

Research Article

Microwave-assisted Synthesis, Characterization and Antibacterial Study of Schiff Base Metal (II) Complexes Derived from 2-Aminophenol and *P*-chlorobenzaldehyde

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Abstract

The growing resistance of microorganisms to existing antibiotics has increased the need for new and more effective antimicrobial agents. In this study, Schiff base ligand was synthesized from 2-aminophenol and *p*-chlorobenzaldehyde, and its cobalt (II) and lead (II) complexes. The compounds were characterized using melting point determination, solubility tests, Ultraviolet–visible (UV–Vis), Fourier Transform Infrared spectroscopy (FTIR), Elemental analysis, and Magnetic susceptibility measurements. The FTIR spectrum of the ligand showed a characteristic band at 1610 cm⁻¹ due to the formation of azomethine (C=N) group, confirming Schiff base formation. In the metal (II) complexes, this band shifted to lower frequencies, 1638 and 1612 cm⁻¹ indicating coordination of the azomethine nitrogen to the metal ions. The O–H stretching band at 3429 cm⁻¹ for ligand shifted to 3257 and 3047 cm⁻¹ upon complexation, suggesting involvement of the phenolic oxygen atom. UV–visible and magnetic data indicated an octahedral geometry around Pb(II) and tetrahedral around Co(II). Elemental analysis revealed the expected molecular compositions of the prepared Schiff base ligand. Antibacterial studies using *Staphylococcus aureus* and *Escherichia coli* showed that the metal complexes displayed higher inhibitory activity against the tested bacterial strains compared with the free ligand. This enhancement suggests that coordination with metal ions increases the biological effectiveness of the Schiff base. Overall, the results indicated formation and characterization of the Schiff base ligand and its cobalt (II) and lead (II) complexes, demonstrating their potential as promising antimicrobial agents.

Keywords

Schiff Base, Metal (II), 2-aminophenol, Antibacterial, Spectroscopy

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1. Introduction

Schiff base ligands and their metal complexes have become important in coordination chemistry due to their ease of synthesis, structural diversity, and wide range of applications [11, 39]. The presence of the azomethine ($-C=N-$) linkage allows Schiff bases to act as good donors to metal ions, forming stable chelate rings [3, 7, 31, 38]. The importance of coordination compounds has significantly increased in recent years. Metal complexes play crucial roles in homogeneous and heterogeneous catalysis [5] materials chemistry (including metal-organic frameworks and coordination polymers) [8] photochemistry and optoelectronics [39] as well as biomedicine (such as diagnostics and metal-based nanomedicines used in anticancer and antimicrobial therapies) [6, 10, 12, 16, 24, 35]. The significance of Schiff base metal complexes stems from their diverse applications across biological, industrial, and environmental sectors. Schiff bases possess a strong metal-binding capacity, stability, and versatile donor characteristics due to the presence of the azomethine ($-C=N-$) group, making them highly effective ligands in coordination chemistry [20, 23]. Schiff bases containing oxygen and nitrogen donor atoms are especially attractive for forming stable metal complexes that exhibit significant, biological activities such as antimicrobial, anticancer, antifungal, antioxidant and anti-inflammatory properties [14, 30, 37]. In this study, we presented the synthesis of Schiff base metal complexes using, (E)-2-[(4-chlorobenzylidene) amino] phenol Schiff base ligand, Co (II), and Pb (II). There is an increasing abundance of drug-resistant pathogens despite continuous efforts by researchers to mitigate the resistance; hence, synthesis and characterization of Schiff base derive from 2-aminophenol and chlorobenzaldehyde. The synthesized compounds were characterized using spectroscopic techniques and their antibacterial properties were examined.

2. Material and Methods

All chemicals and reagents utilized in this research were of analytical quality and were employed without additional purification processes. Chemicals used for the preparation of Schiff base Ligands include: 2-aminophenol, *p*-chlorobenzaldehyde (Sigma Aldrich), sulphuric acid; cobalt (II) and lead (II) salts (obtained from central Laboratory, Bamidele Olu-milua University of Education Science and Technology, Ikere Ekiti. Fourier Transform Infrared (FTIR) spectra ($4000-400\text{ cm}^{-1}$) using KBr discs were recorded with Cary 630 FTIR. Magnetic susceptibility measurements were carried out at room temperature on a Gouy balance using Swissmake-H-1640 with maximum capacity of 80 g. Electronic spectra were recorded using SpectraLab 7525 spectrophotometer in the range of 0-400, 400-900 for ligand and complexes. Elemental analysis was determined using Perkin Elmer, USA 2400 series

II. Antibacterial study was performed on two organisms; *Staphylococcus aureus* and *Escherichia coli* obtained from the Federal Medical Centre, Owo, Ondo State. Agar well diffusion method is used to evaluate the antimicrobial activity the prepared compounds against bacterial cultures. The synthesized compounds were dissolved separately in DMSO to prepare stock solutions for the antimicrobial testing. The resulting solutions were subsequently diluted with DMSO where necessary, to obtain the required concentration of $10\text{ }\mu\text{g}$ per test sample. Ciprofloxacin was used as the control drug for the antibacterial study.

2.1. Synthesis of Schiff Base Ligand

The Schiff base ligand (L) was prepared by reacting, 2-aminophenol (1.50 g, 0.0137 mol.) with *p*-chlorobenzaldehyde (1.93g, 0.0124 mol) in ethanol (20 ml) under constant stirring at room temperature, little drops of sulphuric acid was introduced to aid the condensation process. This reaction in ethanol was stirred for 20 minutes, the mixture was then subjected to microwave irradiation for 6 minutes at $50-70^\circ\text{C}$ as shown in Figure 1. The resulting gold coloured precipitate formed was filtered, washed and re-crystallized in warm ethanol. The product obtained was dried in an oven.

2.2. Synthesis of Cobalt (II) and Lead (II) Complexes

2.2.1. Synthesis of Cobalt(II) Complex

The Schiff base ligand (0.80 g, 0.00345 mol) was dissolved in ethanol and mixed with an ethanolic solution of cobalt(II) nitrate hexahydrate, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.50 g, 0.00172 mol), in a 2:1 molar ratio of ligand to Co(II). The reaction mixture was stirred for 20 min and subsequently subjected to microwave irradiation for 8 min at $60-80^\circ\text{C}$. A pink-coloured precipitate of the Co(II) complex was obtained. The precipitate was filtered, washed thoroughly with ethanol to remove unreacted materials and impurities, and dried in air overnight.

2.2.2. Synthesis of Lead (II) Complex

The Schiff base ligand (0.80 g, 0.00345 mol) was dissolved in ethanol and mixed with an ethanolic solution of lead(II) acetate trihydrate, $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ (0.64 g, 0.00169 mol), at a 2:1 molar ratio of ligand to metal. The reaction mixture was stirred for 20 min and subsequently subjected to microwave irradiation for 8 min at $60-80^\circ\text{C}$. A cream-coloured precipitate of the Pb(II) complex was formed. The precipitate was filtered, washed with ethanol to remove unreacted materials and other contaminants, and dried overnight in air.

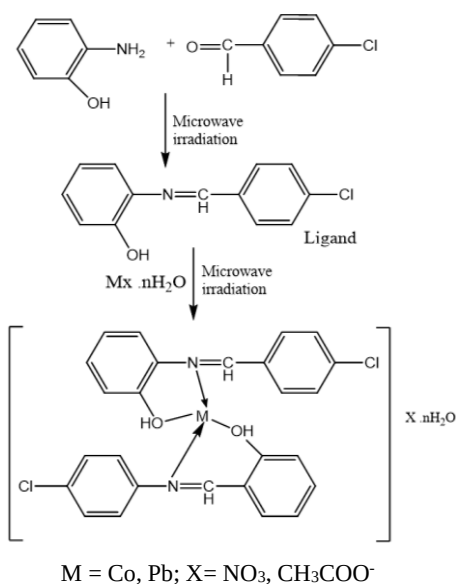


Figure 1. Synthesis of Schiff base ligand and its metal complexes.

3. Results and Discussion

As shown in Table 1, the Schiff base ligand dissolved readily in ethanol, and dimethyl sulfoxide (DMSO); in contrast, the metal (II) complexes were soluble only in DMSO. The difference in solubility behavior between the ligand and its metal complexes may be attributed to coordination and increased molecular weight after complexation, which reduces their ability to dissolve in most organic solvents. Elemental analysis of prepared ligand was carried out to determine the mass fraction of the elements present in the compounds. The observed values are in agreement with those calculated based on the proposed molecular formula.

Table 1. Analytical data and Physical properties of Schiff base ligand and its complexes.

Compound	Empirical formula / Colour	Yield (%)	M.P (°C)	M.wt. (g)	Solubility	Elemental analysis (found/calc)			
						C	H	N	O
Ligand	C ₁₃ H ₁₀ NCIO Gold	72	200	232	DMSO/Et hanol	67.31 (67.24)	4.38 (4.31)	5.84 (6.04)	6.70 (6.90)
[Co(L) ₂]	Co[C ₂₆ H ₂₀ Cl ₂ N ₂ O ₂] Pink	64	218	522	DMSO	58.52 (59.79)	3.45 (3.86)	5.63 (5.36)	6.38 (6.13)
[Pb(L) ₂]	Pb[C ₂₆ H ₂₀ Cl ₂ N ₂ O ₂ (H ₂ O) ₂] Cream	57	222	706	DMSO	43.46 (44.20)	2.84 (3.42)	3.58 (3.96)	8.67 (9.06)

3.1. Infrared Spectra Analysis of the Compound

The relevant FT-IR spectra band assignments of the ligand and the metal complexes of cobalt (II) and lead (II) are shown in Figures 2-4. These techniques provide insights into the electronic transitions, bonding modes, and functional group interactions that validate successful complexation between the ligand and the metal ions [21]. The spectrum of the free Schiff base ligand showed a band at 1610 cm⁻¹, corresponding to the azomethine (C=N) stretching vibration, confirming the formation of the Schiff base [3, 29, 37]. In the spectra of the Co(II) and Pb(II) complexes, this band shifted to 1638 and 1612 cm⁻¹ respectively, indicating coordination of the azomethine nitrogen atom to the metal ions [1, 4, 9, 18, 26]. A broad absorption band observed at 3429 cm⁻¹ in ligand shifted to 3257 and 3047 cm⁻¹ in Co(II) and Pb(II) complexes, assignable to O-H stretching vibration. This suggests participation of the phenolic oxygen in coordination with the metal center via O-H group [13]. The FTIR spectrum of the free Schiff base ligand and Co(II) complex

showed no δ(H₂O) stretching vibration, indicating that they are both anhydrous, and contained no coordinated water molecules. Appearance of a new band at 851 cm⁻¹ in the spectrum of Pb(II) suggested coordination of water molecule [25]. Two new bands in the far-IR region at 432 and 542 cm⁻¹ in the spectra of Co(II) complex corresponding to ν(M-O) and ν(M-N) vibrations, while the new bands at 445 and 546 cm⁻¹ in the spectra of Pb(II) complex indicating ν(M-O) and ν(M-N) vibrations [1, 4, 26, 29].

3.2. Electronic Spectroscopy

The UV-visible spectral analysis also provided strong evidence for the coordination and geometry of the complexes. The free ligand exhibited an intense band around 273 nm, corresponding to π→π* within the aromatic and azomethine systems (10) Upon complexation, new bands appeared at higher wavelengths for both Co(II) and Pb(II) complexes, which are characteristic of d-d transitions and charge transfer processes. For the cobalt complex, absorption bands at 301nm indicating

$n \rightarrow \pi^*$, a charge-transfer (CT) band at 412 nm and d-d absorption at the visible region around 560 nm of less intensity indicating a distorted tetrahedral environment of the ligand around the Co ion due to $^4A_2(F) \rightarrow ^4T_1(P)$ transition [15, 33]. This geometry is in agreement with magnetic moment value in the range of 4.2 - 4.8 BM expected for tetrahedral Co [2, 17, 22]. Similarly, Pb(II) complex displayed bands at 287, 540, and

701 nm, and magnetic moment of 0 B.M., indicating $\pi \rightarrow \pi^*$, $^2B_{1g} \rightarrow ^2B_{2g}$ and $^2B_{1g} \rightarrow ^2E_g$ transitions for distorted octahedral geometry [19, 27]. These findings agree with previous studies that describe Co(II) and Pb(II) Schiff base complexes as exhibiting tetrahedral or pseudo-octahedral coordination environments [32, 34].

Table 2. Electronic data of the ligand (L) and its complexes.

Compound	Wavelength λ_{max} (nm)	Absorption band (cm^{-1})	M_{eff} (B.M)	Transition (s) band assignment	Geometry
Ligand (L)	273, 318	36630, 31446	—	$\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$	—
Pb(L) ₂	287, 540, 701	34843, 18518, 14265	0, B.M	$\pi \rightarrow \pi^*$, d $\rightarrow$ d	Distorted Octahedral
Co(L) ₂	301, 412, 560	33222, 24271, 17857	4.21 B.M	$n \rightarrow \pi^*$, d $\rightarrow$ d	Tetrahedral

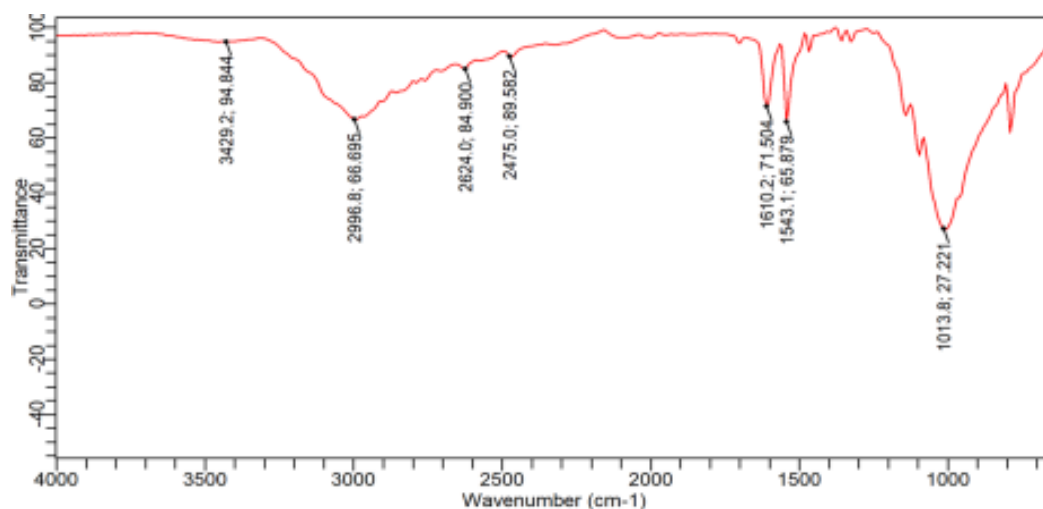


Figure 2. FTIR spectrum of the ligand (L).

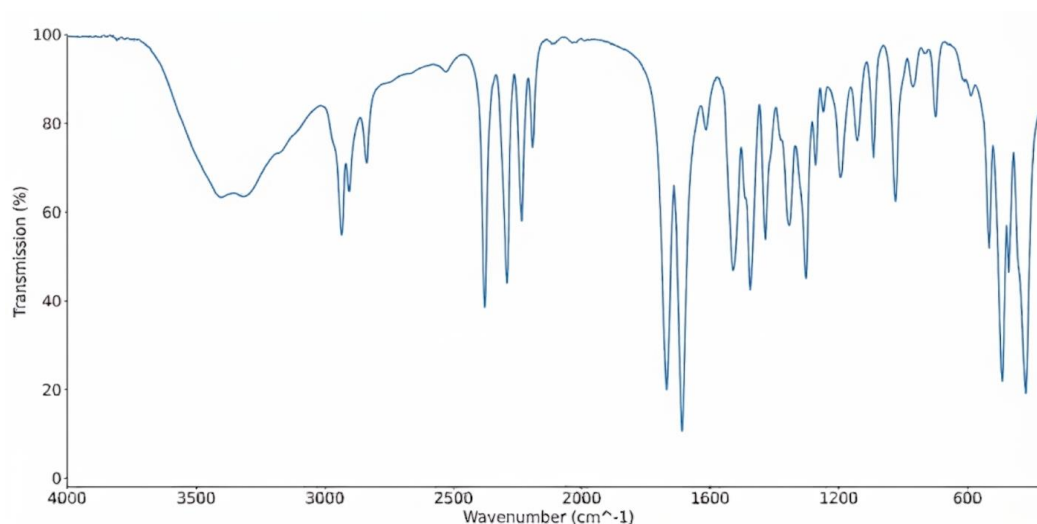


Figure 3. FTIR spectrum of Co(L)₂.

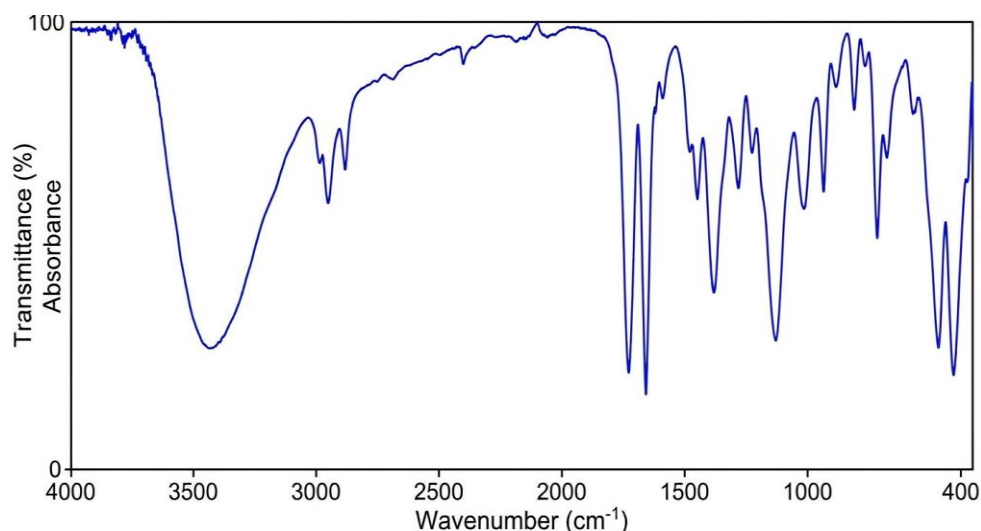


Figure 4. FTIR spectrum of $Pb(L)_2$.

3.3. Antibacterial

The Schiff base ligand and its metal (II) complexes were screened against selected microorganisms, the zones of inhibition are tabulated in Table 3. The Schiff base is moderately active and its activity arises from the presence of an imine and chloro groups in its structure [2, 7, 36, 39]. The activity of cobalt (II) was found to be almost the same with the standard drug Ciprofloxacin for *Staphylococcus aureus*. From the biological study, the antibacterial screening revealed that both metal complexes exhibited higher inhibitory activity against the tested microorganisms compared to the free ligand (18, 29). This enhancement can be explained by the chelation theory, which postulates that coordination reduces the polarity of the metal ion through partial sharing of its positive charge with donor atoms and increases the lipophilic character of the complex. Cobalt (II) complex demonstrated the most significant antibacterial activity, which may be linked to its higher stability and effective coordination geometry. The order of activity, *Escherichia coli*: ciprofloxacin (28) > Pb(L) (19) > Co(L) (15) > Ligand (10); *Staphylococcus aureus*: ciprofloxacin (30) > Co(L) (24) > Pb(L) (18) > Ligand (12). These findings agree with reports that Co(II) Schiff base complexes often exhibit superior biological performance compared with their corresponding ligands and other metal analogues [11, 24, 28].

Table 3. Antimicrobial activity of ligand and its metal complexes at (10 μ g).

Compound	<i>Staphylococcus aureus</i>	<i>Escherichia coli</i>
Ligand (L)	12	10
Co(L)	24	15
Pb(L)	18	19

Compound	<i>Staphylococcus aureus</i>	<i>Escherichia coli</i>
Ciprofloxacin	30	28
Mean	21	18

Zone of inhibition (mm)

Gram - positive. Gram - negative

4. Conclusion

The study synthesized and characterized Schiff base ligand derived from 2-aminophenol and p-chlorobenzaldehyde and its corresponding cobalt (II) and lead (II) complexes using FTIR, UV-VIS magnetic susceptibility measurement. The spectroscopic results indicated coordination through both the azomethine nitrogen and phenolic oxygen donor atoms, while magnetic and UV-visible data suggested tetrahedral geometry for cobalt (II) complex. The lead (II) complex was found to be diamagnetic, consistent with its $6s^2$ electronic configuration and closed-shell structure. Elemental data confirmed the proposed formulations of the prepared. Biological studies revealed that the metal complexes displayed higher antimicrobial activity compared with the free ligand, demonstrating chelation enhances the lipophilic character and biological effectiveness of Schiff base compounds. The prepared complexes could serve as good candidates for pharmaceutical applications.

Abbreviations

BM	Borhn Magneton
DMSO	Dimethyl Sulfoxide
L	Ligand
Pb (L)	Lead (II) Complex
Co (II)	Cobalt (II)

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Author Contributions

Kehinde Ezekiel Ajibulu: Conceptualization, Formal Analysis, Supervision, Writing – original draft, Writing – review & editing

Barizomdu Tina Pii: Supervision, Writing – review & editing

Victoria Ibukun Adebisi: Formal Analysis, Writing – review & editing

Rashidat Adebukola Nasiru: Conceptualization, Formal Analysis, Writing – original draft, Writing – review & editing

Data Availability Statement

The data presented in this study are available on request from the corresponding author

Conflicts of Interest

The authors declared no conflict of interest.

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