ORIGINAL RESEARCH PAPER
Volume - 14 | Issue - 04 | April - 2025 | PRINT ISSN No. 2277 - 8179 | DOI : 10.36106/ijsr
INTERNATIONAL JOURNAL OF SCIENTIFIC RESEARCH

SYNTHESIS AND SPECTRAL CHARACTERIZATION OF SCHIFF BASE LIGAND DERIVED FROM SUBSTITUTED BENZIMIDAZOLE AND ITS COBALT METAL COMPLEX

Chemistry

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ABSTRACT

TheSchiffbase is produced by the condensation reaction of primary amines with carbonyl compounds derivative. Benzimidazole is composed of a benzene ring, which fuses with five members imidazole rings, and is an important heterocyclic connection. Due to its association with a diverse range of activities, benzimidazole holds significant importance in heterocyclic chemistry. This paper discusses the synthesis and spectral characterization of a Co(II) metal complex derived from a Schiff base ligand formed from 5-amino-2-mercapto-benzimidazole and salicylaldehyde. The synthesized Schiff base and its Co(II) metal complex were characterized using multiple techniques, including elemental analysis, molar conductance, FT-IR spectroscopy, NMR, Mass spectrometry, X-ray diffraction, thermo gravimetric analysis, and scanning electron microscopy, along with an assessment of their biological activities. The analytical data indicate a metal to ligand stoichiometry of 1:2.

KEYWORDS

Schiff base, Metal complex, Substituted Benzimidazole, Salicylaldehyde, IR, Mass Spectra, NMR, XRD, SEM.

INTRODUCTION

The Schiff bases are a well-established class of compounds characterized with the functional group (-C=–), named after chemist Hugo Schiff, who first synthesized members of this class in 1864 [1-3]. Schiff base ligands can form complexes with metal ions, positioning them as significant ligands in coordination chemistry [4-6]. These compounds result from the condensation reaction between aromatic or aliphatic aldehydes with amines [7]. The term "Schiff base" is synonymous with "azomethine" [8-9]. Schiff bases serve as powerful chelating ligands for metal complex formation, thanks to their diverse donor groups of sulfur, nitrogen, and oxygen. Their versatility makes them an invaluable tool in coordination chemistry and metal ion interactions. [10-12].

Grasping the complex formation chemistry of transition metal ions is greatly enriched by the study of Schiff bases and its associated metal complexes. In this research, we are excited to present our findings on the preparation, structural characteristics, and comprehensive characterization of a Co(II) metal complex featuring a Schiff base. This work deepens our understanding of these crucial compounds and their potential applications in various fields [13-15].

EXPERIMENTAL

Chemicals

All chemicals utilized in this study were of high-purity analytical grade (A.R.). The metal salt $CoCl_2$. $6H_2O$, 5-amino-2-mercapto-benzimidazole, and salicylaldehyde purchased from E. Merk (India) For the synthesis of the ligand and metal complex.

Synthesis of Schiff Base

An ethanolic solution (100 mL) of 5-amino-2-mercapto-benzimidazole (0.1 M, 1.34 g) was combined with an ethanolic solution (100 mL) of salicylaldehyde (0.1 M, 1.23 g) and the solution molar ratio have 1:1, while stirring continuously and refluxing for 6 hours. After cooling overnight at room temperature, yellow crystals were obtained via filtration and allowed to dry in air.

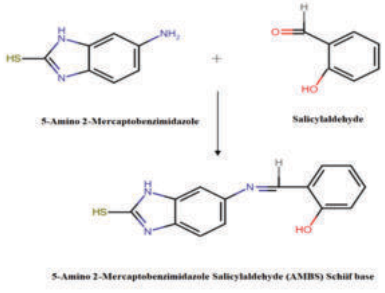

Figure 1. AMBS Schiff Base

Synthesis of the Complex

To prepare the complex, we combined 50 mL of an ethanolic solution containing 10 mmol of metal chloride with an equal volume of Schiff base ethanol solution, maintaining a precise 1:2 molar ratio. This mixture was then subjected to gentle heating in a water bath at 50°C for 3 hours, ensuring optimal conditions for reaction. Afterward, we allowed the mixture to sit undisturbed at room temperature overnight, promoting crystal formation. The final step involved carefully filtering the resulting crystals, followed by thorough washing with water and ethanol, and then air-drying them to preserve their integrity. This meticulous process not only yields high-quality crystals but also enhances the efficiency of future applications.

RESULTS AND DISCUSSION

Table 1. Synthesis and Physico-chemical Characteristics of Schiff Base and its Complex

Sr. No.	Ligand/ Complexes	Colour	% Yield	M.P.° C	Metal/Ligand Ratio
1	AMBS	Digital Yellow	73	119	1:1
2	[Co(AMBS)2]	Algae Green	59	>250	1:2

Table 2. Analytical Data of Ligand AMBS and its Complex

Sr. No.	Molecular Formula (Molecular Weight)	Elemental Analysis (%) Found (Calculated)				
		C	H	N	S	Metal
1	$C_{17}H_{11}N_3SO$ (269.34)	75.74 (75.53)	4.08 (3.93)	15.59 (15.42)	11.88 (11.78)	---
2	$C_{34}H_{20}N_6S_2O_2Co$ (595.61)	68.50 (67.92)	3.35 (3.06)	14.10 (13.68)	10.74 (10.63)	9.89 (8.72)

IR Spectra

The Schiff base IR spectrum displays a sharp band near 1609 cm^{-1}, likely indicative of the azomethine linkage, with a notable decrease in frequency in the metal complex, suggesting coordination of the metal ion through this azomethine bond. The band at 1616 cm^{-1} in the AMBS-Co complex indicates the presence of the CH=N linkage. Additionally, a band observed at 1137 cm^{-1} is characteristic of the C-N linkage, while a band at 1463 cm^{-1} signifies the presence of a chelate ring in the complex. The M-O coordination is supported by the presence of bands at 483 cm^{-1}, while the absorption band at 1383 cm^{-1} indicates the presence of sulfur in the heterocyclic ring. Furthermore, a band at 630 cm^{-1} suggests the M-N linkage. The absence of the phenolic –OH group in the complex further supports its role in complexation.

NMR Spectra

The Schiff base 1H NMR spectrum was recorded in DMSO, revealing two signals at 2.50–2.51 ppm and 3.40 ppm, corresponding to hydrogens from the N-$(CH)_2$ and C-(CH) groups, respectively. The aromatic rings exhibit a series of multi-signals between 6.94–7.66 ppm, while the azomethine hydrogen (HC=N) is found at 8.98 ppm as a

singlet. The peak associated with the phenolic-OH group is noted at 12.64 ppm as a singlet. Notably, NMR spectra were not recorded for the Co(II) ion complex due to its paramagnetic nature, resulting from the presence of unshared odd electrons.

Mass Spectra

Mass spectrometry was conducted to determine the molecular weight and fragmentation patterns of the compounds. High-resolution mass spectra were obtained for free Schiff base ligand and Co(II) complex, as well as for larger fragments related to decomposition products. The ligand demonstrated a precise molecular weight is 269.34 g/mol. Significantly, the spectrum showed a peak at 268 m/z, which corresponds to the new Schiff base moiety, $C_{17}H_{11}N_3SO$, assigned to $[L]^+$. Additional characteristic peaks were observed at 242.6, 216.6, 137.4, and 121.4 m/z, which have been assigned to various fragments. Their intensities suggest the stability of these fragments. The mass spectrum of the $[Co(L)_2]^+$ complex exhibited a peak at 595 m/z, representing the complex moiety $[C_{34}H_{20}N_6S_2O_2Co]^+$.

X-Ray Diffraction Studies

The X-ray diffractogram of Schiff base metal complex was meticulously recorded over a range of 0–80° (2θ), with the diffractogram for the Schiff base illustrated in Figure 2. Notably, major peaks with a relative intensity exceeding 10% were successfully indexed using an advanced computer program. Key diffraction data, including angle (2θ), interplanar spacing (d), and relative intensity (%), are presented succinctly in Table 3. This data compellingly reveals that the Schiff base exhibits sharply defined crystalline peaks, underscoring its crystalline structure. Remarkably, the XRD graph of the complex closely resembles those of the Schiff base, highlighting the consistency and robustness of its crystalline characteristics.

Table 3. X-Ray Diffraction Analysis

Pos. [°2θ]	FWHM Total [°2θ]	d-spacing [Å]	Rel. Int. [%]	Area [cts*°2θ]
27.8392	0.5163	3.20211	15.34	399.92

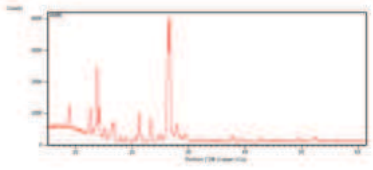

Figure 2. X-ray Diffraction Graph of Schiff Base AMBS

Thermo-Gravimetric Analysis

Thermogravimetric measurements of AMBS and the Co(II) complex were conducted from room temperature to 900°C. The water molecules present as lattice water were typically eliminated at temperatures higher than 150°C, which classifies as coordination waters. The Co(II) complex decomposes through a four-stage process.

Scanning Electron Micrograph Analysis

The Scanning Electron Micrographs (SEM) analysis reveals that Schiff base ligand shows a striking fibrous and grass-like structure, showcasing its unique characteristics. However, the metal complex astounds with its markedly crystalline appearance, a transformation likely resulting from the ligand interaction with metal ions that compresses surface voids. This not only underscores the effectiveness of metal complexation but also highlights the impressive changes that occur at a microscopic level. With an approximate particle size of 10 µm, the Schiff base demonstrates remarkable scalability. Moreover, the distinct morphology of the metal Schiff base complex sets it apart dramatically from the original Schiff base, emphasizing the impact of metallization on structural properties. These observations offer powerful evidence of the transformative effects of metal ion coordination.

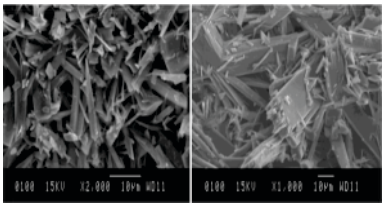

Figure.3. SEM of AMBS Schiff Base Biological Activities

Bacterial cultures of Staphylococcus aureus, Streptococcus mutans, Klebsiella pneumoniae, and Escherichia coli were obtained from the microbiology laboratory. These cultures were carefully incubated at 37°C for 24 hours, following inoculation onto nutrient agar. Schiff bases were stored in a dry environment at room temperature and were subsequently dissolved to a 20 mg/ml concentration in dimethylsulfoxide (DMSO). The antimicrobial activity of the samples, adhering to standard protocols. Various concentrations of 25, 50, and 100 µg/ml were tested for each microorganism in the antimicrobial studies by well diffusion method. A critical aspect of the procedure involved placing the antibiotic-impregnated wells onto the agar surface immediately following inoculation with the tested organism. It is essential to avoid using undiluted overnight broth cultures as inocula. Following 24-hour incubation at 37°C, the plates were meticulously analyzed for distinct zones of inhibition. These clear areas, measured in millimeters, formed around the wells that contained varying concentrations of the respective drugs.

Table 4. Zones of Inhibition for Schiff Base AMBS and Its Metal Complex (mm)

Compounds	Gram Positive Bacteria		Gram negative Bacteria	
	Staphylococcus aureus,	Streptococcus mutans	Klebsiella pneumonia	Escherichia coli
AMBS	7±0.47	7±0.84	7±0.57	7±0.94
AMBS-Co	7±0.32	7±0.94	8±0.56	7±0.34
Cipro-floxacin	20±00	21±0.67	27±0.47	28±0.44

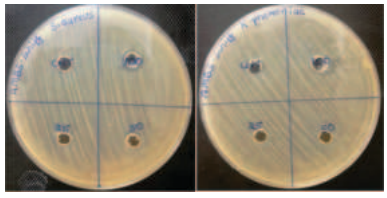

Figure 4. Zones of Inhibition Against Bacteria

CONCLUSION

This study explored the preparation and identification of a Schiff base ligand alongside its Cobalt(II) metal complex through spectroscopic analysis. Our finding reveal that the ligand is neutral and tridentate in its coordination. The complex exhibits a 1:2 (M:L) stoichiometry according to the analytical and spectral data. We confidently propose an octahedral geometry for the complex, underscoring its significance in the field of coordination chemistry.

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