



Research Article

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Pharmacological Activities of a Synthesized Schiff Base and its Polymer Complexes

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ABSTRACT

A Schiff base was prepared via condensation reaction of 4-chlorobenzaldehyde, 2-hydroxybenzaldehyde, and *p*-phenylenediamine, and its Co(II), Ni(II), and Cu(II) polymeric complexes were obtained in similar manner.  $^1\text{H}$  and  $^{13}\text{C}$ NMR, FT-IR, UV-Vis, CHN, ICP, and conductivity measurements were utilized for structural investigation. The  $^1\text{H}$ NMR confirmed the formation of the azomethine by detecting two singlet peaks of the protons that are associated with C=N groups, and the peaks related to the carbons of the azomethine groups were revealed by  $^{13}\text{C}$ NMR. The FT-IR also disclosed sharp strong bands at 1680 and 1537  $\text{cm}^{-1}$  in the spectrum of the SB ligand, and in the spectra of the polymeric complexes these bands shifted to higher or lower frequencies, proving the coordination to the ligand. The UV-Vis has clearly shown abroad absorption bands at 255 and 339 nm for the synthesized SB ligand, accredited to the  $\pi \rightarrow \pi^*$  transitions of the C=C aromatic rings and  $n \rightarrow \pi^*$  transitions of C=N azomethine groups. These transitions have also shifted to higher or lower frequencies in the UV-Vis spectrum of the polymeric complexes, affirming their formation. Moreover, it was determined that the polymeric complexes possess an octahedral geometry shapes, which were detected by the *d-d* transitions in the UV-Vis spectrum. The polymeric Cu(II) and Ni(II) complexes were tested for their antibacterial activities, and had shown a potent effect against *Salmonella typhi*.

**Keywords:** *Salmonella typhi*, Azomethine, Hydroxybenzaldehyde, Phenylenediamine, Chlorobenzaldehyde

INTRODUCTION

In recent years, the development of innovative materials with enhanced antibacterial properties has emerged as a critical area of research in the field of polymer science and medicinal chemistry. Among the various classes of compounds investigated for their antibacterial potential, Schiff base complexes have garnered significant attention due to their versatile coordination chemistry and ability to form stable complexes with a wide range of metal ions [1]. The incorporation of Schiff base ligands into polymeric complexes has yielded a class of materials known as polymer complexes of Schiff base, which exhibit remarkable antibacterial activity against pathogenic microorganisms [2–3].

Schiff bases, derived from the condensation of aldehydes or ketones with primary amines, possess unique structural features that contribute to their antibacterial efficacy. These compounds often contain functional groups with electron-

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donating or withdrawing properties, which play a crucial role in their interactions with bacterial cells. Additionally, the presence of metal ions in the polymer complex further enhances the antibacterial activity through mechanisms such as chelation, disruption of bacterial cell walls, and interference with enzymatic processes [4].

The design and synthesis of polymer complexes of Schiff bases with antibacterial activity involve the careful selection of both the Schiff base ligand and the metal center. Tailoring the chemical structure of the ligand allows for the optimization of antibacterial properties, including selectivity, potency, and spectrum of activity. Furthermore, the choice of metal ion can influence the mode of action and overall effectiveness of the polymer complex against specific bacterial strains [5].

This study aims to provide an in-depth exploration of the synthesis strategies, structural features, and antibacterial mechanisms of polymer complexes of Schiff base, which is 2-((E)-((4-((E)-(4-chlorobenzylidene)amino)phenyl)imino)methyl)phenol (prepared from p-phenylenediamine, salicylaldehyde and 4-chlorobenzaldehyde), is the structural and biological activity (antibacterial), and its polymeric Co(II), Ni(II), and Cu(II) complexes.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopy, Fourier transform infrared spectroscopy, ultraviolet-visible spectroscopy, CHN elemental analysis, and molar conductivity measurements, were employed for structural determinations.

## MATERIALS AND METHODS

### Experimental section

**Materials and instrumentation:** The following chemicals were purchased from Sigma-Aldrich, Fluka, and E. Merck and used as obtained: 4-chlorobenzaldehyde, 2-hydroxybenzaldehyde, and p-phenylenediamine, as well as the appropriate metal salts ( $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ ,  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ , and  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ). Absolute ethanol and DMSO were purchased from Sigma-Aldrich for the synthesis of the Schiff base ligand and its metal complexes without any further purification.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopy of the Schiff base ligand was performed on Bruker 850 MHz and 213 MHz spectrometers, respectively, using  $\text{DMSO-d}_6$ . An Agilent spectrometer was used to register transmission spectra in the region 4000-400  $\text{cm}^{-1}$ . Electronic UV-Visible spectra were recorded using the Shimadzu spectrophotometer (UV-1650PC, Japan). C, H and N analyses were performed using Eurovector CHN Elemental Analyzer (EA3000, Italy). Metal percentages were measured using Inductively Coupled Plasma (ICP 710-ES Varian). Conductivity/DTS Meter was put to measure the molar conductivity. The melting points of SB ligand and its complexes were determined in open capillary tubes using Stuart SMP30 Apparatus.

### Synthesis of the Schiff base

The following method was used to prepare the SB ligand under study, 2-((E)-((4-((E)-(4-chlorobenzylidene)amino)phenyl)imino)methyl)phenol. A solution of 2-hydroxybenzaldehyde (0.590 g; 5 mmol) and 4-chlorobenzaldehyde (0.703 g; 5 mmol) in 20 mL absolute ethanol were gradually added to a stirred mixture of p-phenylenediamine (0.540 g; 5 mmol) in hot absolute ethanol (15 mL) solution. The mixture's color changed from brown to yellow after being refluxed with heating for four hours and stirred without heating for the entire precipitation. The yellow product that had precipitated was then isolated by filtration, washed numerous times with hot absolute ethanol, and vacuum-dried. Yield: 91.7%. Decomposition point:  $160^\circ\text{C}$ .  $^1\text{H}$  NMR (850.000 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  (ppm): 13.0 (s, 1H, Ar-OH), 9.0 (s, 1H, HC=N), 8.9 (s, 1H, HC=N), 8.3-6.9 (m, 12H, Ar-H).  $^{13}\text{C}$  NMR (213.77 MHz,  $\text{DMSO-d}_6$ ) ( $\delta$ , ppm): 162.1–11.8. IR,  $\nu(\text{cm}^{-1})$ : 3100(O-H), 1680(C=N), 1537(C=N), 1245(C-O). UV-Vis ( $\text{DMSO}$ ),  $\lambda_{\text{max}}$  (nm): 255 ( $\pi \rightarrow \pi^*$ ), 339 ( $n \rightarrow \pi^*$ ). Elemental Analysis (%) For  $\text{C}_{20}\text{H}_{15}\text{ClN}_2\text{O}$  Calculated: C: 71.76, H: 4.38, N: 12.17; Cl: 10.59. Found: C: 71.75, H: 4.37, N: 12.17, Cl: 12.59 Molar conductance ( $\text{DMSO}$ ):  $4.7 \Omega^{-1} \cdot \text{mol}^{-1} \cdot \text{cm}^2$ .

### Synthesis of the Schiff base polymeric transition metal complexes

All complexes were synthesized in a similar manner. To a stirred solution of the synthesized ligand of (0.690 g, 2 mmol) in hot absolute ethanol (20 mL), the appropriate metal salts,  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  (0.340, 2 mmol),  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  (0.476 g, 2 mmol), and  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  (0.476 g, 2 mmol in hot absolute ethanol (15 mL), were added separately. The solutions were

refluxed for 3 h for complete precipitation. Then, the products were washed with absolute ethanol several times and dried by vacuum.

**[Ni<sub>2</sub>L<sub>2</sub>Cl<sub>2</sub> (H<sub>2</sub>O)<sub>4</sub>]<sub>n</sub>**

**Yield:** 87.3 %. Decomposition Point: 250 °C. IR,  $\nu(\text{cm}^{-1})$ : 1672 (C=N), 1543 (C=N), 1240 (C-O), 520 (M-O), 460 (M-N). UV-Vis (DMSO),  $\lambda_{\text{max}}$  (nm): 260 ( $\pi \rightarrow \pi^*$ ), 325 ( $n \rightarrow \pi^*$ ), 510, 588. Elemental analysis (%): calculated for [Ni<sub>2</sub>L<sub>2</sub>Cl<sub>2</sub> (H<sub>2</sub>O)<sub>4</sub>]: C: 53.82, H: 4.36, N: 5.78, Cl: 14.62, Ni: 12.10; Found: C: 53.20, H: 4.36, N: 5.76, Cl: 14.62, Ni: 12.09. Molar conductance (DMSO):  $8.9 \Omega^{-1} \cdot \text{mol}^{-1} \cdot \text{cm}^2$ .

**[Cu<sub>2</sub>L<sub>2</sub>Cl<sub>2</sub> (H<sub>2</sub>O)<sub>4</sub>]<sub>n</sub>**

**Yield:** 89.8 %. Decomposition Point: >300°C. IR,  $\nu(\text{cm}^{-1})$ : 1675 (C=N), 1535 (C=N), 1253 (C-O), 519 (M-O), 433 (M-N). UV-Vis (DMSO),  $\lambda_{\text{max}}$  (nm): 242 ( $\pi \rightarrow \pi^*$ ), 340 ( $n \rightarrow \pi^*$ ), 560. Elemental analysis (%): calculated for [Cu<sub>2</sub>L<sub>2</sub>Cl<sub>2</sub> (H<sub>2</sub>O)<sub>4</sub>], C: 53.72, H: 4.32, N: 5.72, Cl: 14.47, Cu: 12.97; Found: C: 53.72, H: 4.32, N: 5.72, Cl: 14.47, Cu: 12.13. Molar conductance (DMSO):  $11.6 \Omega^{-1} \cdot \text{mol}^{-1} \cdot \text{cm}^2$ .

**[Co<sub>2</sub>L<sub>2</sub>Cl<sub>2</sub> (H<sub>2</sub>O)<sub>4</sub>]<sub>n</sub>**

**Yield:** 95.3%. Decomposition Point: 290°C. IR,  $\nu(\text{cm}^{-1})$ : 1682 (C=N), 1525 (C=N), 1255 (C-O), 527 (M-O), 440 (M-N). UV-Vis (DMSO),  $\lambda_{\text{max}}$  (nm): 238 ( $\pi \rightarrow \pi^*$ ), 329 ( $n \rightarrow \pi^*$ ), 368, 580. Elemental analysis (%): calculated for [Co<sub>2</sub>L<sub>2</sub>Cl<sub>2</sub> (H<sub>2</sub>O)<sub>4</sub>]: C: 53.22, H: 4.36, N: 5.77, Cl: 14.61, Co: 12.14; Found: C: 53.23, H: 4.36, N: 5.77, Cl: 14.61, Co: 12.13. Molar conductance (DMSO):  $13.4 \Omega^{-1} \cdot \text{mol}^{-1} \cdot \text{cm}^2$ .

**Antibacterial activity**

Employing the hole plate diffusion technique, the Schiff base ligand, along with its Co(II), Ni(II), and Cu(II) complexes, were diluted using DMSO to achieve concentrations of 100 Parts Per Thousand (PPT). Subsequently, their antibacterial properties were assessed against *Salmonella typhimurium* [6–7]. Various quantities of each compound (30, 20, and 10  $\mu\text{L}$ ) were introduced into individual wells in the Petri dish using a sterile micropipette tip. Gentamicin (10  $\mu\text{g/mL}$ ) was utilized as the positive control.

## RESULTS AND DISCUSSION

### Chemistry

After the successful formation of synthesizing the SB ligand, following seven's procedure [8], and its polymer complexes of Co(II), Ni(II) and Cu(II), it was determined that the polymeric complexes were insoluble in water while they were moderately soluble in organic solvents like MeOH, EtOH, DMF, and DMSO, which could be concluded that the complexes possess polymeric nature. Figures 1 and 2 present the final obtained structure of SB ligand and its polymeric complexes.

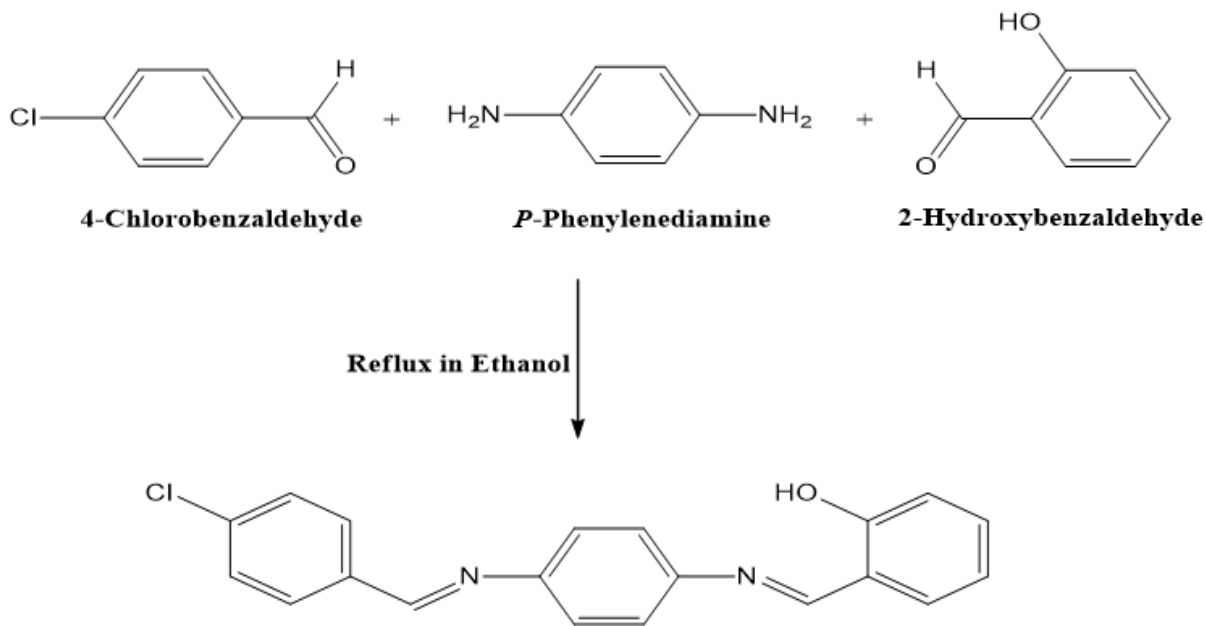
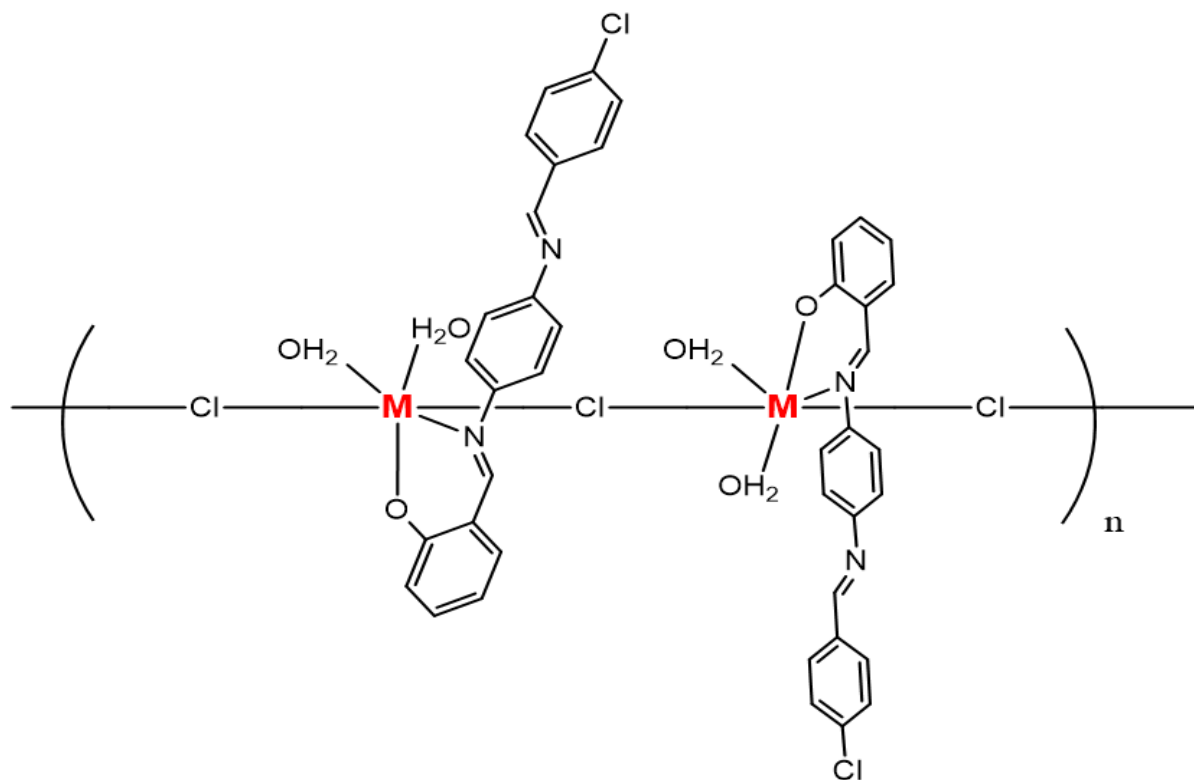


Figure 1: Synthesis and proposed structure of Schiff base ligand.



Where **M= Co, Ni, and Cu**

Figure 2: Proposed structure of Co (II), Ni(II) and Cu(II) polymeric complexes.

## <sup>1</sup>H and <sup>13</sup>C NMR

The <sup>1</sup>H NMR spectrum of the Schiff base assured the proposed structure of the synthesized SB ligand. The aromatic protons of the ligand are discerned within the projected chemical shift range of 6.9–8.3 ppm, manifested as complex multiplets [9]. Notably, two peaks at 8.9 and 9.0 ppm are assignable to the HC=N protons [10], while a singlet at 13.0 ppm in the downfield region is indicative of the phenolic proton [9–10]. On the other hand, In the <sup>13</sup>C NMR, distinctive peaks emerged at 163.7 ppm, 160.8 ppm, and 159.1 ppm, assigned to the phenolic, azomethine, and azomethine carbons, respectively. Furthermore, signals within the chemical shift range of 117.09–149.69 ppm are referred to the aromatic carbons (Figures 3 and 4) [10].

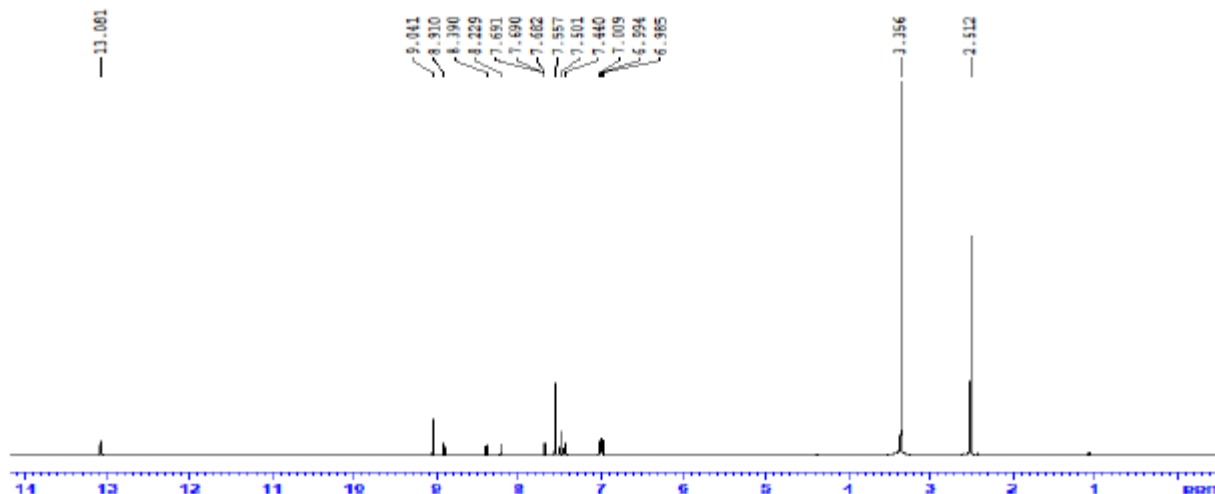


Figure 3: <sup>1</sup>H NMR of Schiff base ligand.

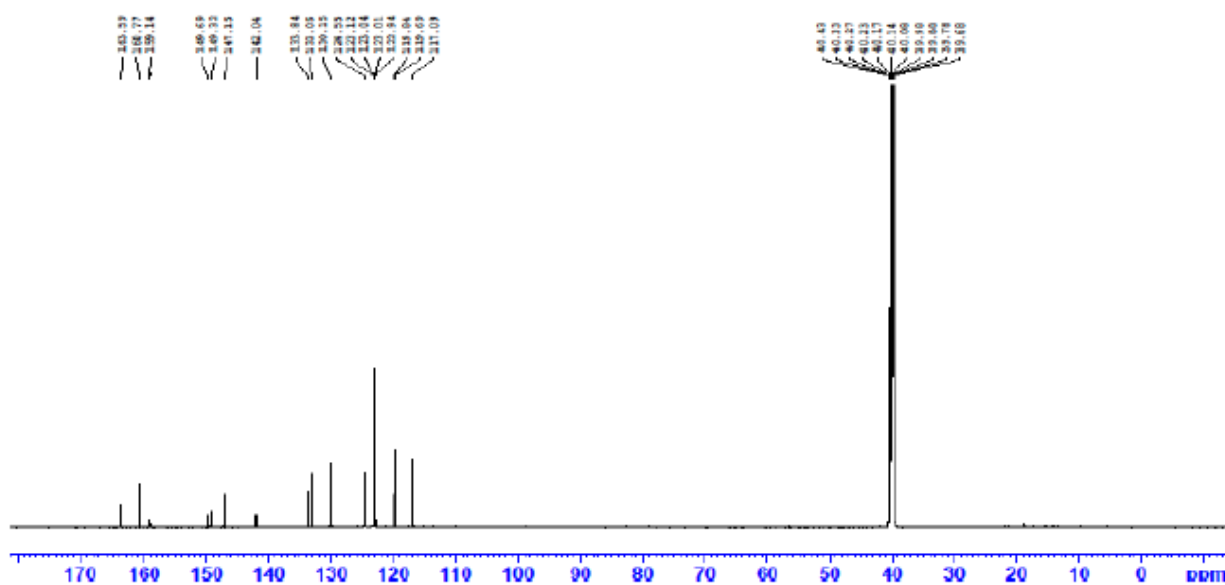


Figure 4: <sup>13</sup>C NMR of Schiff base ligand.

## FT-IR spectroscopy

The IR spectral analyses of the Schiff base ligand and the resulting polymeric metal complexes are reported in Table 1. The high-intensity absorption bands located at 1680 and 1537  $\text{cm}^{-1}$  in the Schiff base ligand spectra are unequivocally ascribed to the stretching modes of the azomethine moieties [9–10]. Meanwhile, the discernible broad absorption at 3100  $\text{cm}^{-1}$ , attributed to the phenolic group in the unbound SB ligand, is evident [10]. Noteworthy shifts in the band positions characteristic of the C=N stretching modes, consistently observed across all complex spectra, manifested the complexation of azomethine groups to the transition metal ions [9].

Furthermore, the emergence of new, broad absorptions in the complexes, in the range of 3600–3300  $\text{cm}^{-1}$ , are ascribed to the symmetric and asymmetric stretching modes of coordinated water molecules [11]. Additionally, the distinctive band associated with the C-O stretching mode, is observed at 1245  $\text{cm}^{-1}$  in the free Schiff base, undergoes a notable shifts in the IR spectra of the metal complexes, affirming the deprotonation and the consequent formation of phenolate-type coordinated Schiff base-transition metal complexes [10]. The respective M-O bands appeared in the spectral region of 510–523  $\text{cm}^{-1}$ , while the M-N bands are distinctly observed within the range of 440–460  $\text{cm}^{-1}$  [12].

**Table 1: Characteristic IR absorption bands ( $\text{cm}^{-1}$ ) of the prepared Schiff base ligand and its metal (II) and Metal (III) complexes.**

Compound	$\nu(\text{OH}) \text{ cm}^{-1}$	$\nu(\text{C}=\text{N}) \text{ cm}^{-1}$	$\nu(\text{C}-\text{O}) \text{ cm}^{-1}$	$\nu(\text{M}-\text{O}) \text{ cm}^{-1}$	$\nu(\text{M}-\text{N}) \text{ cm}^{-1}$
$\text{L}=\text{C}_{20}\text{H}_{15}\text{ClN}_2\text{O}$	3100	1680–1537	1245	-	-
$[\text{Ni}_2\text{L}_2\text{Cl}_2 (\text{H}_2\text{O})_4]\text{n}$	-	1672–1543	1240	520	460
$[\text{Cu}_2\text{L}_2\text{Cl}_2 (\text{H}_2\text{O})_4]\text{n}$	-	1675–1535	1253	519	433
$[\text{Co}_2\text{L}_2\text{Cl}_2 (\text{H}_2\text{O})_4]\text{n}$	-	1682–1525	1255	527	440

## CHN analyses

All the data obtained from the CHN elemental analyzer were in good agreement with the proposed structures and the calculated values of the C, H, and N content. The found values were in agreement with the expected values (Table 2). The ICP confirmed the percentage of the metal present in the complexes, and the chloride analysis is in good agreement with theoretical values of the proposed structure.

**Table 2: Analytical and physical data of Schiff base and its polymeric complexes.**

Compound	D.P. ( $^{\circ}\text{C}$ )	M	C	H	N	Cl
		Calculated (Found) (%)				
$\text{L}=\text{C}_{20}\text{H}_{15}\text{ClN}_2\text{O}$	160	-	71.76 (71.75)	4.38 (4.37)	12.17 (12.15)	10.59 (12.59)
$[\text{Ni}_2\text{L}_2\text{Cl}_2 (\text{H}_2\text{O})_4]\text{n}$	250	12.10 (12.09)	53.82 (53.20)	4.36 (4.36)	5.78 (5.76)	14.62 (14.62)
$[\text{Cu}_2\text{L}_2\text{Cl}_2 (\text{H}_2\text{O})_4]\text{n}$	>300	12.97 (12.97)	53.72 (53.72)	4.32 (4.32)	5.72 (5.72)	14.47 (14.47)
$[\text{Co}_2\text{L}_2\text{Cl}_2 (\text{H}_2\text{O})_4]\text{n}$	290	12.14 (12.13)	53.22 (53.23)	4.36 (4.36)	5.77 (5.77)	14.61 (14.61)

## Electronic (UV-Visible) spectra

The UV-Visible (UV-Vis) spectrum of each compound was recorded in DMSO ( $1 \times 10^{-4}$  M) (Table 3). The spectrum of the SB ligand exhibits bands at 255 and 339 nm, which are ascribed to the  $\pi \rightarrow \pi^*$  transitions of the C=C aromatic rings and  $n \rightarrow \pi^*$  transitions of C=N azomethine groups, respectively [13–14]. These bands were shifted in the spectra of metal complexes, which can be attributed to the metal-ligand coordination [13]. The electronic spectra of the Ni(II) complex show d-d transitions at 510 and 588 nm, the Co (II) complex shows at 560 nm, and the Cu(II) complexes shows

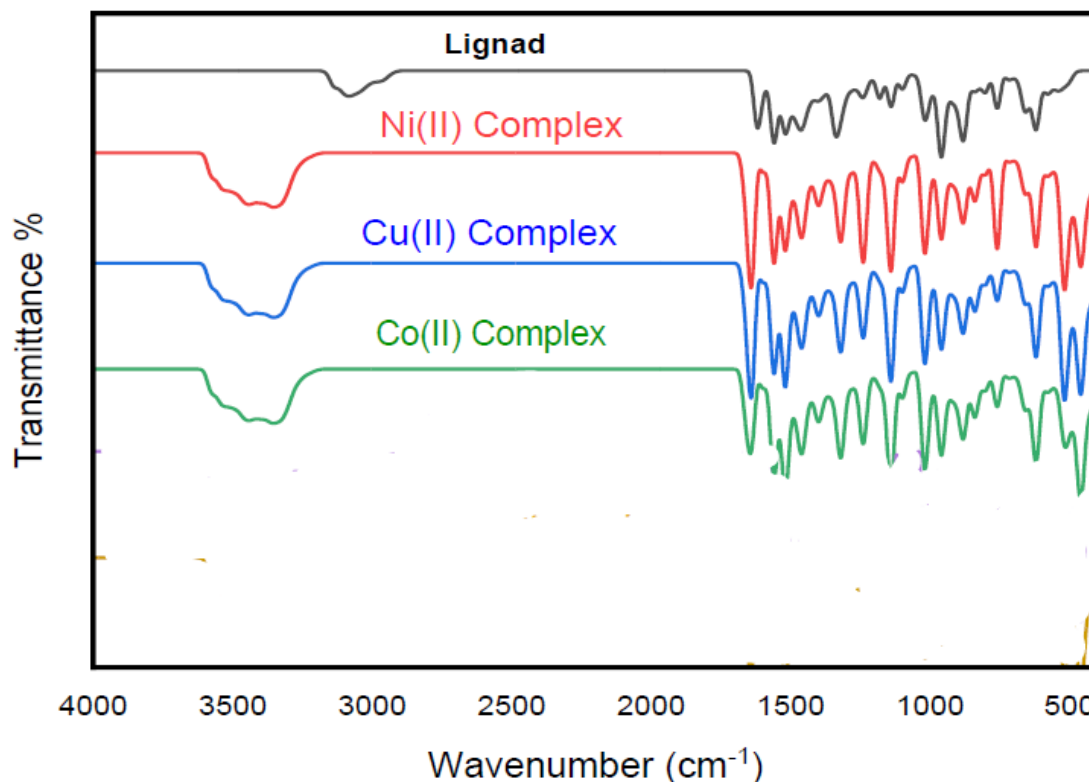
at 690 nm, which were all in the characteristics of octahedral arrangements in all three polymeric complexes [15].

**Table 3: Antibacterial activities of the Schiff base ligand and its Polymeric complexes.**

Compound	Volumes			
	30 $\mu$ L	20 $\mu$ L	10 $\mu$ L	Gentamycin (10 $\mu$ g/mL) positive control
$L=C_{20}H_{15}ClN_2O$	0	0	0	2
$[Ni_2L_2Cl_2(H_2O)_4]_n$	11	9	7	
$[Cu_2L_2Cl_2(H_2O)_4]_n$	13	11	8	
$[Co_2L_2Cl_2(H_2O)_4]_n$	0	0	0	

### Molar conductivity

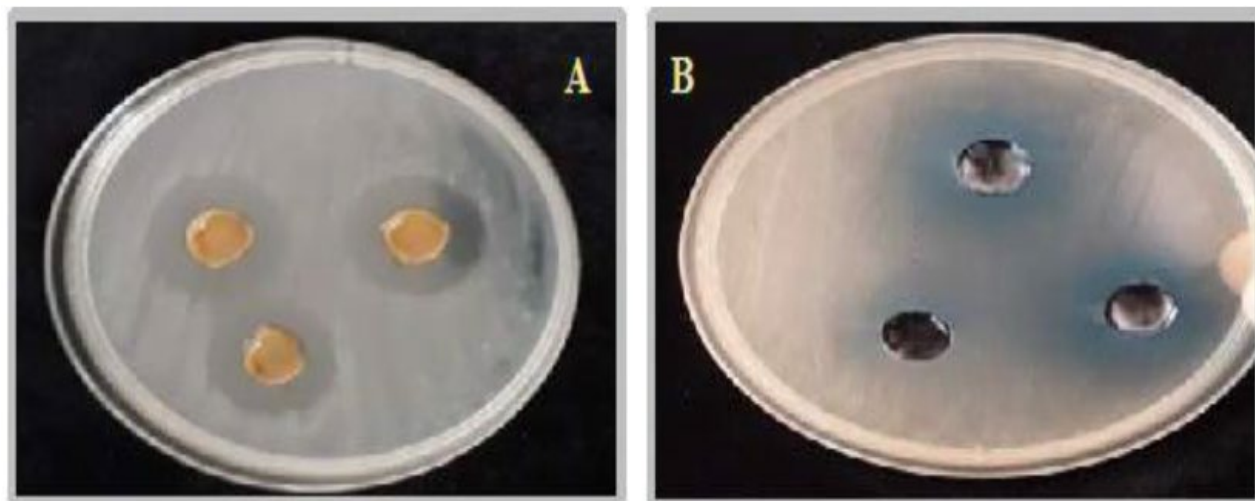
The molar conductivities ( $10^{-3}$  M) of the prepared polymeric complexes dissolved in DMSO were determined at room temperature. The molar conductivities of the synthesized polymeric complexes were found in the range 8.9, 11.6 and  $13.4 \Omega^{-1} \cdot \text{mol}^{-1} \cdot \text{cm}^2$  for the Ni(II), Cu(II) and Co(II) complexes, which signify the none electrolyte nature [13,16]. Also, it was figured out that all chlorine ions are involved in coordinating with the metals and not located outside the coordination sphere by adding  $AgNO_3$  solution to the complexes, which indicated that no white precipitate has formed [13].



**Figure 5: FT-IR spectra of the Schiff base ligand and its complexes.**

### Antibacterial activity

Table 3 presents the Schiff base ligands along with their respective complexes, detailing their *in vitro* antibacterial activity against *Salmonella typhi*. The Cu(II) complex exhibited pronounced activity at a sample volume of 30  $\mu$ L, while demonstrating intermediate activity at sample volumes of 20  $\mu$ L and 10  $\mu$ L. Conversely, the Ni(II) complex displayed intermediate activity across all sample volumes (30  $\mu$ L, 20  $\mu$ L and 10  $\mu$ L). It is noteworthy that both the Ni(II) and Cu(II) complexes exhibited superior antibacterial properties compared to the free Schiff base ligand.



**Figure 6: Antibacterial activities of (A) Ni(II) and (B) Cu(II) complexes with different volumes of 30  $\mu$ L, 20  $\mu$ L and 10  $\mu$ L against the growth of *S. typhi*.**

### CONCLUSION

In this study, a successful synthesis of Schiff base and its polymeric Co(II), Ni(II), and Cu(II) complexes has been performed. Instrumental characterization were done by  $^1\text{H}$  and  $^{13}\text{C}$  NMR, FT-IR, UV-Vis, CHN, ICP, and Conductivity. It could be concluded that the instruments confirmed the polymeric complexes. Finally, the antibacterial activity showed and excellent result for the Ni(II) and Cu(II) complexes.

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