Synthesis, Characterization and Biological Studies of Schiff Base Complexes of Fe(II), Cd(II), Hg(II) and Co(II) Derived from Lysine and Salicylaldehyde

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ABSTRACT: This study reports the synthesis and characterization of Fe(II), Cd(II), Hg(II) and Co(II) complexes with a Schiff base derived from lysine and salicylaldehyde. Elemental analysis confirmed successful synthesis. UV-Vis spectroscopy revealed significant electronic transitions and metal-to-ligand charge transfers. Thermogravimetric analysis showed distinct thermal decomposition patterns, indicating the stability and composition of the complexes. Magnetic susceptibility measurements indicated paramagnetic behavior for Fe(II) and Co(II) complexes, suggesting tetrahedral geometry, while Cd(II) and Hg(II) complexes were diamagnetic. X-ray diffraction analysis revealed a crystalline primitive lattice structure for the Fe(II) complex, with the Cd(II), Hg(II) and Co(II) complexes being amorphous. The Co(II), Cd(II) and Hg(II) complexes demonstrated substantial antibacterial activity against both gram-positive and gram-negative bacteria. Additionally, the Cd(II) and Co(II) complexes exhibited strong activity against all tested fungal strains. All complexes also showed promising antioxidant activity.

Key words: Schiff base metal complexes, spectral studies, antibacterial activities, antifungal activities, antioxidant activities.

INTRODUCTION

Metal complexes, particularly those involving transition metals, have significant interest in coordination chemistry due to their versatile chemical properties and wide range of applications.[1–4] Schiff bases, formed by the condensation of primary amines with carbonyl compounds are known to form stable complexes with various metal ions.[5,6] These complexes often exhibit unique structural, electronic and magnetic properties .[5–7]

In this study, we focus on the synthesis and characterization of metal complexes involving Fe(II), Cd(II), Hg(II) and Co(II) with a Schiff base derived from lysine and salicylaldehyde. The choice of these metal ions is motivated by their diverse electronic configurations and coordination behaviors, which offer a comprehensive platform for exploring structure-property relationships.[8–12] Fe(II) and Co(II) are known for their paramagnetic properties due to unpaired electrons in their d-orbitals, while Cd(II) and Hg(II) typically exhibit diamagnetic behavior due to their filled d-orbitals.[8–14]

The complexes were synthesized and subjected to a range of analytical techniques to elucidate their structures and properties. Elemental analysis was performed to confirm the metal content, while UV-Vis spectroscopy provided insights into their electronic transitions and potential charge transfer mechanisms. Infrared (IR) spectroscopy was used to study the vibrational modes and confirm the coordination of the ligands to the metal centers. Thermogravimetric analysis (TGA) offered information on the thermal stability and decomposition patterns of the complexes. Magnetic susceptibility measurements provided data on the magnetic properties of the complexes, shedding light on their electronic configurations and coordination environments. Finally, X-ray diffraction (XRD) studies were conducted to determine the crystallinity and lattice structures of the complexes.

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The results from this research revealed significant differences in the solubility, electronic spectra, thermal stability, magnetic properties and crystallinity of the complexes. Such comprehensive characterization not only enhances our understanding of the fundamental chemistry of these metal-ligand systems but also opens avenues for their potential applications in fields such as catalysis, material science and medicine. Additionally, the observed antibacterial, antifungal and antioxidant activities of the Schiff base metal complexes against a range of pathogens highlight their potential impact in biological and pharmaceutical research.

MATERIALS AND METHODS

Iron(II) sulphate heptahydrate ($FeSO_4.7H_2O$), Cadmium perchlorate hexahydrate ($CdCl_2O_8.6H_2O$), Mercury(II) chloride ($HgCl_2$), Cobalt(II) chloride hexahydrate ($CoCl_2.6H_2O$) and various solvent were used in this study. All the chemicals were analytical grade and received from E-Merck and Sigma Aldrich.

The complexometric titration method for metal determination involves preparing a stock solution by digesting 0.05 g of the metal complex with nitric acid at ~100°C, then cooling and diluting it in a 50 ml volumetric flask. For cadmium determination, 10 ml of the solution is diluted to 50 ml, buffered with 1 ml ammonia-ammonium chloride (pH 9-10) and titrated with standard EDTA (~0.01 M) using Eriochrome Black T indicator until the color changes from radish-yellow to violet. The cobalt determination follows a similar procedure, using Murexide indicator, observing a color change from radish-yellow to violet. For mercury, xylenol orange indicator is used, with the color change from yellow to red. In iron determination, o-mercaptobenzoic acid indicator is employed, noting the color change from blue to yellow. Each titration requires the solution to be buffered to maintain the desired pH and prevent precipitation, ensuring accurate endpoint detection.

The characterization of the metal complexes was conducted using various analytical techniques. Infrared spectra were recorded using a Shimadzu FTIR spectrophotometer (IRPrestige-21) in the range of 400-4000 cm^{-1} with KBr pellets. Ultraviolet-visible (UV-Vis) spectral analysis was performed using a Shimadzu UV-visible spectrophotometer (UV-1650 PC) with 1 cm path length quartz cells. Thermogravimetric analysis (TGA) was conducted with a Shimadzu TGA-50 analyzer, using a platinum pan, from 30°C to 800°C at a rate of 10°C/min under nitrogen atmosphere with a flow rate of 10 ml/min and a 5-minute hold at the final temperature. X-ray powder diffraction (XRD) analysis was carried out using a Rigaku XRD-53 analyzer with Cu Kα radiation. Magnetic susceptibility was measured with a SHERWOOD SCIENTIFIC Magnetic Susceptibility Balance (Magway MSB Mkl) based on a stationary sample and magnets.

Synthesis of the complex. The metal-Schiff base complexes of Fe(II), Cd(II), Hg(II) and Co(II) were synthesized using the following procedure. Initially, 1 mmol of lysine was dissolved in 15 ml of ethanol and placed in a 50 ml conical flask. Potassium hydroxide (1 mmol) was dissolved in 10 ml of ethanol in another conical flask. The solution was then added to the lysine solution to deprotonate and stabilize the amine group, with the mixture being heated and stirred continuously for 20 minutes. Separately, 1 mmol of salicylaldehyde was dissolved in 15 ml of ethanol and placed in a refluxing flask. The lysine-potassium hydroxide mixture was added dropwise to the salicylaldehyde solution using a burette and the combined solution was heated and stirred continuously for 40 minutes to form the Schiff base.

Following this, an aqueous solution of the respective metal salt was prepared by dissolving 1 mmol of the metal salt in 10 ml of water. This metal salt solution was added dropwise to the Schiff base solution using a burette, and the resulting mixture was refluxed and stirred for 4 hours to facilitate the formation of the metal complex. The reaction yielded precipitates—orange for the Fe(II) and Hg(II) complexes, and yellow for the Cd(II) and Co(II) complexes—which were then filtered and washed several times with water before being dried at room temperature.

RESULTS AND DISCUSSION

The synthesized metal complexes exhibit distinct solubility characteristics in various solvents. Fe(II), Cd(II), and Hg(II) are soluble in dimethyl sulfoxide (DMSO), indicating a preference for polar solvents. These complexes are sparingly soluble in ethanol and hot ethanol, reflecting limited solubility in moderately polar solvents. However, they are insoluble in water, hot water and n-hexane, suggesting poor solubility in highly polar and nonpolar solvents. Additionally, Co(II) complex is soluble in DMSO and sparingly soluble in ethanol, hot ethanol, and n-hexane, but insoluble in water and hot water. These solubility patterns highlight the varying interactions between the complexes and the solvents based on their polarity and chemical nature.

Metal contents of the complexes were determined by complexometric method. For the complex Fe(II) complex, the experimentally determined metal content was 16.88%, close to the calculated value of 17.33%. The Cd(II) complex showed a metal content of 29.08%, slightly lower than the calculated 29.67%. Similarly, the Hg(II) complex had a measured metal content of 41.16%, which is marginally below the calculated 42.63%. Lastly, for the Co(II) complex, the metal content was found to be 18.02%, compared to the calculated 18.11%. These results indicate that the experimental values closely match the theoretical calculations, confirming the successful synthesis and composition of the metal complexes.

The electronic spectra of the metal complexes are shown in figure 1. The UV-visible spectra of the complexes revealed distinct λ_{max} values: Fe(II) complex at 355 nm, Cd(II) complex at 380 nm, Hg(II) at 320 nm and 390 nm, and Co(II) at 385 nm. The ligand displayed characteristic bands attributed to π-π^* transitions indicating metal-to-ligand charge transfer. Despite expectations of d-d transitions above 500 nm, their absence in the spectra was attributed to their low intensity.

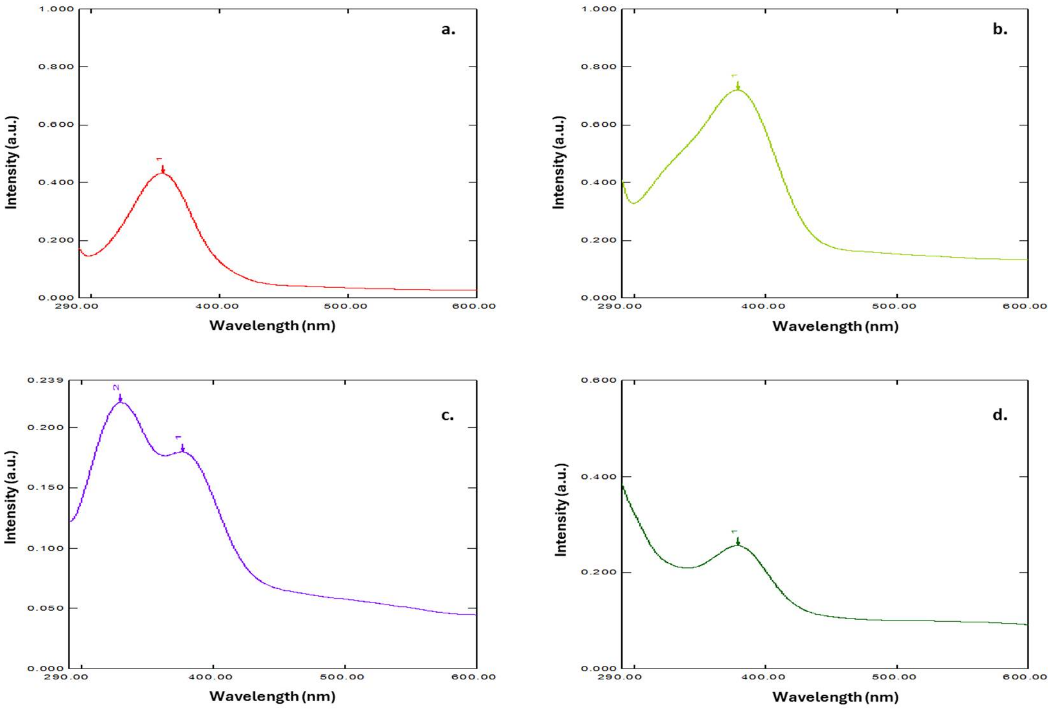


Figure 1. UV-vis spectrum of (a) iron(II) complex, (b) cadmium(II) complex, (c) mercury(II) complex and (d) cobalt(II) complex.

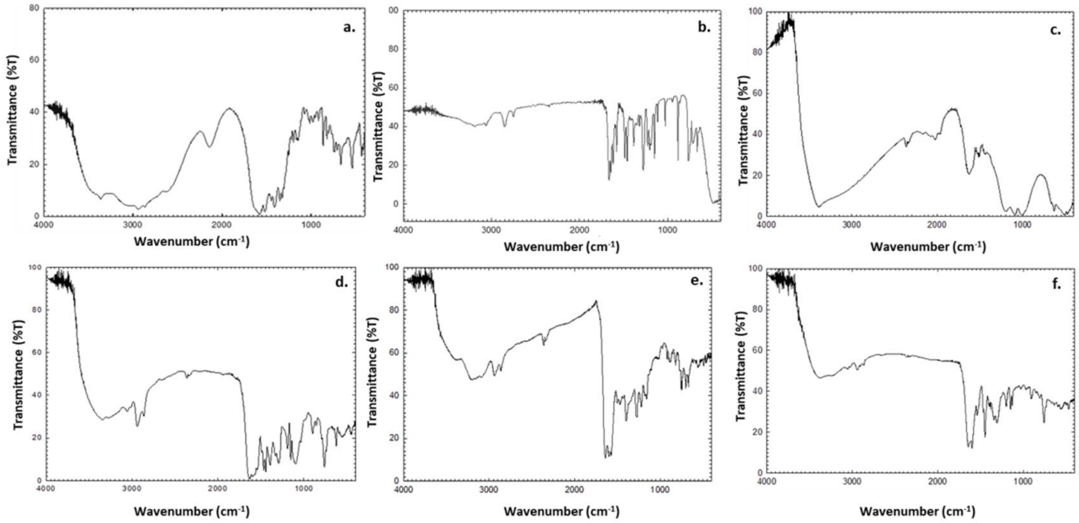


Figure 2. FTIR spectrum of (a) lysine, (b) salicylaldehyde, (c) iron(II) complex, (d) cadmium(II) complex, (e) mercury(II) complex and (f) cobalt(II) complex.

The FTIR spectra of lysine, salicylaldehyde, and their metal complexes revealed significant vibrational features that confirm their molecular structures and coordination behavior (Figure 2). The FTIR spectrum of lysine showed broad absorption between 3400-3000 cm^{-1}, indicating N-H stretching from amine groups, while bands around 1650-1500 cm^{-1} suggested amine bending and potential C=O stretching, which supports the presence of carboxyl groups. The fingerprint region also showed C-N and C-C skeletal vibrations, characteristic of lysine's structure. Salicylaldehyde's spectrum features a broad peak at 3500-3300 cm^{-1}, indicating O-H stretching from the phenolic hydroxyl group, while a sharp peak around 1700 cm^{-1} was attributed to C=O stretching from the aldehyde group. Additionally, peaks in the 3000-2800 cm^{-1} range corresponded to C-H stretching and the C=C stretches near 1600-1500 cm^{-1} confirmed its aromatic structure.

The Fe(II) complex exhibited broad O-H and N-H stretching bands in the 3400-3000 cm^{-1} region, along with a prominent C=O stretching band near 1600 cm^{-1}, confirming the presence of water, lysine and salicylaldehyde as ligands. The Cd(II) complex showed O-H and N-H stretching between 3400-3000 cm^{-1}, C-H stretching between 3000-2800 cm^{-1} and C=O stretching around 1700-1600 cm^{-1} along with C=C stretching in the 1600-1500 cm^{-1} region suggesting aromatic and aliphatic ligand coordination. The Hg(II) complex similarly displayed N-H, O-H and C-H stretching vibrations, with C=O and C=C stretching bands, supporting a ligand environment containing carbonyl groups and aromatic rings. The Co(II) complex showed N-H and O-H stretching between 3400-3000 cm^{-1} and C=O or N-H stretching near 1600-1500 cm^{-1}. The lower frequencies (below 1000 cm^{-1}) of the FTIR indicated M(II)-N or M(II)-O bonding. The consistent observation of O-H, N-H and C=O stretching, along with the C=C stretching of aromatic rings, confirmed the presence of lysine and salicylaldehyde in these metal complexes. These spectral features collectively suggested the coordination of both ligands with transition metals, forming stable metal-ligand frameworks in the respective complexes.

The TGA results indicated the stability and decomposition behavior of these metal complexes, with distinct stages corresponding to the loss of coordinated water, lysine and other ligands (Figure 3). For the Fe(II) complex, significant weight loss at 150.12°C, attributed to the loss of coordinated water (5.27% experimental vs. 5.59% calculated). Further weight loss up to 425.36°C corresponded to the decomposition of lysine (42.53% experimental vs. 44.45% calculated). Even further weight loss continued up to 716.72°C, resulting in the loss of salicylaldehyde (32.06% experimental vs. 32.62% calculated). The Cd(II) complex started losing weight at 129.8°C, with a weight loss corresponding to the loss of coordinated water (4.56% experimental vs. 4.71% calculated). Up to 363.36°C, there was a significant weight loss due to the removal of lysine (37.05% experimental vs. 37.87% calculated). Further decomposition up to 670.08°C resulted in the loss of salicylaldehyde (26.62% experimental vs. 27.77% calculated).

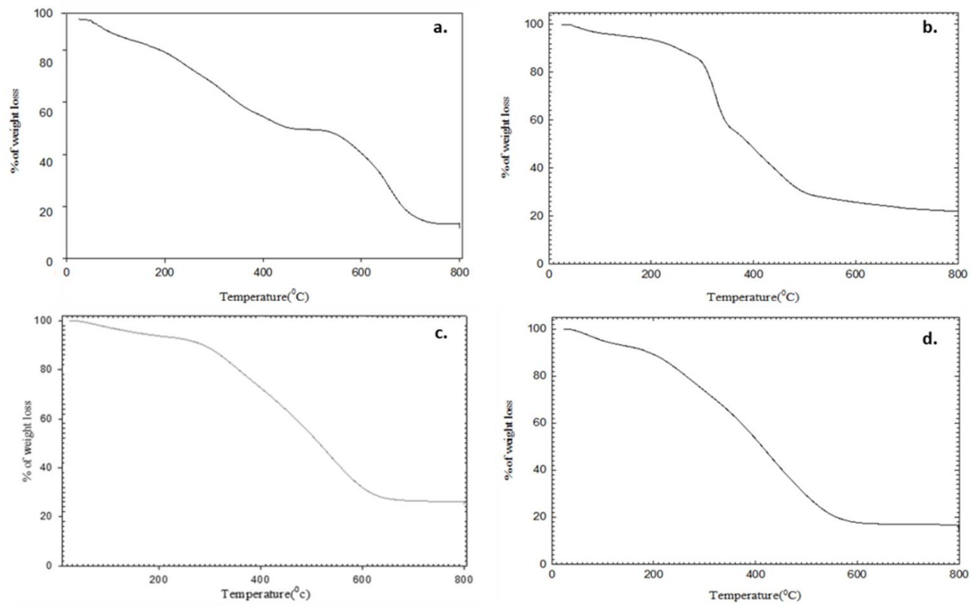


Figure 3. TGA curve of (a) iron(II) complex, (b) cadmium(II) complex, (c) mercury(II) complex and (d) cobalt(II) complex.

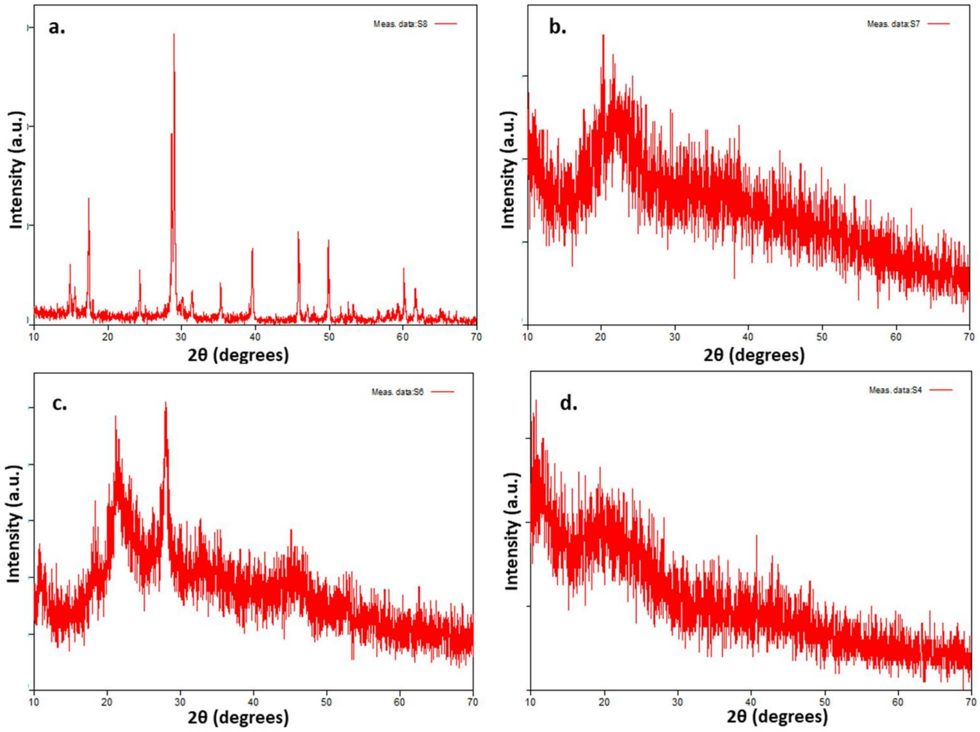
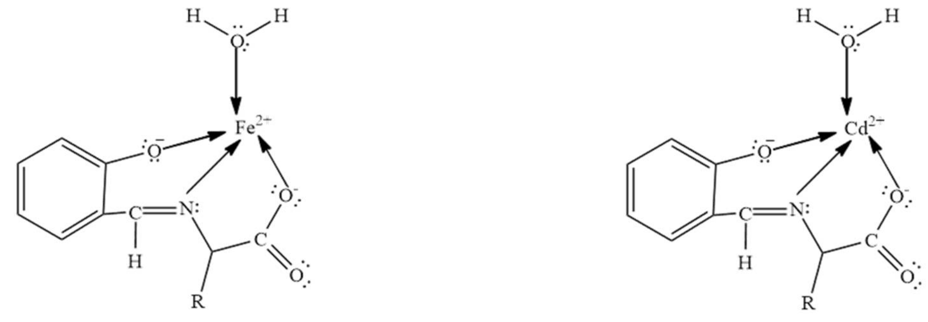


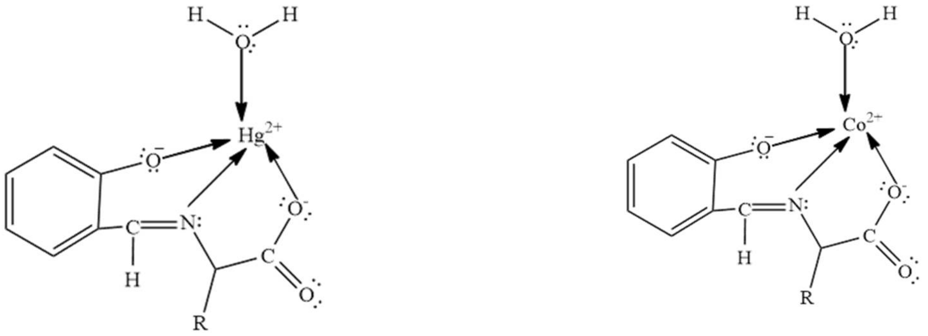
Figure 4. XRD pattern of (a) iron(II) complex, (b) cadmium(II) complex, (c) mercury(II) complex and (d) cobalt(II) complex.

For the Hg(II) complex, rapid weight loss started at 150.83°C, corresponding to the loss of coordinated water (3.66% experimental vs. 3.86% calculated). From 200.28°C to 497.27°C, weight loss was attributed to the loss of lysine (30.15% experimental vs. 30.63% calculated). Continued weight loss up to 685.88°C resulted in loss of salicylaldehyde. The Co(II) complex began losing weight at 166.8°C due to the loss of coordinated water (5.14% experimental vs. 5.54% calculated). Up to 411.68°C, weight loss was attributed to the loss of lysine (42.95% experimental vs. 43.88% calculated). Further weight loss up to 676.64°C was due to the loss of salicylaldehyde (32.25% experimental vs. 32.30% calculated).

The magnetic susceptibilities of Fe(II), Cd(II), Hg(II) and Co(II) complexes were measured, revealing diamagnetic nature for Hg(II) and Cd(II) complexes and paramagnetic behavior for Fe(II) and Co(II) complexes. The Fe(II) and Co(II) complexes were paramagnetic due to unpaired electrons in their 3d orbitals, with magnetic moment values of 1.53 BM and 4.59 BM, respectively, indicative of tetrahedral geometry. The observed magnetic behavior aligned with the expected coordination environments and electron configurations. The study underscores the influence of ligand strength on complex properties, reflected in their magnetic susceptibilities and colors. The X-ray diffraction patterns (Figure 4a) of the Fe(II) complex revealed sharp peaks while the Cd(II), Hg(II), and Co(II) complexes displayed broad and featureless patterns, indicating an amorphous nature without distinct crystalline peaks (Figure 4b-c). The ratio of the values of $\sin^2\theta$ of the Fe(II) complex satisfied the conditions of being a primitive lattice. While, the Fe(II) complex exhibited crystallinity with a primitive lattice structure, the Cd(II), Hg(II), and Co(II) complexes were found to be amorphous (Figure 4b-c). Based on the findings of this research, the most probable structures of the Fe(II), Cd(II), Hg(II) and Co(II) complexes are shown in figure 5.



(a) $[Fe(C_{13}H_{16}N_2O_3)(H_2O)]$ (b) $[Cd(C_{13}H_{16}N_2O_3)(H_2O)]$



(c)$[Hg(C_{13}H_{16}N_2O_3)(H_2O)]$ (d) $[Co(C_{13}H_{16}N_2O_3)(H_2O)]$

Figure 5. Most probable structure of (a) iron(II) complex, (b) cadmium(II) complex, (c) mercury(II) complex and (d) cobalt(II) complex.

Table 1. Antibacterial and antifungal activities of the complex.

Antibacterial activity					
	Diameter of the zone of inhibition (mm)				Standard 400 µg/disc
Name of bacteria	Fe(II) Complex 400 µg/disc	Cd(II) Complex 400 µg/disc	Hg(II) Complex 400 µg/disc	Co(II) Complex 400 µg/disc	
Gram positive bacteria					
Bacillus cereus	-	30	10	30	30
Bacillus megaterium	-	30	9	25	35
Bacillus subtilis	-	15	30	31	35
Staphylococcus aureus	-	32	10	27	40
Micrococcus luteus	-	20	14	20	40
Gram negative bacteria					
Escherichia coli	-	35	25	25	30
Pseudomonas aeruginosa	-	32	10	25	40
Salmonella paratyphi	-	30	10	38	38
Salmonella typhi	-	39	17	40	38
Vibrio mimicus	-	35	11	25	37
Vibrio parahemolyticus	-	35	10	15	32
Shigella dysenteriae	-	20	12	25	43
Shigella boydii	-	24	10	30	40
Antifungal activity					
Saccharomyces cerevacae	-	26	-	30	30
Candida albicans	-	30	10	22	30
Aspergillus niger	-	30	11	40	25

The Fe(II), Cd(II), Hg(II) and Co(II) complexes were evaluated for their antibacterial activity against various gram-positive and gram-negative bacteria, using ciprofloxacin as a standard and DMSO as the solvent control (Table 1). The complexes were tested against eight gram-negative bacteria, including *Escherichia coli* (*E. coli*), *Pseudomonas aeruginosa* (*P. aeruginosa*), *Salmonella paratyphi* (*S. paratyphi*), *Salmonella typhi* (*S. typhi*), *Vibrio parahaemolyticus* (*V. parahaemolyticus*), *Vibrio mimicus* (*V. mimicus*), *Shigella dysenteriae* (*S. dysenteriae*), *Shigella boydii* (*S. boydii*), and five gram-positive bacteria, namely *Bacillus cereus* (*B. cereus*), *Bacillus megaterium* (*B. megaterium*), *Bacillus subtilis* (*B. subtilis*), *Staphylococcus aureus* (*S. aureus*), *and Micrococcus luteus (M. luteus).* Table 1 showed that Cd(II), Hg(II), and Co(II) complexes showed significant antibacterial activity against all tested microorganisms. However, the Cd(II) complex exhibited the highest antibacterial activity against *S. typhi* (39 mm), while the Hg(II) complex was most effective against *B. subtilis* (30 mm). In comparison, the Co(II) complex demonstrated the strongest antibacterial activity against *S. typhi* (40 mm). For comparison, the activity of the standard drug ciprofloxacin was also included in table 1. While the biological activity of the Cd(II), Hg(II) and Co(II) complexes in this study was consistent with other Schiff base complexes reported in the literature, their antibacterial activity against *S. typhi* surpassed previously reported values.[3,15-17] Notably, this activity even slightly exceeded that of the standard antibiotic ciprofloxacin (Table 1), highlighting the potential of the complex as potent antimicrobial agent. This suggested that the unique properties of the complexes may significantly enhance their efficacy against this pathogen.

The antifungal activity of Fe(II), Cd(II), Hg(II) and Co(II) was assessed using a protocol similar to that for antibacterial screening, with each sample and the standard fluconazole tested at a concentration of 400 µg/disc as shown in table 1. Both Cd(II) and Co(II) complex displayed consistent and high antifungal activity across all tested fungi. These findings highlighted the potential of these metal complexes as effective antibacterial and antifungal agents.

Table 2. IC_{50} values of the complex.

Name of sample	IC_{50} value
Fe(II) Complex	44.70
Cd(II) Complex	46.03
Hg(II) Complex	34.15
Co(II) Complex	32.53
BHT	63.55

The free radical scavenging activity of the synthesized metal complexes was evaluated using tert-butyl-1-hydroxy toluene (BHT) as a standard antioxidant (Table 2). IC_{50} values, representing the concentration required to achieve 50% inhibition, were determined for each synthesized complex. The Fe(II), Cd(II), Hg(II) and Co(II) complexes demonstrated significant antioxidant potential, with IC_{50} values of 44.70, 46.03, 34.15 and 32.53 µM respectively.

CONCLUSION

This study successfully synthesized Schiff base metal complexes of Fe(II), Cd(II), Hg(II), and Co(II) using lysine and salicylaldehyde. Through comprehensive characterization techniques including elemental analysis, thermogravimetric analysis, infrared spectroscopy, UV-visible spectroscopy, magnetic susceptibility measurements, and X-ray powder diffraction studies, the complexes were structurally elucidated. The coordination behavior of lysine with salicylaldehyde Schiff base was confirmed, revealing a tridentate ligand coordinating with metal ions via nitrogen and oxygen atoms. The Co(II), Cd(II) and Hg(II) complexes exhibited significant activity against both gram-positive and gram-negative bacteria while Cd(II) and Co(II) complex showed high antifungal activity across all tested fungi. Additionally, all the complexes showed promising antioxidant activity. Further investigations are warranted to elucidate their precise mechanisms of action and potential implications.

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