Synthesis, characterization, and structural assessment of a Ni (II) complex of 2-2'((1*E*,1'*E*-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol and evaluation of its biological activity

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Abstract— **Preparation of a metal complex We used a number of physicochemical tests to learn more about the newly synthesised Metal complex of Schiff base . These tests included FTIR, ^{1}H NMR, mass spectroscopy, P-XRD, and ICP-OES. The metal complex of Schiff base was made by reacting 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol with nickel chloride hexahydrate to make a unique metal complex. examination of the antibacterial characteristics of the metal complex. They show promising activity. The compound's pharmacological potential is underscored by its successful synthesis, structural characterisation, and significant biological activity. More research is needed to find more ways to use it therapeutically and to make the most of its bioactivity.**

Key words— **Schiff base, metal complex, nickel chloride, antibacterial activity.**

I. INTRODUCTION

The first Schiff bases were found in 1864 by German chemist Hugo Schiff, who explained how primary amines and carbonyl compounds came together to make them. These compounds, which have the azomethine (-C=N-) functional group, have gotten a lot of attention in coordination chemistry because they can change their structure and work as good ligands. Schiff bases readily form complexes with metal ions, especially when derived from substituted aromatic compounds containing functional groups such as thiol (–SH) or hydroxyl (–OH), which significantly enhance their chelating properties [1-14].

There are many Schiff base ligands, but the ones made from ethylenediamine and 2-hydroxyaryl ketones have gotten a lot of attention. When there are both hydroxyl and imine groups, a number of metal ions can make stable bidentate or tetradentate complexes. When compared to Schiff base ligands that aren't coordinated, these metal complexes often have better thermal and chemical stability and more biological activity [9-14]. The metal chelates' better ability to pass through membranes and higher lipophilicity also make them work better in biological systems. Schiff base metal complexes have been shown to have many biological uses, such as being anti-inflammatory, analgesic, anticancer, antiviral, antifungal, pesticidal, bactericidal, insecticidal, herbicidal, and growth-regulating. Schiff bases are important for biology, but they are also important for materials science and industry. They are used to make semiconductors, cross-linked polymers, corrosion inhibitors, antiglare mirrors, deodorants, dental materials, and perfumes [15-20]. They are helpful in organic transformations like carbonylation, hydroformylation, oxidation, epoxidation, and hydrolysis because they can work as good catalysts in a wide range of conditions, such as high temperatures and moisture [21-25]. Schiff base complexes have garnered increased attention in the fields of medical and bioinorganic chemistry in recent decades due to their anticancer, antifungal, antiviral, antitubercular, and herbicidal properties. Their strong ability to bind to other substances, their ability to change shape, and their ability to do more than one thing make them good candidates for making new medicines and functional materials [26-28]. This study concentrates on the synthesis, characterisation, and evaluation of metal complexes derived from Schiff bases of 2-hydroxyaryl ketones and ethylenediamine, highlighting their antibacterial effectiveness against specific pathogenic strains.

II. EXPERIMENTAL

2.1. Materials

Make of 2-hydrxy-3- phenyl propiophenone is sigma Aldrich, ethylenediamine from SD fine, ethanol, AR Grade nickel chloride Hexahydrate, DM water.

2.2. Physical measurements

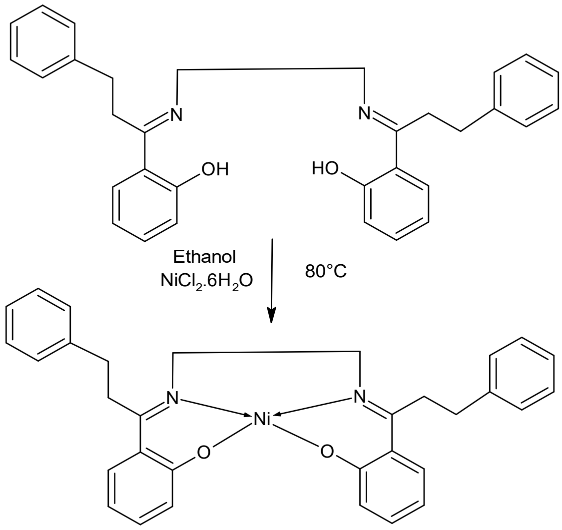
Using a SHIMADZU FT-IR 8300 spectrophotometer, we got Fourier-transform infrared (FT-IR) spectra in the 4000–200 cm^{-1} range to find the unique vibrational modes of functional groups. proton nuclear magnetic resonance to get the NMR spectra, the BRUKER FT-NMR spectrometer ran at 75.45 MHz and 400 MHz. We used DMSO-d^2 to take spectra and tetramethylsilane (TMS) as an internal standard. We used a Thermo Fisher Scientific CHNS Analyzer (FLASH SH 1112 Series) to check the compounds' composition and purity by analysing the elements C, H, N, and S. You can use an Agilent LC-MS machine to find molecular masses and fragment patterns. We used inductively coupled plasma optical emission spectroscopy (ICP-OES) to find the metal concentration by following standard ways to measure elements. We utilised a BRUKER AXS diffractometer (Germany) equipped with Cu $K\alpha_1$ radiation (λ = 1.5406 Å) for powder X-ray diffraction (P-XRD) to analyse the crystalline structure and phase composition of the synthesised samples.

2.3 Syntehisis of Schiff base 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol.

Preparation 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol carried out using condensation reaction of ethylenediamine and 2-hydroxy-3-phenylpropiophenone in ethanol at 80°C. Crystalline It in Ethanol wash with ethanol dry it [29].

2.4. Synthesis of nickel metal complex of 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol.

Add 50 millilitres of ethanol to a round-bottom flask that already has 2.0 grams of Schiff base in it. The Schiff base is 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol. For one hour, heat the mixture to 85 degrees Celsius. Next, add 1.05 grams of nickel chloride hexahydrate in five millilitres of water and heat it to 80 degrees Celsius until all of the Schiff base has turned into a nickel metal complex of 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol. Then It Is Characterised by Using Different Analytical Methods Like H1 NMR, Metal Content and FT IR



Scheme 1: nickel metal complex of 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol.

III. RESULT AND DISCUSSION

INFRARED SPECTROSCOPY STUDY.

The interaction between the Schiff base ligands and the metal complex of nickel was studied by comparing the IR spectra of the complexes and the free ligand.

The FT-IR spectra of L1 showed signal band at (1405), (3010), (1295, 1078), (1631), and (3250) cm−1. These are due to (ν Ar-C-C), (ν Ar C-H), (ν CH2N), (ν C=N), (ν C-O), and (ν O-H), in that order. The (C=N) and (CH2N) stretching bands in the L1M1 complex moved to the left and got weaker. L1 had a hydroxy group at the o-position, which gave it the unique phenolic ν (OH) at 3250 cm−1. It also had a band at 1455 cm−1 because of the ν (C–O) of the phenolic group. The complex L1M1 doesn't have the sharp band that comes from the ligand's phenolic (OH). Instead, the phenolic oxygen is linked to the metal ion.

After deprotonation, the phenolic ν(C–O) stretching band shifts to a lower wavenumber at 1415 cm^{-1} in the L1M1 complex, indicating stronger coordination. Additionally, the appearance of two new non-ligand bands in the regions 510–600 cm^{-1} and 400–460 cm^{-1}, attributed to ν(M–O) and ν(M–N) vibrations respectively, further confirms the involvement of phenolic oxygen and azomethine nitrogen in coordination with the metal centre in complex L1M1.

Complex/ligand	υ Ar C=C	υ Ar C-H	υ CH2-N	υ C=N	υ N=O	υ C-O	υ M-O	υ M-N	υ O-H
(L1)	1405 (s)	3010 (m)	1295, 1078(s)	1631 (s)	-	1455 (s)	-	-	3250 (m)
(L1M1)	1451.17 (s)	3050 (m)	1290,1080 (s)	1610 (s)	-	1415 (s)	510–600 (m)	400–460 (m)	

NMR SPECTROSCOPY

Nickel Metal complex 1H NMR were recorded as (400MHz, DMSO-d6). 7.62 (d, J=8 Ar–H, 2H), 7.32–7.11 (m, Ar–H, 12H), 6.74 (d, Ar–H, 2H), 6.71–6.53 (m, Ar–H, 2H),3.30 (s, $–N–CH_2$, 4H), 3.15–3.09 (t, $–CH_2–$, 4H), 2.82–2.78 (t, $Ar–CH_2–CH_2–$, 4H) . The disappearance of the phenolic – OH signal at 13–15 ppm indicates coordination of the phenolic oxygen to the metal ion, confirming complex formation.

POWDER X-RAY DIFFRACTION (PXRD)

Powder X-ray diffraction (PXRD) studies of complex 1 were carried out at room temperature to assess its crystallinity and phase purity. The diffraction data were recorded using Cu Kα radiation (λ = 1.5405 Å) operated at 45 kV and 40 mA, with a scanning speed of 5° (2θ) per minute. Additionally, PXRD analysis was performed on a Shimadzu XR-6100 diffractometer employing Cu Kα radiation (λ = 1.54 Å), a scan rate of 2° per minute, and a receiving slit of 0.15 mm. The resulting diffraction pattern exhibited sharp and well-resolved peaks, indicative of a highly crystalline material, thereby confirming the successful synthesis of the complex in a phase-pure form.

PHYSICAL CHARACTERISTICS AND ELEMENTAL ANALYSIS

COMPLEX/ LIGAND	MW	COLOUR	%YIELD	% C	% H	% O	%N	% Ni
				Found (Calculated)				
L1M1	533.28	BROWN COLOUR	87%	71.84(72.07)	5.60(5.67)	5.92(6.0)	5.20(5.25)	11.1(11.0)

ANTI-BACTERIAL

We tested the synthesized Ni (II) complex's ability to kill bacteria in vitro against four harmful strains: Bacillus subtilis, Staphylococcus aureus, Salmonella typhi, and Escherichia coli. Streptomycin (1 mg/mL) was used as the standard reference drug for the study, and DMSO was used as the negative control. The compound was evaluated at two different concentrations, 5 mg/mL and 10 mg/mL, to study its dose-dependent response.

The compound showed a clear concentration-dependent pattern, with increased zones of inhibition at higher concentration. At 5 mg/mL, the inhibition zones were 10 mm for S. aureus, 4 mm for S. typhi, 2 mm for E. coli, and 8 mm for B. subtilis. When the concentration was increased to 10 mg/mL, the antibacterial activity improved significantly, showing inhibition zones of 15 mm, 27 mm, 4 mm, and 17 mm against S. aureus, S. typhi, E. coli, and B. subtilis, respectively.

The standard drug Streptomycin exhibited strong antibacterial activity against all tested strains, with inhibition zones ranging from 25 to 30 mm, whereas the negative control (DMSO) showed no activity.

These results indicate that the synthesized Ni (II) complex exhibits moderate to strong antibacterial activity, particularly against S. aureus, S. typhi, and B. subtilis, while showing weak activity against E. coli. The overall findings confirm that the antibacterial activity increases with concentration, suggesting its potential as a promising antimicrobial agent.

Antibacterial Activities of samples against S. aureus, S. typhi, E. coli & B. subtilis.

Sr. No	Sample/Concentration	S. aureus (mm)	S. typhi (mm)	E. coli (mm)	B. subtilis (mm)
1	Control (DMSO)	-	-	-	-
2	Streptomycin (1 mg/ml)	25	30	30	28
3	Nim/G-03/25 (5 mg/ml)	10	4	2	8
4	Nim/G-03/25 (10 mg/ml)	15	27	4	17

The synthesized compounds exhibited relatively weak antifungal activity.

IV. CONCLUSION

The nickel metal complex of 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol was successfully synthesized. The synthesized metal complex was characterized using FT-IR, ^{1}H NMR, elemental analysis, and P-XRD techniques. The Ni (II) complex was evaluated against bacterial strains such as *Staphylococcus aureus*, *Salmonella typhi*, *Escherichia coli*, and *Bacillus subtilis* to investigate its antibacterial activity.

The biological activity data revealed that the Ni (II) complex exhibits good antibacterial activity, particularly against *Staphylococcus aureus*, *Salmonella typhi*, and *Bacillus subtilis*, while comparatively lower activity was observed against *Escherichia coli*. The results also demonstrated a clear concentration-dependent antibacterial effect, with enhanced inhibition observed at higher concentration (10 mg/mL).

Although the activity is lower than the standard drug Streptomycin, the overall findings suggest that nickel metal complex of 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol possesses promising antibacterial potential. This activity may be attributed to the chelation effect, which enhances the interaction of the metal complex with microbial cell membranes, improving its biological efficiency but this metal complex shows weak antifungal activity.

Therefore, the Ni (II) complex of 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol can be considered a potential candidate for further research in pharmaceutical and biological fields, particularly for the development of new antibacterial agents with improved efficacy and broader spectrum of activity.

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