ligand.

Keywords : Metal Complexes, Antifungal Activity, Schiff Base

I. INTRODUCTION

Transition metals are metallic elements that have an incomplete d or f shells in the neutral or cationic states. These incomplete valence shell orbitals allow it to accept electrons from Lewis bases to form coordination complexes very easily compared to other group of elements. Ligands therefore must be a Lewis base. They must contain at least one pair of non-bonding electrons that can be donated to a metal ion. The Schiff base is named after Hugo Schiff and is a compound with a functional group that contains a -C=N- double bond with the nitrogen connected to an aryl or alkyl group.[1] Schiff bases in a broad sense have the general formula $R_1 R_2 C = NR_3$, where R is

an organic side chain. Schiff base is synonymous with azomethine and may also be referred to as imines. Recently, Schiff bases are used as intermediates for the synthesis of ligands for preparation of metal complexes having a series of different structures[2]

Schiff's base has an azomethine group which is reported to play an important role and shows high antibacterial activity [3]. Schiff bases are usually synthesized from the condensation of a primary amine and a carbonyl group by nucleophilic addition to form a hemiaminal, followed by dehydration to give azomethine [4]. So, to be able to synthesize Schiff bases, carbonyl precursors and amine precursors are needed.

Schiff base compounds and their metal complexes are very important as catalysts in various biological systems, [5] polymers, [6] dyes [7] and medicinal & pharmaceutical fields [8], they comprise miscellaneous therapeutically potent applications in the field of medicinal chemistry [9]. Schiff bases have played an important role in the development of coordination chemistry as they readily form stable complexes with most of the transition metals [10]. They have been shown to exhibit a broad range of biological activities, including antifungal, antibacterial, [11] anti malarial, [12] anti-inflammatory, [13] antiviral, [14] and antipyretic properties [15]. A variety of Schiff's base and its complexes have been studied extensively. Several model systems, including those with bidentate, tridentate, tetradentate multidentate Schiff base ligands, [16] and their coordination chemistry of Cu(II), Co(II) & Ni(II) attracts much attention because of its biological relevance and its own interesting coordination chemistry such as geometry, flexible redox property. [17]

Then, one of the efforts to increase the antibacterial activity of Schiff base compounds is to make complex compounds. According to Hashem et al. [18], the complex formation can increase the activity of Schiff base as an antibacterial. Schiff base complexes with Co(II), Ni (II), and Cu (II) metals are reported to have high antibacterial activity [19]. Schiff bases can act as mono-, di-, tri-, or tetra-dentate ligands depending on the number of coordination atoms present in the molecule and can generally form five- or six-membered chelate rings on reactions with metal ions. In addition, if it contains additional functional groups such as -OH, -NH₂, or -CO, the resulting Schiff base can serve as a mixed donor ligand that can participate in bi-, tri-, tetra-, and higher coordination modes [4]. In this paper the synthesis, spectroscopic and antimicrobial studies of Schiff's base derived from 4-chloroaniline and 5-chloro-2-hydroxybenzaldehyde and its cobalt (II) complexes were studied. In addition,

research was also carried out on the chelating behavior of this ligand against transition metal ions Co (II) and its antimicrobial activity for the ligand and its complexes.

II. METHODS AND MATERIAL

All the chemical of analytical grade. All salts are metal nitrates i.e. CO(NO₃)₂·6H₂O (Sigma-Aldrich) were purchased from Sigma-Aldrich and used without further purification. 4-chloroaniline and 5-chloro-2-hydroxybenzaldehyde (Sigma-Aldrich) were purchased from Sigma-Aldrich and used without further purification. Distilled Ethanol used for synthesis of metal complexes and ligand diethyl ether (Sigma-Aldrich). IR Spectra recorded on Perkin Elmer Spectrometer in range 4000-400 cm⁻¹ KBr pellets. ¹H and ¹³C NMR Spectra were recorded on BRUKER AVANCE III HD NMR 500 MHz spectrophotometer. Room Temperature magnetic moments by Guoy's method in B.M. Electronic Spectra using DMSO on Varian Carry 5000 Spectrometer. Molar Conductance measurements in dry DMSO having 1×10⁻³ concentration on Systronics conductivity bridge at room temperature. Elemental analysis (C, H, N) were carried out by using perkin Elmer 2400 elemental analyzer. Mass Spectra were recorded on Bruker IMPACT HD.

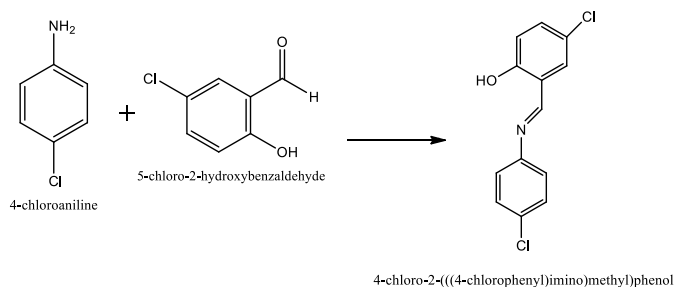
Antimicrobial Activity

The synthesized compounds were screened *in vitro* for their antibacterial activities against bacteria *S. aureus* and *B. subtilis* and antifungal activity against *A. Niger* and *F. Oxysporum* using the Kirby-Bauer disc diffusion method. [20].

Synthesis of 4-chloro-2-(((4-chlorophenyl) imino) methyl) phenol

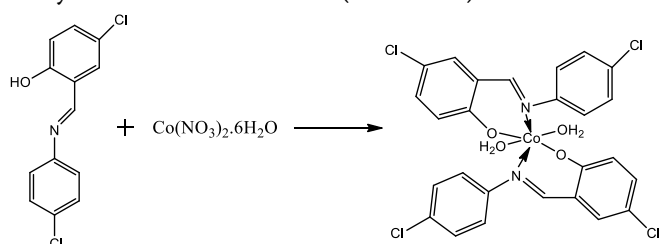
The ligand is synthesized by reported method. [21] The mixture 1:1 of 4-chloroaniline (0.255g, 2 mmol), 5-chloro-2-hydroxybenzaldehyde (0.313 g, 2 mmol) dissolved in 20 ml ethanol in a 50mL round bottomed Flask at room temperature. few drops of glacial

acetic acid was added to the mixture, to adjust its pH to 6 upon which a thick yellow orange precipitate formed instantly. the precipitate was separated by filtration and purified by recrystallization from ethanol. The purity of compound was checked by TLC using Silica Gel method. (Scheme.1).



Scheme 1. Synthesis of Ligand

Synthesis of metal complexes of Co(II):- The schiff base metal (II) complexes (C) (Scheme 2) were prepared by reacting the schiff base with the metal (II) ions as per the literature methods[21].The metal complexes were prepared by mixing of metal nitrates i.e.Co(NO₃)₂. 9H₂O with (25 ml) ethanolic solution of Ligand 4-chloro-2- (((4-chlorophenyl) imino) methyl) phenol in (metal: ligand) 1:2 ratio. The resulting mixture was magnetically stirred and refluxed for 3 hours on an oil bath at 65°C where upon a precipitated product formed. A colored product obtain washed with ethanol, filtered, and recrystallised with ethanol. (Scheme.2).



Scheme 2. Synthesis of metal complexes

III. RESULTS AND DISCUSSION

The ligand 4-chloro-2-(((4-chlorophenyl) imino) methyl) phenol (Scheme 1). and its Metal Complexes of Co(II) is stable at room temperature in solid state (Scheme 2).The schiff base ligand is soluble in hot ethanol and solvents such as DMF and DMSO. The

imine and its metal (II) complexes are coloured solids which are stable in air. the complexes were insoluble in common organic solvents such as methanol, dichloromethane, ethanol, and acetone but soluble in DMSO and DMF. the melting points of the complexes were higher than that of the Schiff base ligand indicating that the complexes are more stable than the ligand.

The physical and analytical data shown in Table1. Spectral evaluation shows formation of ligand and its metal complexes. The synthesized complexes having 1:2 metal to ligand stoichiometric ratio.

Table 1: Proposed Structures of metal complexes M: Co(II)

Entry	product	Time h	Yield %	M.P. °C
Ligand		3-4	90	138-140
MC		5-6	68	>300

Characterization of ligand 4-chloro-2-(((4-chlorophenyl) imino) methyl) phenol

Yellow orange Solid; C₁₃H₉Cl₂NO, M.P.:138°C;Yield:90%;MS:(70eV)m/z:266 [M+H,100%], Anal. Calcd. For C₁₃H₉Cl₂NO C, 58.67; H, 3.41;N, 5.26 Found : C, 56.21; H, 3.11;N, 5.62 Molar Cond. (DMSO 1×10⁻³conc., ohm⁻¹ cm² mol⁻¹):5.12; UV(DMSO, cm⁻¹): 231 (π→π* tran. of benzene ring), 352 (n→π* azomethine moieties and phenolic -OH.); IR(KBr Cm⁻¹):ν =3390 (O-H str. in aromatic ring),ν= 1605 (C=N azomethine), ν=1278 (C-O Phenolic), ¹H NMR (ppm d₆ 400MHZ,DMSO) δ

ppm: δ . 8.85 (s, 1H, CH=N), 11.95 (s, 1H, OH), 7.10–7.35 (m, 7ArH). ^{13}C NMR (DMSO- d_6 , 400 MHz,) δ ppm : 135–122 (Ar, C=C), 161 (HC=N), 129(C-O).

Characterization of metal complexes M: Co(II).

Cobalt (II) Complex, Brown Solid; $\text{C}_{26}\text{H}_{20}\text{Cl}_4\text{CoN}_2\text{O}_4$, M.P.: >300°C; Yield: 68%; MS: (70eV) m/z : 625 [M+H, 100%], Anal. Calcd. For $\text{C}_{26}\text{H}_{20}\text{Cl}_4\text{CoN}_2\text{O}_4$, C, 49.95; H, 3.22; N, 4.48 Found : C, 48.27; H, 3.32; N, 4.52 Molar Cond. (DMSO 1×10^{-3} conc., $\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$): 18.2; UV(DMSO, cm^{-1}): 250, 392, 460 ($\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$, d-d transition); IR(KBr cm^{-1}): ν 3442.4 ν (O-H), 1560.4 ν (HC-N), 1218.1 ν (C-O), 686.4 ν (H₂O), 542.2 ν (Co-N), 472.7 ν (Co-O).

The Schiff base ligand was synthesized using the reflux method. In this research, however, the ligand was prepared using the room temperature method which afforded a higher yield Schiff base ligand is soluble in hot ethanol and solvents such as DMF and DMSO. ligand and its metal (II) complexes are coloured solids which are stable in air. The complexes were insoluble in common organic solvents such as methanol, dichloromethane, ethanol, and acetone but soluble in DMSO and DMF.

The ^1H and ^{13}C NMR spectra of the Schiff base ligand were recorded in DMSO- d_6 . The ^1H NMR spectrum of the Schiff base showed a singlet peak at $\delta = 8.85$ ppm corresponding to the azomethine proton ($-\text{N}=\text{CH}-$) [22], an indication that the Schiff base was formed during the condensation reaction. The expected ^1H NMR peak, at $\delta = 11.95$ ppm, assignable to the phenolic proton in the free Schiff base ligand [23] Observation of a peak at $\delta = 161$ ppm in the ^{13}C NMR Azomethine spectrum was further proof that the ligand was successfully synthesized [24]. The mass spectrum of the Schiff base ligand showed a molecular ion peak at m/z 266 [M+H, 100%], which is consistent with the molecular weight of the Schiff base ligand.

The molar conductance values of the synthesized compounds in 10^{-3}M DMSO were measured at room temperature. the conductance values of the synthesized compounds were below $50 \text{ Ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$, indicating their nonelectrolytic nature [25]. the electronic spectral data of the Schiff base ligand showed three bands at 231 nm due to $\pi \rightarrow \pi^*$ transition of benzene ring), the band appearing at 271.2 nm is assignable to $n \rightarrow \pi^*$ transition of nonbonding electrons present on the nitrogen of the azomethine group. the band at 325 nm is due to $n \rightarrow \pi^*$ transition of the phenolic group [26]. The UV-Vis spectra of the Schiff base Co (II) metal complex. two bands were observed at 250 nm and 460 nm This was a shift towards longer wavelength with respect to the spectrum of the Schiff base ligand. The band at 250 nm was due to intraligand transition, and the band at 460 nm was as a result of d-d transition. which is within the range for octahedral configuration as reported in many octahedral cobalt (II) complexes [27–28].

The binding mode of the Schiff base ligand to the metal ions in complexes was determined by comparing the FT-IR spectrum of the free ligand with the spectra of the metal (II) complexes. The azomethine peak of ligand observed at 1605 cm^{-1} ($-\text{C}=\text{N}-$ azomethine) while in metal complexes it is observed at 1560 cm^{-1} . a shift to lower wave numbers. this is indicated coordination of Schiff base through the azomethine nitrogen [29]. Moreover, the appearance of additional weak bands in the region $536.4\text{--}546.2 \text{ cm}^{-1}$ and $456.3\text{--}478.7 \text{ cm}^{-1}$ attributed to $\nu(\text{M-N})$ and $\nu(\text{M-O})$, respectively [30], further confirmed complexation [31]. This showed that the Schiff base ligand coordinated to the metal via “N” and “O” atoms. The FT-IR spectra of the complexes also showed strong bands in the $3152.2\text{--}3442.4 \text{ cm}^{-1}$ regions, suggesting the presence of coordinated/lattice water in the complexes. This was further confirmed by the appearance of nonligand band in the $682.1\text{--}686.4 \text{ cm}^{-1}$ region, assignable to

the rocking mode of water [31]. In the free Schiff base ligand, the band at 1278 cm^{-1} due to $\nu(\text{C-O, phenolic})$ shifted to lower wave number by $60\text{--}62.1\text{ cm}^{-1}$ in the complexes indicating the coordination of the phenolic oxygen atom to the metal ion [32]. Therefore, it can be concluded that coordination took place via phenolic oxygen and azomethine nitrogen of the Schiff base ligand molecule.

The microanalysis data suggested that all the complexes were mononuclear where two moles of the ligand and two moles of water molecules were coordinated to the central metal atom. The data, therefore, suggested that the metal to ligand ratio in the complex was 1 : 2 the general formula for the complexes as $[\text{M}(\text{L})_2(\text{H}_2\text{O})_2]$ ($\text{M}=\text{Co (II)}$; $\text{L} =$ deprotonated imine) [33]. The theoretical (calculated) values were found to be in good agreement with the experimental values.

Antimicrobial Activity

The antimicrobial activity in vitro on selected two gram positive bacteria *S. aureus* and *B. Subtlis* two fungi *A. niger* and *F. Oxysporum* was carried out shown in table.2. The ability of the Schiff base ligand and its metal (II) complexes to inhibit the growth of the bacteria and fungi was compared to that of the known standard antibacterial and antifungal drugs, Ciprofloxacin and Miconazole respectively. 4-chloro-2-(((4-chlorophenyl) imino) methyl) phenol show substantial antimicrobial activity against bacteria *S. aureus*, *B. Subtlis* and fungal species *A. niger* and *F. Oxysporum*. The results indicated that synthesized metal complexes show good antibacterial and antifungal activity as compared to free ligand against all tested bacteria and Fungi. Overall, the inhibition zones of the Schiff base ligand and its complexes showed that the complexes had enhanced bactericidal activity than the ligand [34].

Table 2. Antimicrobial activity of Imidazole

Compound s	Antibacterial Activity		Antifungal Activity	
	<i>S.aureu s</i>	<i>B.subti lis</i>	<i>A.niger</i>	<i>F.oxyspo rum</i>
	Diamet er of inhibit ion Zone in mm	Diamet er of inhibit ion Zone in mm	Diamet er of inhibit ion Zone in mm	Diameter of inhibitio n Zone in mm
	500 ppm	500 ppm	500 ppm	500 ppm
Ligand	23	22	21	19
MC	29	28	27	24
Ciprofloxac in (Standard)	34	33	----	----
Miconazol e (Standard)	----	----	31	27

IV. CONCLUSION

The Schiff base ligand was prepared. The synthesized Schiff base ligand were formed with Co (II) complex. The structure of the synthesized compounds was elucidated with the help of various spectral and physicochemical techniques. Preliminary in vitro antibacterial and antifungal study indicated that the prepared complex showed slightly higher activity against the tested bacterial and fungal strains as compared to the ligand.

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