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SCHIFF BASE COMPLEXES OF CO(II) AND NI(II) DERIVED FROM 2-HYDROXY-5-NITROBENZALDEHYDE AND 8-AMINOQUINOLINE: SYNTHESIS, CHARACTERIZATION AND ANTIBACTERIAL STUDIES

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ABSTRACT

A Schiff base ligand, *(E)-4-nitro-2-((quinolin-8-ylimino)methyl)phenol* ($C_{16}H_{11}N_3O_3$), was synthesized through condensation of 2-hydroxy-5-nitrobenzaldehyde with 8-aminoquinoline in ethanolic medium. The ligand was subsequently coordinated with cobalt(II) and nickel(II) ions to form the corresponding metal complexes. The synthesized ligand and its complexes were characterized using elemental analysis, FT-IR spectroscopy, molar conductivity measurements, and magnetic susceptibility studies. Spectral analysis revealed coordination of the ligand to the metal ions via the azomethine nitrogen and phenolic oxygen atoms, resulting in stable complexes with octahedral geometry around the metal centers. Molar conductivity measurements in DMSO indicated the non-electrolytic nature of the complexes, while magnetic susceptibility values confirmed their paramagnetic behavior. The antibacterial activities of the ligand and its metal complexes were evaluated in vitro against *Staphylococcus aureus* (ATCC 29213) and *Escherichia coli* (ATCC 25922) using the agar diffusion method. The results demonstrated that the metal complexes exhibited significantly enhanced antibacterial activity compared with the free ligand. Among the synthesized compounds, the Co(II) complex showed the highest activity, with minimum inhibitory concentration (MIC) values of 8 µg mL^{-1} against *E. coli* and 10 µg mL^{-1} against *S. aureus*. In contrast, the Ni(II) complex showed moderate activity. The improved biological activity of the complexes is attributed to the chelation effect, which increases lipophilicity and facilitates penetration through the bacterial cell membrane. Although the activity of the synthesized complexes was lower than that of the standard antibiotic tetracycline, the results suggest that the compounds may serve as promising scaffolds for the development of new antimicrobial agents.

Keywords: Schiff base ligand, cobalt(II) complexes, nickel(II) complexes, coordination chemistry, antibacterial activity.

INTRODUCTION

In recent years, Schiff base ligands and their corresponding metal complexes have attracted considerable attention in the field of inorganic medicinal chemistry. This interest arises from their distinctive structural features. It is also due to their flexible coordination modes and diverse biological activities. These compounds can form stable complexes with a wide range of metal ions. In addition, their chemical structures can be easily modified. This versatility makes them valuable in the design and development of novel therapeutic agents (Osigbemhe et al., 2021). The common method of synthesis of a Schiff base ligand involves the condensation reaction between a primary amine ($R–NH_2$) and a carbonyl-containing compound (R'–CHO or R'–CO–R"), whereby nucleophilic attack of the amine nitrogen on the electrophilic carbonyl carbon is followed by elimination of water (Abbasi et al., 2018). The azomethine or imine linkage is a crucial feature of Schiff bases, and their versatility makes Schiff base ligands highly adaptable for coordination to metal ions, leading to the formation of metal-Schiff base complexes with varied stoichiometries, geometries, and properties (Mahmoud et al., 2016). The exceptional structural distinctiveness of Schiff base complexes becomes apparent from the flexibility of the ligand, which can coordinate to metal ions in diverse configurations, including monodentate, bidentate, or polydentate modes (Alkış et al., 2021). This pliability allows Schiff base ligands to form stable complexes with various metal ions, including transition metals such as cobalt(II) and nickel(II) (Kumar Naik et al., 2014). The coordination environment around the metal ion, ranging from square-planar to octahedral geometries, significantly influences the chemical, physical, and biological properties of the resulting Schiff base metal complexes (Al-Hamdani et al., 2015). The biological activity of these complexes is primarily due to their capacity to interact with biomolecules, including proteins, nucleic acids, and lipids, often through metal-ligand coordination interactions(Bhaskar et al., 2020). Additionally, the metal center in these complexes is crucial for catalyzing biochemical reactions(Bao et al., 2020).

Cobalt(II) and nickel(II) Schiff base metal complexes are among the inorganic compounds that have been widely studied for their biological properties, especially their antimicrobial activities (Sakthivel et al., 2021). Both elements are biologically relevant transition metals that participate in various biological processes (Fekri et al., 2019). Cobalt, for instance, is a central element in vitamin B12, which is involved in critical biochemical pathways in living organisms(Alkış et al., 2021). Nickel, on the other hand, is a cofactor in certain enzymes, such as urease, which are essential for metabolic processes(Insiti et al., 2016). The ability of these metal ions to form stable coordination complexes with Schiff base ligands provides valuable opportunities to explore their antimicrobial efficacy, particularly in the face of increasing antibiotic resistance (Sovari & Zobi, 2020).

In this research article, we focus on the synthesis, characterization, and antibacterial activity of Schiff base complexes of (Co(II)) and

(Ni(II)), derived from the condensation of 2-hydroxy-5-nitrobenzaldehyde and 8-aminoquinoline. 2-hydroxy-5-nitrobenzaldehyde is an aromatic aldehyde containing a hydroxyl group that can provide additional coordination sites to the metal ion. At the same time, 8-aminoquinoline is a nitrogen-containing heterocyclic compound known for its ability to form stable metal-ligand complexes. The study of Schiff base complexes of Co(II) and Ni(II) derived from 2-hydroxy-5-nitrobenzaldehyde and 8-aminoquinoline is of particular interest due to the combination of their structural diversity, biological potential, and the relevance of the metal centers in biological systems(Arunadevi & Raman, 2020). These complexes could serve as the basis for the development of novel therapeutic agents to combat bacterial infections, contributing to the ongoing efforts to discover new antimicrobial compounds(Malik et al., 2018)

MATERIALS AND METHODS

2-hydroxy-5-nitrobenzaldehyde, 8-aminoquinoline, Cobalt(II) Chloride hexahydrate and Nickel(II) Chloride hexahydrate were procured from Sigma Aldrich, Mumbai. All the chemicals and reagents used were of analytical grade. All the solvents were further purified with standard methods (Uba et al., 2020). Melting points (m.p.) were recorded on Electrothermal MEL-TEP 1002D. Elemental analysis was performed on a Thermal- Scientific Flash 2000 CHNS/O analyzer. FT-IR spectra were obtained on a PerkinElmer IR Version 10.6 (4000–400 cm^{-1}).

Synthesis of the Schiff base ligand

The Schiff base ligand *(E)-4-nitro-2-((quinolin-8-ylimino)methyl)phenol* was synthesized by condensation of 2-hydroxy-5-nitrobenzaldehyde with 8-aminoquinoline.

Exactly 300 mg (2.77 mmol) of 8-aminoquinoline was dissolved in 10 mL of absolute ethanol and mixed with 464 mg (2.77 mmol) of 2-hydroxy-5-nitrobenzaldehyde. The reaction mixture was refluxed with continuous stirring for 6 hours at 70 °C. After completion of the reaction, the mixture was allowed to cool to room temperature, resulting in a yellow precipitate. The product was filtered, washed several times with cold ethanol, and dried under vacuum.

Yield: 86%. Elemental analysis (%): Calc. for $\mathbf{C_{16}H_{11}N_3O_3}$: C, 65.53; H, 3.78; N, 14.33; O, 16.73. Found: C, 65.22; H, 3.56; N, 13.95; O, 16.32

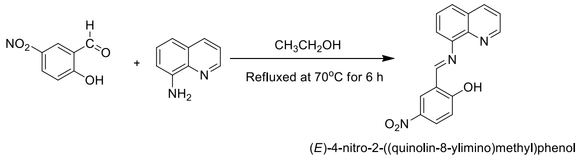

Figure 1: Synthesis of Schiff base ligand

Synthesis of Schiff Metal Complexes

Exactly 220mg (0.76 mmol) of (E)-4-nitro-2-((quinolin-8-ylimino)methyl)phenol was mixed with 180mg (0.76 mmol) of $CoCl_2.6H_2O$ in 10 ml of ethanol. The mixture was stirred under reflux for 4 hours at 70 °C. The solid product formed was separated by evaporation, washed with ethanol, and then dried. Ni(II) complex was synthesized by following a procedure similar to that of the Co(II) complex.

Characterization of the Schiff Base ligand and its metal Complexes

The synthesized Schiff base ligands and their corresponding metal complexes are characterized by various spectroscopic and analytical techniques to confirm their structures and properties.

RESULTS AND DISCUSSION

Table 1a: Physical properties and Analytical Data of Ligands/Complexes

Compounds	M. Wt	color	Percentage yield	Melting point (°C)
$C_{16}H_{11}N_3O_3$ (L)	(293).	Yellow	78%	195
CoL_2	-	Yellowish Brown	66%	259
NiL_2	-	Dark green	62%	289

Table 1b: Solubility in various solvents

compound	Distilled water	Ethanol	Chloroform	Diethyl ether	Methanol
$C_{16}H_{11}N_3O_3$ (L)	IS	S	S	SS	SS
CoL_2	IS	SS	S	SS	SS
NiL_2	IS	SS	S	SS	SS

Keys: S = Soluble, SS = Sparingly Soluble, IS = Insoluble

FT-IR spectral studies

IR spectra of the Schiff base complexes give important information about the coordination environment. The Schiff base ligand displayed typical absorption bands of 1610$cm^{-1}$for the azomethine (-C=N-) stretching vibration and 3220cm^{-1} for the free hydroxyl group in the Schiff base ligand. Upon complexation with Cobalt(II) and Nickel(II), shifts in the stretching frequency are observed, clearly indicating coordination of the ligand to these metal ions. Additionally, the O-H stretch of the hydroxy group is also affected by metal binding. New bands were observed in the 500–600 cm^{-1} range, corresponding to M–N and M–O vibrations, as shown in figure 1a-1c.

Conductivity measurement and Magnetic susceptibility studies

Absorption spectra of **L** and its Co(II) and Ni(II) complexes were obtained in DMF. UV–visible spectra were recorded for 1 × 10−4 M prepared solutions in the range 200–700 nm. A band at 270 nm was observed, which may be attributed to the π–π* transition of the aromatic ring. A second band observed at 320 nm could be attributed to the n– π* electronic transition of the azomethine group. Both bands were highly shifted (bathochromic shift) in the spectra of the complexes to 269–296 and 320–350 nm, respectively, due to chelation with metal ions. The spectra of Co(II) and Ni(II) complexes also showed absorption bands at 455 and 435 nm, which may be assigned to d–d orbital splitting. For Co(II) and Ni(II) complexes, a band at 332 nm appeared, which may be attributed to ligand-to-metal charge transfer.

Table 2: Conductivity Measurement of Complexes in DMSO Solution (1x10 ^{3}moldm^{-3}).

Compound	**Concentration (mol^{-1} dm^{-3})**	**Specific conductance Ohm^{-1}cm^{-1}**	**Molar conductance Ohm^{-1}cm^{2} mol^{-1}**
CoL_2	1 x 10^{-3}	$26.4x10^{-6}$	25.5
NiL_2	1 x 10^{-3}	$27.3x10^{-6}$	25.3

Electrolytic conductivity is a measure of the ability of ions in solution to carry an electric current, which is attributed to their ionic mobility. Ionic mobility depends on factors such as the charge and ionic radius of the metal ion, as well as its interactions with surrounding solvent molecules (Robinson & Soke, 2002). The effective charge and hydrodynamic radius of metal ions change when they coordinate with solvent molecules or other ligands to form complexes, typically causing a reduction in conductivity (David et al., 1999).

In the present study, the electrical conductivity of the divalent metal complexes (CoL_2 and NiL_2) was investigated in 10^{-3} M dimethyl sulfoxide (DMSO) solutions at room temperature. The measured molar conductance values for the Co(II) and Ni(II) complexes were 25.4 and 25.3 Ω^{-1} cm² mol^{-1}, respectively. The values are significantly lower than those commonly observed for strong electrolytes. These low conductance values indicate a non-electrolytic or weakly dissociated character of the complexes in solution. This observation aligns with findings reported by Parveez et al. (2014), who noted similarly reduced molar conductivities for transition-metal complexes in coordinating solvents due to limited ion dissociation.

Table 3: Magnetic Susceptibility of Metal (II) Complexes

Compound	**Magnetic susceptibility (cm^{3} g^{-1})**	**molar magnetic susceptibility (cm^{3} mol^{-1})**	**B.M(μ_{eff})**	**Magnetism**
CoL_2	17.684 x 10^{-9}	9.68 x 10^{-4}	4.80	Paramagnetic
NiL_2	1.758 x 10^{-9}	9.7 x 10^{-6}	4.72	Paramagnetic

The magnetic susceptibility measurement was carried out at room temperature. The result showed effective moments of 4.80 Bohr magnetons (BM) for the Co(II) complex and 4.72 BM for the Ni(II) complex. These values are consistent with high-spin electronic configurations and confirm the paramagnetic nature of both complexes. The observed magnetic moments align well with theoretical predictions for high-spin (Co2+) and (Ni2+) ions in octahedral coordination environments, where significant orbital contributions and spin-only behavior are expected (Huheey et al., 1993; Miessler et al., 2020).

Additionally, these results fall within the typical range reported for octahedral high-spin transition metal(II) complexes: 4.7-5.2 BM for Co(II) and 4.8- 5.4 BM for Ni(II) (Figgis & Hitchman, 2000). The alignment with existing literature substantiates an octahedral geometry around the metal centers. This interpretation is corroborated by Khan et al. (2014). Who observed comparable magnetic moments for analogous DMSO-coordinated(II) complexes and related them to octahedral coordination spheres with weak field ligand behavior.

Antibacterial Activity

Antibacterial activity was investigated using the agar well diffusion method. The free Schiff base ligand (**$C_{16}H_{11}N_3O_3$**) and the metal complexes (**Co(II)** and **Ni(II)**) were tested in vitro for their antibacterial activity against Gram-positive bacteria, *Staphylococcus aureus (ATCC 29213), and Gram-negative bacteria, Escherichia coli* (ATCC 25922). Tetracycline was used as a standard drug for both Gram-positive and Gram-negative bacteria. DMSO was used as a solvent control. Then the synthesized compounds were tested against bacterial strains. Sterilized media were carefully poured into the sterilized Petri dishes (20 ml per dish) and then left to solidify. 6 mm-diameter wells were made in the solidified media using a sterile borer. The microbial suspensions were distributed over the surface of the solidified medium using a sterile swab, and the test compounds were added to each well using a micropipette. The plates were incubated at 37°C for 24 h for antibacterial activity (Abd El-Halim et al., 2018).

Table 4: Antibacterial activity of ligand (**L**) and its metal complexes

Serial No.	Compound Name	Minimum Inhibitory Concentration (MIC) in µg mL^{-1}	
		Escherichia coli	*Staphylococcus aureus*
1	**Ligand (L)**	64	64
2	**CoL_2**	8	10
3	**NiL_2**	12	20
4	**Tetracycline (Control)**	0.5	0.25

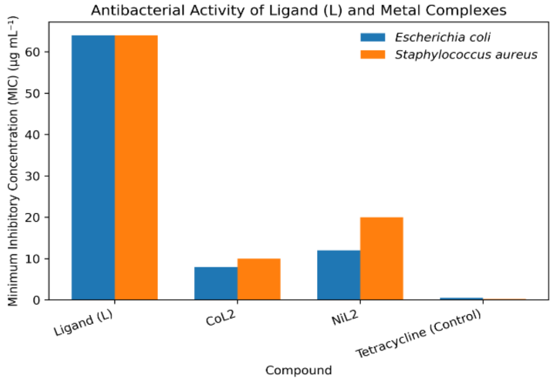

Figure 2: Antibacterial activity of the synthesized ligand (L) and its Co(II) and Ni(II) complexes against *Escherichia coli* and *Staphylococcus aureus*, expressed as minimum inhibitory concentration (MIC, µg mL^{-1}).

It has been established that several factors affect the biological activity of metal complexes, which include the nature of the donor atoms/metal ions/counter ions that neutralize the complex, the chelate effect of the ligands, the total charge on the complex ion, and the geometric structure of the complex (Catalano et al., 2021).

Additionally, chelation has been found to reduce the polarity of the metal ion due to partial sharing of its positive charge with the donor groups and possibly p-electron delocalization within the entire chelate ring system formed during coordination (El-Sonbati et al., 2019). All the factors mentioned equally increase the lipophilicity of the central metal atom, thereby increasing the hydrophobic character and liposolubility of the complex, thereby enhancing its permeation through the lipid layer of a bacterial membrane. This increases uptake rates, thereby improving the antibacterial activity of the evaluated compounds.

The antibacterial activity results (Table 3) revealed that the free ligand (L) exhibited relatively weak activity, with an MIC of 64 µg ml^{-1} against both test organisms. This limited efficacy suggests that the ligand alone is not sufficiently potent as an antibacterial agent. A considerable improvement in antibacterial activity was observed upon complexation with metal ions. The cobalt complex (CoL_2) showed the highest MIC values, with 8 µg/mL against E. coli and 10 µg/mL against S. aureus. This noticeable increase in activity can be explained by chelation, which reduces the polarity of the metal ion. As a result, the complex's lipophilicity increases. This enhanced lipophilicity allows the complex to penetrate the microbial cell membrane more effectively. The slightly higher activity against *E. coli* suggests that the complex can, to some extent, overcome the permeability barrier associated with Gram-negative bacteria (Khan, 2020).

The nickel complex (NiL_2) also demonstrated enhanced antibacterial activity relative to the ligand, with MIC values of 12 µg/mL against *E. coli* and 20 µg/mL against *S. aureus*. Although less potent than the cobalt complex, it still shows enhancement in comparison to the free ligand. This indicates that metal coordination plays a crucial role in modulating biological activity, though the extent of enhancement is dependent on the nature of the metal ion. The standard drug, tetracycline, showed the highest antibacterial activity, with MIC values of 0.5 µg/mL against *E. coli* and 0.25 µg/mL against *S. aureus*.

Comparatively, both metal complexes exhibited slightly better or comparable activity against *E. coli* than *S. aureus*, which is somewhat atypical, as Gram-negative bacteria are generally more resistant due to their outer membrane. This observation suggests that the complexes may possess structural features that enhance their permeability or their interactions with intracellular targets in Gram-negative organisms (Uddin et al., 2020).

Conclusion

In this study, *(E)-4-nitro-2-((quinolin-8-ylimino)methyl)phenol* Schiff base ligand and its metal complexes were successfully synthesized. The ligand and the resulting metal complexes were characterized using spectroscopic, analytical, and magnetic techniques. The spectroscopic data confirmed coordination of the ligand to the metal ions via the azomethine nitrogen and phenolic oxygen atoms, resulting in stable chelate complexes with octahedral geometry around the metal centers. Molar conductivity measurements indicated that the complexes behave as non-electrolytes in solution. At the same time, magnetic susceptibility results confirmed their paramagnetic nature, consistent with high-spin octahedral Co(II) and Ni(II) configurations.

The antibacterial investigation revealed that metal complexation significantly enhanced the ligand's biological activity. The Co(II) complex exhibited the highest antibacterial activity against both *Escherichia coli* and *Staphylococcus aureus*, followed by the Ni(II) complex, whereas the free ligand showed comparatively weaker activity. The improved antibacterial performance of the complexes can be attributed to the chelation effect, which enhances lipophilicity and facilitates better penetration through bacterial cell membranes.

Although the synthesized complexes exhibited lower activity than the standard antibiotic tetracycline, their moderate antibacterial activity suggests that Schiff base complexes of cobalt(II) and nickel(II) may be promising scaffolds for developing new antimicrobial agents. Further studies involving structural modification, mechanistic investigation, and in vivo evaluation are recommended to explore their pharmaceutical potential fully.

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