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Synthesis, Characterization, and Antimicrobial Evaluation of Co (II) and Zn (II) Complexes with Schiff Base Derived from Pyrrole-2-carboxaldehyde and Thiosemicarbazide

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Abstract

A novel Schiff base ligand was synthesized from the condensation of substituted thiosemicarbazide and 1H- 1H-pyrrole-2-carboxaldehyde. The corresponding Co(II) and Zn(II) complexes were obtained by refluxing the chloride of the metals with the prepared Schiff base in an ethanolic solution. The Schiff base and its complexes were characterized and analyzed using Fourier Transform Infrared (FT-IR), UV-visible, magnetic susceptibility, conductivity measurement, melting point/decomposition temperature, and solubility test. The Infrared spectral data of the Schiff base showed an absorption band at 1585 cm^{-1}, attributed to ν(C=N) stretching. However, this band was shifted to a higher frequency of 1592 cm^{-1} and 1601 cm^{-1} indicating the formation of a Co-N and Zn-N band in the complexes respectively. The UV-Visible studies revealed significant red shifts in the characteristics C=N and C=S bands upon complexation confirming strong ligand-metal coordination. The complexes exhibited enhanced thermal stability (with decomposition temperatures of 221°C and 210°C for the respective complexes. Magnetic measurements indicated a high-spin octahedral geometry for the Co(II) complex ($\mu_{\text{-eff}}$ =4.56B.M) while Zn(II) complex is diamagnetic with a characteristic tetrahedral geometry. The molar conductance value of the Co(II) complex was observed at 24.8 $ohm^{-1}\,cm^2\,mol^{-1}$ while that of the Zn(II) complex was observed at 3.06 $ohm^{-1}\,cm^2\,mol^{-1}$ suggesting non-electrolytic nature of the complexes. The Schiff base and the complexes were screened for antimicrobial activity against *Staphylococcus aureus, Escherichia coli, Pseudomonas aeruginosa (*Bacterial strains*)*, *Candida albicans*, *Tinea capitis and Tinea pedis (*fungal strains with Ciprofloxacin and Ketoconazole serving as control drugs for bacteria and fungi, respectively. The results demonstrated that metal complexation significantly enhances bioactivity relative to the free ligand, although their activity was lower than that of the standards.

Keywords: antimicrobial activity, complexation, pyrrole-2-carboxaldehyde, Schiff base, synthesis, thiosemicarbazide.

Introduction

Schiff base ligands and their metal complexes have garnered significant interest in coordination chemistry due to their versatile bonding capabilities, structural diversity and wide range of applications in catalysis, electrochemistry and medicine [1]. Schiff bases, characterized by an azomethine (-C=N) functional group, are typically synthesized via the condensation of primary amines with carbonyl compounds. Their ability to coordinate with transition metals leads to the formation of stable metal complexes with tunable electronic, magnetic, and biological properties [2].

Among Schiff base ligands, thiosemicarbazone derivatives exhibit enhanced coordination flexibility due to the presence of multiple donor atoms such as N, S or O stabilizing metal centers in different oxidation states [3]. Additionally, incorporating heterocyclic moiety such as Pyrrole can significantly alter the electronic properties of the ligands leading to enhanced redox activity, biological interactions and unique optical characteristics [4].

Metal-based drugs have gained attention as alternatives to traditional organic molecules, particularly in antimicrobial, anticancer and antioxidant properties due to their ability to interact with biomolecules such as DNA and proteins [5], [6]. Metal-based drugs have played a significant role in medicinal chemistry, particularly in the treatment of cancer and infectious diseases. The incorporation of metal ions into drug molecules has led to improved pharmacokinetics, enhanced biological activity and targeted delivery mechanisms [7].

One of the major advantages of the metal-based drugs is their ability to interact with biomolecules in a manner distinct from purely organic drugs. For instance, platinum-based drugs like cisplatin and carboplatin function by forming DNA adducts leading to apoptosis in cancer cells [8]. Despite these advantages, challenges remain in optimizing the selectivity and minimizing the side effects of metal-based drugs.

Recent efforts in drug design have focused on developing theranostic metal complexes, compounds that combine therapeutic and diagnostic functions to improve patient outcomes and personalized medicine approaches [9]. With increasing research on Schiff base ligands derived from thiosemicarbazide and pyrrole-2-carboxaldehyde, there is growing potential for the development of novel metal complexes with enhanced bioactivity and reduced toxicity [10]. Studies suggest that metal complexes with pyrrole–containing Schiff bases exhibit increased reactive oxygen species (ROS) generation, disrupting bacterial and fungal cell membranes [11]. In particular, Co (II) and Zn (II) complexes have been reported to show potent biological activity due to their ability to catalyze oxidation reactions in the cellular environment [12]. Consequently, the research aimed at synthesizing novel Schiff base ligand and to prepare its corresponding Co(II) and Zn(II) complexes and to evaluate the antimicrobial activity of these complexes against bacterial and fungal strains, thereby establishing structure-activity relationships for potential therapeutic applications.

Materials and Methods

All chemicals and solvents used were of analytical grade and were purchased from Sigma-Aldrich. All glass wares used were washed with detergent thoroughly, rinsed with distilled water, and dried in an oven at 110^0C. Thiosemicarbazide derivative (T33405) and 2-pyrrolecarboxaldehyde (P73404) were also obtained from Sigma Aldrich and were used without further purification. Weighing was conducted using Metler balance model AB54. Melting point and decomposition temperatures were recorded using the Gallenkamp SMP10 (Gallenkamp Instrumente GmbH, Germany) melting point apparatus.

Synthesis of ligand

Ethanolic solution of 2-pyrrolecarboxaldehyde (0.952 g, 10 mmol) was added in hot ethanolic solution (20 ml) of thiosemicarbazide (0.911 g, mmol) with a few drops of acetic acid with constant stirring. The mixture was refluxed at 80°C for 2 hours. On cooling, a silver grey colored compound precipitated out. It was then filtered, washed several times with 50% ethanol, dried and recrystallized from methanol [13].

Synthesis of metal complexes

The Schiff base ligand (3.36 g, 20 mmol) was dissolved in ethanol (20 ml) under stirring to which a solution of the corresponding metal salts (2.38g, 10mmol) for Co(II) and (1.71 g,10mmol) for Zn(II) metal salts in ethanol (20 ml) was added dropwise. The reaction mixture was refluxed for 4 hours at 80°C until a colored precipitate was formed. The solid was collected, washed with ethanol and dried in a desiccator [13].

Molar Conductivity Measurement of the Thiosemicarbazone Complexes in DMSO

Conductivity was measured using a Jenway conductivity meter (model 4010, United Kingdom) to determine the ionic nature of the complexes.

Magnetic Susceptibility Measurement

Magnetic moments were determined using Sherwood magnetic susceptibility balance to measure the electronic configuration of the metal centers.

Infrared Spectral Analysis of the Thiosemicarbazone and its Metal (II) complexes

Infrared spectral analysis was recorded using Fourier Transform Infrared Spectrometer (CARY 630 Agilent technology, United States) in the range 4000-400 cm^{-1} to confirm functional group formation and metal coordination.

UV-Visible Spectral Analysis of the Thiosemicarbazone and its Metal (II) Complexes

The electronic spectra of the ligand and its complexes were recorded between 200-600nm at room temperature using Perkin-Elmer Lambda 35(Massachusetts, USA) to observe d-d transition and ligand-metal charge transfer bands %.

Job's Method of Continuous Variation

This technique is used to determine the stoichiometry of the metal complexes by mixing equimolar solutions of the Schiff base and the metal salts in varying ratios. The absorbance was measured using UV-Visible spectroscopy and a plot of molar fraction against absorbance was plotted.

Gravimetric Method for Chlorine determination in the Complexes

The metal complex (0.2 g) was dissolved in 10ml concentrated nitric acid (approximately 70% HNO_3) solution to release chloride ions which were then precipitated as silver chloride (AgCl) using a standardized $AgNO_3$ solution. The AgCl precipitate was filtered, washed, dried, and weighed. The mass measured of the AgCl allowed for the calculation and confirmation of the presence of the coordinated chloride in the complexes.

Antimicrobial Assay

The antibacterial and antifungal activity tests of the synthesized thiosemicarbazone and its respective complexes were carried out against three pathogenic bacterial isolates *Staphylococcus aureus, Escherichia coli,* and *Pseudomonas auriginosa*, and three fungal isolates *Candida albicans*, *Tinea capitis* and *Tinea pedis* using well diffusion methods as reported by Mohammad *et al*. [11].

Results and Discussion

The results obtained from the physicochemical analysis, spectral characterization, and *in-vitro* antimicrobial evaluation of the thiosemicarbazones and their complexes were presented in Tables 1-6.

Table 1: Percentage yield and some Properties of Thiosemicarbazone and its Metal (II) Complexes

Compounds	Colour	Melting pt. (^{0}C)	Decomposition temp(^{0}C)	Yield (%)
Ligand, L	Silver grey	179		73.18
$[CoL_2.Cl_2].3H2O$	Dark brown	-	221	71.45
$[ZnL_2 Cl_2].4H_2O$	Grey	-	210	69

L=$C_6H_8N_4S$

Table 2: Conductivity measurement of Co(II) and Zn(II) Complexes in 1×10^{-3} DMSO

Compounds	Specific conductivity Ω^{-1} cm^{-1}	Molar conductivity Ω^{-1}cm^{2}mol^{-1}
$[CoL_2.Cl_2].3H2O$	24.8×10^{-6}	24.8
$[ZnL_2 Cl_2].4H_2O$	3.06×10^{-6}	3.06

L=$C_6H_8N_4S$

Table 3: Magnetic Susceptibility data of Co(II) and Zn (II) Complexes

Compounds	Xg (g^{-1})	Xm (mol^{-1})	µeff (B.M)	Magnetic property
$[CoL_2.Cl_2].3H2O$	1.4806×10^{-6}	8.6280×10^{-3}	4.53	Paramagnetic
$[ZnL_2 Cl_2].4H_2O$	-5.1023×10^{-7}	-2.7388×10^{-4}	-ve	Diamagnetic

L=$C_6H_8N_4S$

Table 4: Solubility test of Thiosemicarbazone and its Metal (II) Complexes in various solvents

Compounds	Distilled water	Ethanol	DMF	DMSO	n-Hexane	Diethyl ether
Ligand, L	Insoluble	Sparingly soluble	Soluble	Soluble	Sparingly soluble	Sparingly soluble
$[CoL_2.Cl_2].3H2O$	Insoluble	Sparingly soluble	Soluble	Soluble	Insoluble	Insoluble
$[ZnL_2 Cl_2].4H_2O$	Insoluble	Sparingly soluble	Soluble	Soluble	Insoluble	Insoluble

L=$C_6H_8N_4S$

Table 5: Infrared Spectral data of Thiosemicarbazone and its Metal (II) Complexes

Compds	ν(OH) cm^{-1}	ν $(NH)_{sec}$ cm^{-1}	ν $(NH)_{pri}$ cm^{-1}	ν (C=N) cm^{-1}	ν (C=S) cm^{-1}	ν (C-S) cm^{-1}	ν(M-N) cm^{-1}	ν (M-S) cm^{-1}	ν (M-Cl) cm^{-1}
Ligand,L	-	3443	3260	1618	1552	817	-	-	-
$[CoL_2.Cl_2].3H_2O$	3410	3237	3127	1590	1508	830	704	408	348
$[ZnL_2 Cl_2].4H_2O$	3313	3268	3166	1601	1540	827	728	521	443

$L=C_6H_8N_4S$

Table 6: UV-Visible Spectral data of Thiosemicarbazone and its Co(II) and Zn(II) Complexes

Compds	n-π*(nm) C=S	n-π*(nm) C=O	n-π*(nm) C=N
Ligands, L	295	327	383
$[CoL_2.Cl_2].3H_2O$	279	328	380
$[ZnL_2 Cl_2].4H_2O$	333	343	350

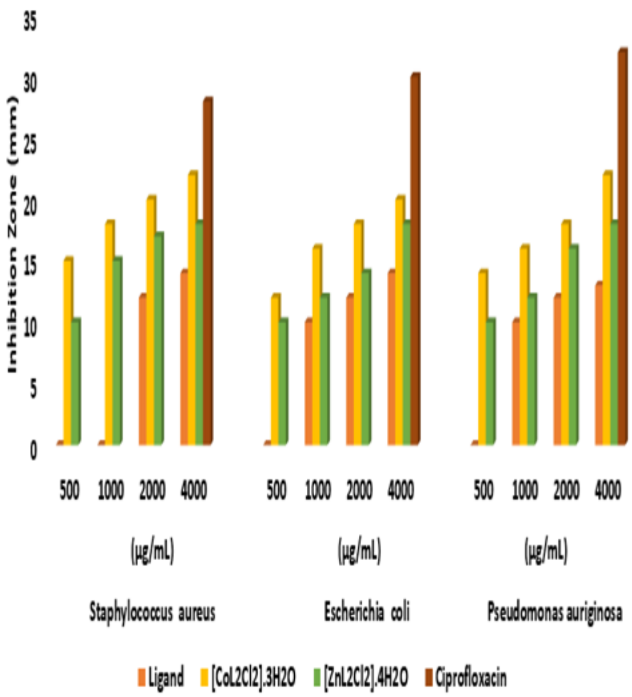

Fig 1. Bar chart showing the antibacterial activity of the Thiosemicarbazone and its complexes.

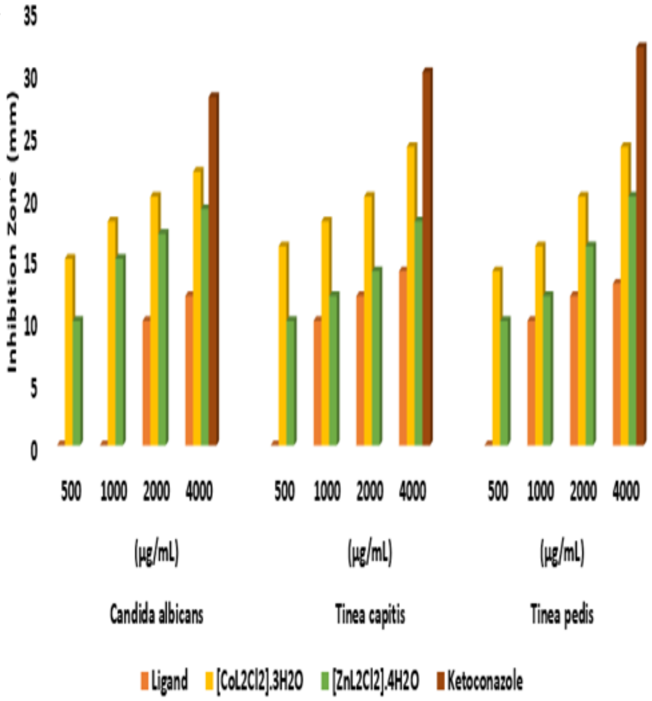

Fig 2. Bar chart showing the antifungal activity of the Thiosemicarbazone and its complexes.

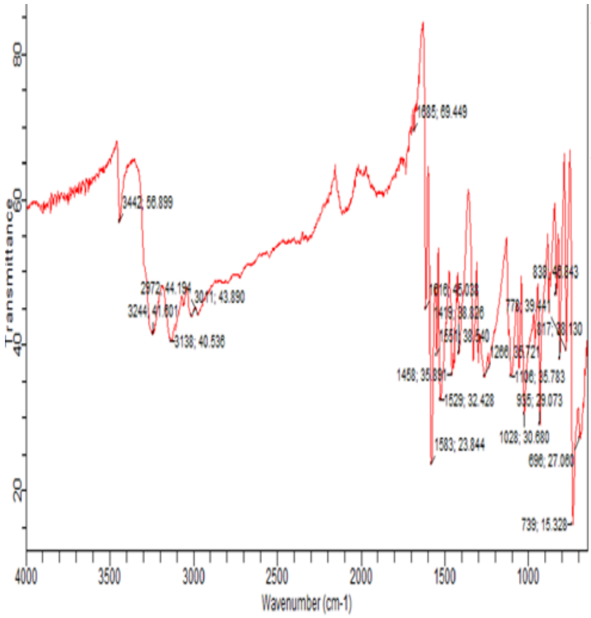

Fig 3. FTIR Spectrum of thiosemicarbazone

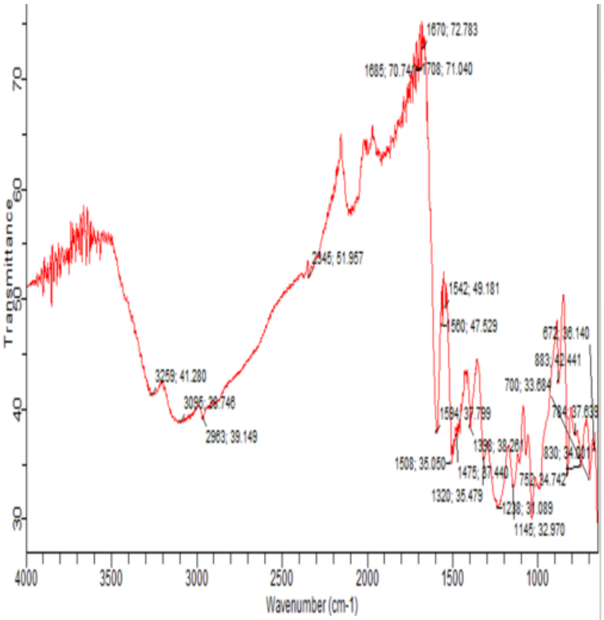

Fig 4. FTIR Spectrum of $[CoL_2Cl_2].3H_2O$ complex

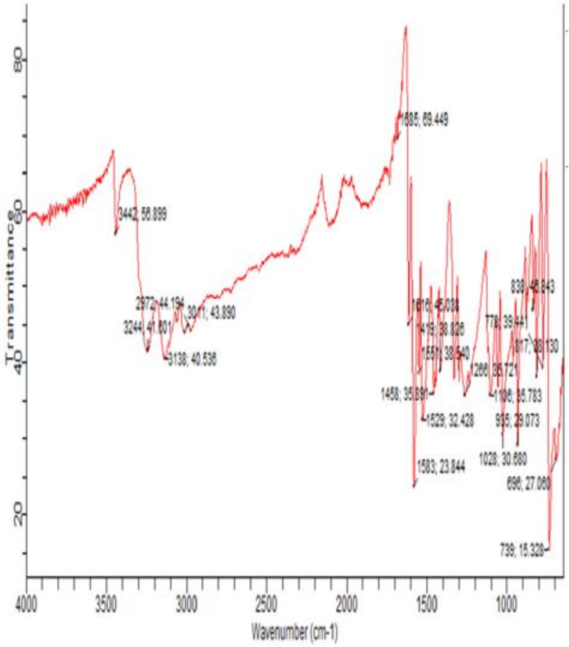

Fig.5. FTIR Spectrum of Thiosemicarbazone

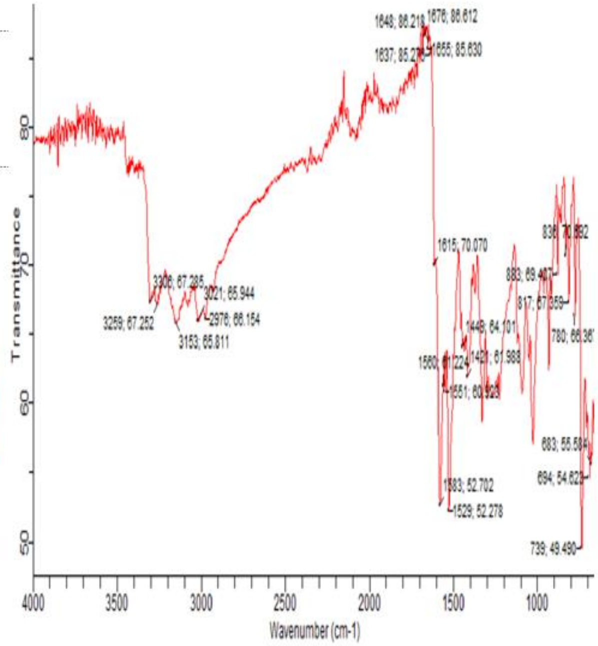

Fig.6. FTIR Spectrum of $[ZnL_2Cl_2].4H_2O$ Complex

Table 1 shows the physicochemical properties of the thiosemicarbazone and its metal (II) complexes. The thiosemicarbazone appears silver grey, indicating a neutral electronic environment with minimal conjugation effects. The Co(II) complex is dark brown while the Zn(II) complex is grey indicating a strong ligand–to–metal charge transfer. The colour variation is likely due to metal-ligand interaction, ligand field effect, and hydration contribution [14], [15], [16] and [17]. The Co(II) complex decomposed at 221^{0}C compared to the Zn(II) complex (210^{0}C). The higher decomposition temperature indicates a more stable coordination environment as similarly reported by Abdullah *et al.* [18] and Jones *et al.* [19]. The thiosemicarbazone yield (73.10%) indicates an efficient condensation reaction between the pyrrole-2-carboxaldehyde and thiosemicarbazide and also proves the reaction is economically feasible and promising. Both synthesized complexes have good percentage yields 71.45% and 69% respectively. These yields are within the range for thiosemicarbazone metal complexes Sharma *et al*. [20], Rahman *et al*. [21].

Table 2, for the molar conductance measurement of the metal (II) complexes was carried out in DMSO. The values obtained for the respective complexes were 24.80 and 3.06 $\Omega^{-1}cm^2mol^{-1}$. These low molar conductance values suggested the non-electrolytic behaviour of the complexes with the chloride ions likely coordinated to the metal center rather than existing freely in solution. This is consistent with literature reports on similar Schiff base metal complexes. Studies have shown that complexes with inner–sphere chloride exhibit molar conductance values, 50 $\Omega^{-1}cm^2mol^{-1}$ whereas complexes that dissociate into free ions display significantly higher values (often > 70 $\Omega^{-1}cm^2mol^{-1}$). Such low conductance values have been reported for Schiff base complexes by Singh *et al.*, [22] and Qureishi *et al*. [6]. The magnetic moment values, as shown in Table3, suggested that the Co(II) complex with an observed µeff value of 4.53 B.M indicated three unpaired electrons in the octahedral high-spin environment which is in agreement with previously reported values for Co(II) Schiff base complexes with the spin-only value in the range, 4.3-5.3 B.M Sharma *et al.* [20], Kumar *et al*. [23]. While Zn(II) is diamagnetic with d^{10} electronic configuration suggesting a tetrahedral geometry. This is in agreement with similarly reported literatures, Singh *et al*. [24], Suleiman *et al.* [25].

Table 4 Shows the solubility test result of the thiosemicarbazone and its metal (II) complexes in various solvents. The thiosemicarbazone Schiff base was found soluble in DMF and DMSO due to strong dipole interactions Reichardt and Welton, [26]. The insolubility of the Schiff base in water, n-hexane and Diethyl ether is probably due to the non-ionic nature and lack of hydrophilic groups, which indicates the non-electrolytic nature of the Schiff base. The metal (II) complexes retain a similar solubility trend. The findings are in agreement with similar reported literature by Li *et al*. [27], and Zhao *et al.* [28].

Table 5 shows the infrared spectral data of the thiosemicarbazone and its complexes. The ν(N-H) stretching bands for secondary and primary amines appear at 3443 cm^{-1} and 3260 cm^{-1} respectively in the spectra of the thiosemicarbazone as indicated in Fig. 3 and Fig. 5 shifts to 3237 cm^{-1} and 3127 cm^{-1} in Co(II) complex and 3268 cm^{-1} and 3166cm^{-1} in spectra of the Zn(II) complex which is an indication of coordination through the nitrogen of the azomethine. The ν(C=N) stretching vibration at 1618 cm^{-1} which indicates the presence of an azomethine group, shifted to a lower frequency of 1590 cm^{-1} and 1601 cm^{-1} in the spectra of the complexes due to coordination through the nitrogen atom of the azomethine group. Similarly, ν(OH) stretching at 3410 cm^{-1} and 3313 cm^{-1} suggests the presence of water molecules. New absorption bands at 704 cm^{-1}, 408 and 348 cm^{-1} in Co(II) and 728 cm^{-1}, 521 cm^{-1} and 443 cm^{-1} in the spectra of the complexes were due to M-N, M-S, and M-Cl respectively as shown in Fig. 4 and Fig. 6. This confirms the coordination of the thiosemicarbazone to the metal ions. This observation agrees with studies on similar Schiff base metal complexes confirming coordination through azomethine nitrogen and thiocarbonyl sulphur atoms [29].

UV-visible spectra of the thiosemicarbazone and that of its complexes were recorded in Table 6. For the thiosemicarbazone absorption bands at C=S(295nm), C=O(327nm) C=N(383nm) corresponds to n→π* transitions. The complexes showed high intensities electronic transitions at 279 and 333(n→π*, C=S), 328 and 343(n→π*, C=O), 380 and 350nm (n→π*,C=N).These shifts in absorption bands to lower λ_{max} (blue shift) or higher λ_{max} (red shift) in the spectra of the complexes are due to coordination of the thiosemicarbazone to the metal ions. These findings are in agreement with reported studies on transition metal Schiff base complexes by Karthika *et al.* [30]. Fig 1 and 2 show the *in vitro* antibacterial and antifungal activities of the thiosemicarbazone and its metal (II) complexes. The compounds were tested against three clinically tested pathogenic bacteria (*Staphylococcus aureus, Escherichia coli,* and *Pseudomonas auriginosa*) and three fungal isolates (*Candida albicans, Tinea capitis* and *Tinea pedis*). The results indicated that the metal complexes exhibit higher antibacterial and antifungal activities than the free thiosemicarbazone. The incorporation of metal ions into the ligand enhances the activity of the compound thereby increasing the ligand's liposolubility and also is concentration dependant, that is, increases generally with increase in concentrations. But their activities are comparable with that of control as indicated in Figures 1 and 2. Co(II) complex showed the highest inhibition against all tested strains particularly *Staphyllococcus aureus* and *Candida albicans* where it showed activity comparable to the standard drugs. These findings are in agreement with previous reports that Co (II) complexes, due to their redox-active nature, exhibit enhanced cell membrane disruption and enzyme inhibition compared to Zn(II) complexes [24].

Conclusion

A Schiff base ligand and its metal (II) complexes were successfully synthesized and characterized using various physicochemical techniques and spectral analysis. Both the Schiff base and the resulting compounds were produced in considerable yields and displayed notable thermal stability. The low conductivity observed in the complexes suggests their non-electrolytic nature, confirming chloride coordination within the inner coordination sphere. Measurements of effective magnetic moments indicate that the Co(II) complex is paramagnetic while Zn(II) complex is diamagnetic.. Infrared spectra exhibited all absorption bands at the anticipated wavenumbers, while electronic spectra revealed n→π* transitions at expected wavelengths. Furthermore, results from Job's method and gravimetric analysis indicate a metal-ligand ratio of 1:2, supporting the proposed structural model. The antimicrobial activity showed significant enhancement upon complexation, underscoring the potential pharmaceutical applications of these complexes.

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