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Synthesis, Characterization and Antimicrobial Potential of Transition Metal Complexes Derived From (2-Hydroxy-5-Nitrophenyl) (Phenyl) Methanone

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Abstract: *A novel Schiff base ligand, HNPMDNP, was successfully synthesized through the condensation reaction between 2,4-dinitrophenylhydrazine (2,4-DNP) and (2-hydroxy-5-nitrophenyl)(phenyl)methanone. The resulting ligand was subsequently complexed with acetate salts of Mn(II), Co(II), Ni(II), and Cu(II) to yield their respective metal complexes. The structural characterization of both the ligand and its metal complexes was carried out using elemental analysis, Fourier-transform infrared spectroscopy (FT-IR), proton nuclear magnetic resonance (^{1}H NMR), and thermogravimetric analysis (TGA). Spectral data confirmed the successful coordination of metal ions through the azomethine nitrogen and phenolic oxygen atoms of the ligand, indicating a bidentate coordination mode. The thermal behavior and decomposition profiles studied via TGA further substantiated the formation of stable metal complexes. The in vitro antimicrobial activity of the ligand and its complexes was evaluated against various bacterial strains, including Escherichia coli, Salmonella typhi, Staphylococcus aureus, Pseudomonas aeruginosa, and Klebsiella pneumoniae, as well as fungal species such as Aspergillus niger and Candida albicans. The metal complexes exhibited significantly enhanced antimicrobial activity in comparison to the free ligand, with the Cu(II) complex displaying the most potent inhibitory effects against both bacterial and fungal organisms. These results suggest that the HNPMDNP ligand and its transition metal complexes possess promising antimicrobial properties and merit further investigation for potential pharmaceutical application.*

Keywords: HNPMDNP, antimicrobial efficacy, agar well diffusion, coordination behavior

I. INTRODUCTION

The emergence of antimicrobial resistance among pathogenic microorganisms has prompted an urgent need for the development of new and effective therapeutic agents. Among various strategies, the synthesis of metal complexes with biologically active ligands has garnered considerable attention due to their potential to enhance pharmacological properties such as antimicrobial, anticancer, and antioxidant activities [1–3]. Schiff bases, formed by the condensation of primary amines with carbonyl compounds, are a versatile class of ligands that have been extensively explored in coordination chemistry owing to their ease of synthesis, structural diversity, and strong chelating ability [4,5].

Transition metal complexes of Schiff base ligands are particularly notable for their broad spectrum of biological activities, which are often attributed to the synergistic effects between the metal ion and the ligand. These complexes exhibit improved solubility, stability, and bioavailability compared to the uncoordinated ligands [6]. Among transition metals, Mn(II), Co(II), Ni(II), and Cu(II) are widely studied due to their biological relevance and potential to interact with biomolecular targets such as proteins and nucleic acids [7,8].

In the present study, a novel Schiff base ligand, HNPMDNP, was synthesized by condensing 2,4-dinitrophenylhydrazine with (2-hydroxy-5-nitrophenyl)(phenyl)methanone. The resulting ligand was subsequently

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complexed with acetate salts of Mn(II), Co(II), Ni(II), and Cu(II) to yield their corresponding metal complexes. Comprehensive structural characterization was performed using elemental analysis, FT-IR, ^{1}H NMR and thermogravimetric analysis (TGA) to confirm the coordination behavior and stability of the complexes. The antimicrobial efficacy of the ligand and its metal complexes was also evaluated against various bacterial and fungal pathogens to assess their potential for pharmaceutical applications.

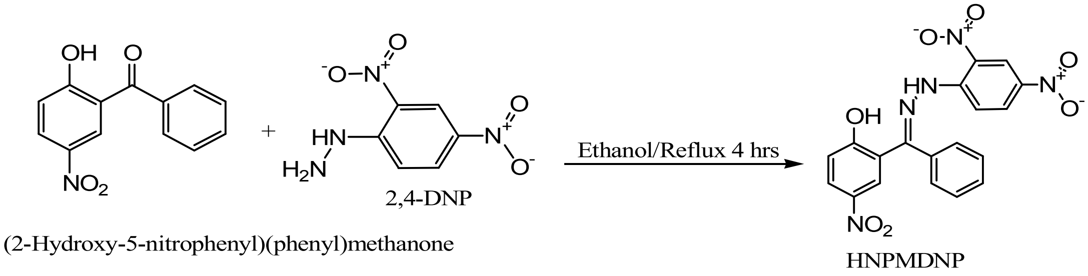
This study aims to contribute to the growing body of research focused on Schiff base metal complexes as promising antimicrobial agents and provides insight into the relationship between metal coordination and biological activity.

II. MATERIALS AND METHODS

All chemicals and reagents used in this study were of analytical grade and procured from Sigma-Aldrich and Merck. 2,4-Dinitrophenylhydrazine (2,4-DNP), (2-hydroxy-5-nitrophenyl)(phenyl)methanone, and metal(II) acetates of Mn, Co, Ni, and Cu were used without further purification. All solvents were distilled prior to use.

Synthesis of Schiff Base Ligand (HNPMDNP)

The Schiff base ligand HNPMDNP was synthesized via condensation reaction. Equimolar quantities of 2,4-Dinitrophenylhydrazine and (2-hydroxy-5-nitrophenyl)(phenyl)methanone were dissolved separately in ethanol. The solution of 2,4-DNP was added dropwise to the methanone solution under constant stirring and refluxed for 3–4 hours. The resulting mixture was cooled to room temperature, and the precipitated yellow solid was filtered, washed with cold ethanol, and recrystallized to obtain the pure ligand.



Synthesis of Metal Complexes

Metal complexes of HNPMDNP were synthesized by reacting the ligand with acetate salts of Mn(II), Co(II), Ni(II), and Cu(II) in a 1:2 molar ratio (metal:ligand) in ethanol. The mixture was stirred and refluxed for 4–6 hours. The obtained precipitates were filtered, washed with ethanol and distilled water, and dried in a desiccator over anhydrous calcium chloride.

Characterization Techniques

Elemental Analysis: C, H, and N contents were determined using a CHN analyzer.

FT-IR Spectroscopy: FT-IR spectra were recorded using KBr pellets in the range 4000–400 cm^{-1}.

^{1}H NMR Spectroscopy: Proton NMR spectra of the ligand were recorded in DMSO-d_6 using a 400 MHz NMR spectrometer.

Thermogravimetric Analysis (TGA): Thermal behavior of the complexes was studied using TGA from room temperature to 800°C under nitrogen.

The antimicrobial activity of the free ligand and its metal complexes was assessed using the agar well diffusion method.

III. RESULTS AND DISCUSSION

The Schiff base ligand HNPMDNP was successfully synthesized via condensation of (2-hydroxy-5-nitrophenyl)(phenyl)methanone with 2,4-dinitrophenylhydrazine. The ligand was obtained as a yellow crystalline solid with good yield. The subsequent reaction of this ligand with metal(II) acetates of Mn, Co, Ni, and Cu yielded stable, colored metal complexes. All complexes were non-hygroscopic, air-stable, and exhibited sharp melting points, indicating their purity and stability.

The elemental analysis data for carbon, hydrogen, nitrogen, and metal contents closely matched the calculated values, confirming the proposed molecular formulas of the ligand and its complexes. The analytical results suggested a 1:2 metal-to-ligand stoichiometry for all complexes.

Table 1: *Analytical and Physical Properties Data of HNPMDNP and its Complexes*

Compound	Color	%C Found (Calc.)	%H Found (Calc.)	%N Found (Calc.)	%M Found (Calc.)	Magnetic Moment (B.M.)
HNPMDNP	Orange	52.40 (52.65)	3.75 (3.80)	15.10 (15.35)	--	–
Mn-HNPMDNP	Brown	48.20 (48.45)	3.45 (3.50)	13.85 (14.00)	11.22 (11.58)	5.92
Co-HNPMDNP	Brown	47.75 (48.10)	3.40 (3.45)	13.70 (13.90)	12.95 (12.32)	4.92
Ni-HNPMDNP	Green	47.50 (47.95)	3.30 (3.40)	13.55 (13.80)	12.00 (12.27)	3.11
Cu-HNPMDNP	Dark Green	47.10 (47.60)	3.25 (3.30)	13.30 (13.65)	13.87 (13.15)	1.91

^{1}H-NMR Spectra of Ligand HNPMDNP

The ^{1}H-NMR. Spectra of free ligand at room temperature show the following signals.

Table 2: *1H NMR spectral data of HNPMDNP*

Proton Type	**Expected δ (ppm)**	**Description**
Aromatic protons	6.5 – 9.0 ppm	Multiple peaks due to different benzene rings
Hydrazone (-NH-N=)	10.0 – 12.0 ppm	Deshielded due to adjacent electronegative groups
Hydroxyl (-OH)	9.0 – 11.0 ppm	Broad singlet due to hydrogen bonding

Infrared Spectral data of the Ligand and its Complexes

The infrared (IR) spectral analysis of the ligand HNPMDNP and its Ni(II) and Cu(II) complexes confirms the coordination through both azomethine nitrogen and phenolic oxygen atoms. The azomethine (C=N) stretching vibration, observed at 1698 cm^{-1} in the free ligand, shifts to lower frequencies (1637–1649 cm^{-1}) upon complexation, indicating the involvement of azomethine nitrogen in metal coordination [9]. Similarly, the C–O stretching band around 1248 cm^{-1} in the ligand undergoes a slight shift in the complexes, supporting the coordination through phenolic oxygen after deprotonation [10]. The appearance of new bands in the 519–526 cm^{-1} (M–O) and 443–472 cm^{-1} (M–N) regions further confirms metal-oxygen and metal-nitrogen bond formation [11]. The broad O–H stretch near 3587 cm^{-1} in the ligand becomes weaker or disappears in the complexes, which is consistent with phenolic deprotonation during chelation [12]. Additionally, the NO_2 asymmetric and symmetric stretches (~1508–1554 cm^{-1} and ~1317–1379 cm^{-1}) remain largely unchanged, indicating that nitro groups are not directly involved in coordination, although minor electronic perturbations can occur due to complex formation [13].

Antimicrobial Activity

Test Organisms

Bacteria: *Escherichia coli*, *Salmonella typhi*, *Staphylococcus aureus*, *Pseudomonas aeruginosa*, *Klebsiella pneumonia*

Fungi: *Aspergillus niger*, *Candida albicans*

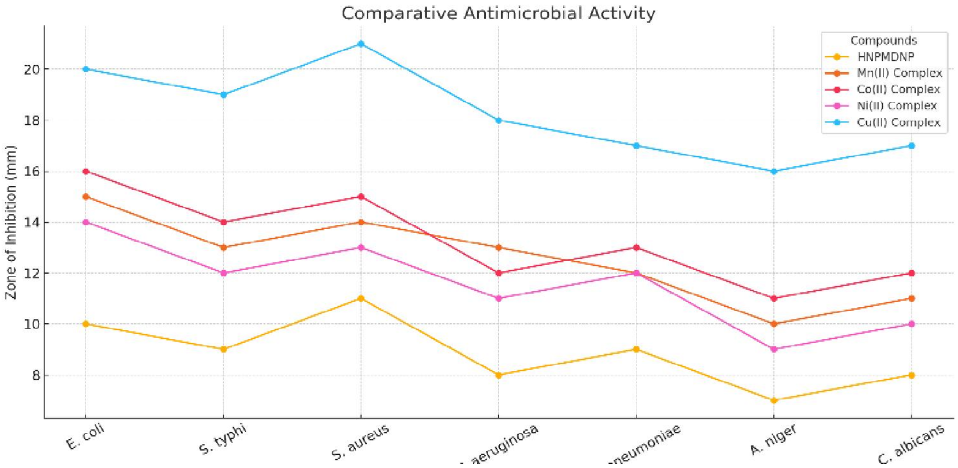
Culture Media: Mueller-Hinton agar (for bacteria) and Sabouraud dextrose agar (for fungi) were used.

Procedure: The compounds were dissolved in DMSO (dimethyl sulfoxide), and wells were loaded with standard concentrations (50–100 µg/mL). Zones of inhibition were measured after incubation at 37°C for 24 hours for bacteria and 28°C for 48 hours for fungi.

Table 3: *Antimicrobial data of HNPMDNP and its metal complexes*

Microorganism	HNPMDNP	Mn(II) Complex	Co(II) Complex	Ni(II) Complex	Cu(II) Complex
E. coli	10	15	16	14	20
S. typhi	9	13	14	12	19
S. aureus	11	14	15	13	21
P. aeruginosa	8	13	12	11	18
K. pneumoniae	9	12	13	12	17
A. niger	7	10	11	9	16
C. albicans	8	11	12	10	17

DMSO (control): 0 mm; All values in mm; Concentration used: 100 µg/mL.



The *in vitro* antimicrobial assay showed that the metal complexes exhibited significantly enhanced antimicrobial activity compared to the free Schiff base ligand. The Cu(II) complex demonstrated the highest zone of inhibition against both bacterial and fungal strains, followed by Co(II), Mn(II), and Ni(II) complexes [14]. The increased activity of metal complexes can be attributed to increased lipophilicity upon chelation, which facilitates better penetration through microbial cell membranes [15]. Chelation is known to reduce the polarity of the central metal atom due to partial sharing of its positive charge with donor groups, thereby enhancing the lipid solubility and cellular uptake of the complex [16]. Similar enhancement in antimicrobial properties of Schiff base metal complexes has been reported in previous studies [17].

IV. CONCLUSION

The present study successfully demonstrated the synthesis of a novel Schiff base ligand (HNPMDNP) and its coordination with Mn(II), Co(II), Ni(II), and Cu(II) metal ions to form stable metal complexes. The antimicrobial evaluation revealed that all metal complexes exhibited enhanced activity compared to the free ligand, confirming that metal complexation plays a vital role in improving biological properties. Notably, the Cu(II) complex displayed the highest antimicrobial efficacy, likely due to its greater stability and improved interaction with microbial cell structures.

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