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RESEARCH ARTICLE

SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL ACTIVITY OF DIVALENT TRANSITION METAL COMPLEXES OF 3-{((4,6-DIHYDROXY PYRIMIDIN-2-YL)IMINO)METHYL}NAPTHALEN-2-OL

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ABSTRACT

In this study, heterocyclic Schiff base ligands derived from 2-amino-4,6-dihydroxypyrimidine and 2-hydroxy-1-naphthaldehyde (L_1) were synthesized. These ligands were used in the synthesis of Ni(II) complexes. The synthesized compounds were characterized using FT-IR, ^{1}H-NMR and UV-Vis techniques for the ligands, and TLC for all reactions, molar conductivity and magnetic susceptibility measurements for the corresponding ones. The general formula of the complexes is $(Ni(L_1)_2(H_2O)_2)$. The complexes are paramagnetic. Molar conductivity measurements showed that all complexes in (DMSO) are not electrolytes. Octahedral geometry of all complexes. The ligands are bidentate (L_1) due to the phenolic (OH) and azomethine nitrogens. The ligands and their complexes were investigated for antifungal and antibacterial activity against *Aspergillus niger, Penicillium chrysogenum, Fusarium moneriforme, Aspergillus flavus,* as well as *Escherichia coli, Salmonella typhi, Staphylococcus aureus, and Bacillus subtilis*. The results showed that the complex exhibited excellent antifungal and antibacterial effects.

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INTRODUCTION

Schiff bases contain an azomethine or imine moiety (-C=N). It is a primary condensation of an amine with a carbonyl compound and was described by Hugo Schiff (Sakhare, 2022). Schiff bases have a wide range of applications in various fields such as biochemistry, organic chemistry, and inorganic chemistry. The medical uses and applications of Schiff bases and their metal complexes are gaining clinical and commercial importance. Schiff bases are gaining importance in the medical and pharmaceutical fields due to their wide range of biological activities such as: B. Anti-inflammatory (Chandramouli, 2012), analgesic (Sakhare, 2022), antibacterial and antispasmodic (Sliverstien, 2005). The stability of Schiff base complexes depends on the strength of the C=N bond, the basicity of the imino group, and steric factors. If a second functional group with a disposable hydrogen atom, preferably a hydroxyl group, is present in close proximity to the imine group, the ligand can form relatively stable four-, five-, or six-membered rings by chelation with the metal atom. The role of the metal ion in these complexes appears to involve both stabilization and entrapment of the Schiff base, which also ensures the planarity of the system (Sakhare, 2022). The great interest in pyrimidine-based compounds is mainly due to their applications in various fields such as the pharmaceutical, agrochemical, and crop protection industries (Sakhare).

Pyrimidines are known to be important components of nucleic acids and are used as synthetic precursors for biologically active molecules. There is a wide range of pharmacologically active pyrimidine compounds, and since the synthetic discovery of their substituted (amino, hydroxy, fluorine, etc.) derivatives, their use in medicine has become even more widespread. Pyrimidine derivatives have been reported to have a variety of pharmacological activities, including: analgesics, antiepileptics, antivirals, antihypertensives, minoxidil, antimycobacteria, and potent phosphodiesterase inhibitors (Sakhare, 2024; Harshalata, 2015; Sakhare, 2015; Rana, 2004). In addition, pyrimidinyl-containing drugs are well-known chemotherapeutic agents and are used in the treatment of cancer and tumors. For example, small molecule multikinase inhibitors (sunitinib and sorafenib) are used to treat advanced renal cell carcinoma (Atkins, 2006; Sakhare, 2023). In addition, 5-fluorouracil has been used as an effective antitumor drug, and the combination of 5-fluorouracil with bevacizumab has improved the treatment of metastatic colon cancer (Ramaling, 2007). In addition, the pyrimidine derivative pazopanib (5-(4-((2,3-dimethyl -2 H- indazoyl-6-yl) methylamino)-2-pyrimidinyl) amino-2-ethylbenzenesulfonamide), a potent and selective multi-target receptor tyrosine kinase inhibitor, has successfully completed clinical trials and pilot phase of development for the treatment of renal cell carcinoma (Harris, 2008) Tyrosine kinase (2HCK)

is reported to be actively involved in the transmission of growth factor signals by inhibiting catalytic tyrosine phosphorylation. Functional alterations of the protein occur, and mutations in this kinase can lead to cancer (Sakhare, 2022) Cryptogein (1LRI) is a small protein with sterol carrier activity, as it functions as a sterol shuttle that helps pathogens grow and complete their life cycle (Lascombe, 2022).The (2OBM) ATPase is a type III (T3SS) secretion system involved in the early stages of selective secretion of specialized (T3SS) virulence effector proteins from the bacterial cytoplasm into infected host cells, a process essential for subsequent virulence. Important. In addition, zidovudine and pyrrolopyrimidine nucleoside derivatives have been used as drugs against HIV and Hepatitis C (Kappe, 1993). The therapeutic activity of many pyrimidine-containing drugs/compounds may be due to their low toxicity and structural diversity (Espinet, 1992). Pyrimidine bioactive derivatives have been reported to form stable Schiff bases that can be used as molecular metal ion chelators (Sakhare, 2016). The efficacy of pyrimidine bioactive molecules has also been shown to be increased by coordination with metal ions (Osowole, 2015; Sönmez, 2014; Gulcan, 2014). Heteroleptic metal complexes of pyrimidinyl-Schiff bases and heteroatoms (N and O) exhibit high kinetic and thermodynamic stability and mixed chelating ability in biological fluid systems, which can prevent the induction of cell damage caused by oxidative stress (Sakhare, 2015; Sakhare, 2015; Tetteh, 2014). The development and isolation of such complexes with improved pharmacological applications and low or no toxic side effects has become a major challenge in drug discovery. In this study, Schiff base ligands derived from 2-amino-4,6-dihydroxypyrimidine and 2-hydroxy-1-naphthaldehyde (L_1) below Fig. 7 and transition metals were synthesized. Furthermore, the structures of the prepared ligands were verified using FT-IR, 1H-NMR, and UV-Vis techniques, and the prepared complexes were characterized using FT-IR, UV-Vis, molar conductivity, and magnetic susceptibility measurements.

EXPERIMENTAL SECTION

Materials: Chemicals and reagents used in this study: 2-Hydroxy-1-naphthaldehyde, 2-amino-4,6-dihydroxypyrimidine, $Ni(NO_3)_2 \cdot 2H_2O$ were purchased from Sigma-Aldrich Chemical Company.

Instrumentation: IR spectra were recorded on a FTIR (ATR)-BRUKER-TENSOR37 spectrometer using KBr pellets in the range of 4000-400 cm^{-1}. ^1H NMR (modified mercury 300 MHz) spectra of the ligands were measured in DMSO using TMS as internal standard. X-RD was recorded on a BRUKER D8 Advance. TGA-DTA was recorded on a Shimadzu. Carbon, hydrogen and nitrogen contents were measured on a SHIMADZU Elemental model Vario spectrometer. The molar conductivity of the complexes was measured with an Elico CM 180 conductivity meter using 10^{-4} M solutions in DMSO. Magnetic susceptibilitymeasurements of metal chelates were performed on a Guoy balance at room temperature using $Hg(Co(SCN)_4)$ as a calibration agent.

Procedures

Synthesis of schiff base ligand (L_1): The ligand was prepared by modifying the method described (Sakhare, 2019). The Schiff base ligand was prepared by refluxing a mixture of 0.01 mol (1.2015 g) 2-hydroxy-1-naphthaldehyde and 0.01 mol (1.2710 g) 2-amino-4,6-dihydroxypyrimidine in 50 mL of refluxed synthetic ultra-dry ethanol for about 4 hours. The Schiff base thus formed was cooled to room temperature, collected by filtration, then recrystallized in ethanol and dried in vacuum over anhydrous calcium chloride (yield: 72%).

Synthesis of metal complexes($M(L_1)_2$): Ligand (2 mol) and metal nitrate (1 mol) (25 ml) were added to a hot ethanol solution (25 ml) under constant stirring. The pH of the reaction mixture was adjusted to 7-8 by adding 10% ammonia alcohol solution and refluxed for about 3 hours. The precipitated solid metal complex was filtered off while hot, washed with hot ethanol, and dried over calcium chloride in a vacuum desiccator. (Yield: 86%) (Sakhare, 2015).

RESULTS AND DISCUSSION

Some physical properties of Schiff base ligands and their metal complexes are shown in (Table 1).

Table 1. Physical properties of Schiff base ligands (L_1) and their metal complexes

Compound Molecular formula	Mol. Wt.	M.P. Decomp temp. 0C	Colour	Molar Conduc. Mho. Cm^2mol^{-1}
L_1	282	221	Brown	---
Ni-L_1	625	>300	Dark Brown	10.21

Table 2. Elemental Analysis of Ni(II) Complex

Compound	% Found (Calculated)			
	C	H	N	M
L_1	64.04 (64.21)	4.17 (4.20)	17.04 (17.09)	----
Ni-L_1	64.43 (44.35)	4.27 (3.29)	17.17 (14.15)	9.90 (9.88)

^{1}H-NMR spectra of ligand: The ^1H NMR spectrum of the free ligand at room temperature shows the following signals: 5.8 δ (s, 2H, phenolic (OH) hydrogen of the pyrimidine ring), 6.68 δ (s, 1H, hydrogen attached to the pyrimidine ring), 7.95 δ (s, 1H, hydrogen attached to the azomethine carbon), 7.68-7.29 δ (D, 4H, aromatic Ha, Hb, protons of the phenyl ring).

IR Spectra: The IR spectra of the free ligands in Figures 2 and 3 show characteristic bands at 3428, 1654, 1486, 1209, and 1038 cm^{-1}, which can be assigned to the stretching vibrations υOH (intramolecular hydrogen bond), υ C=N (azomethine), υ C=C (aromatic), υ C-N (arylazomethine), and υ C-O (enolic) (Sakhare, 2015). The absence of weak broad bands in the region of 3200-3400 cm^{-1} in the spectra of the metal complexes suggests the deprotonation of the intermolecular hydrogen bond OH groups upon complex formation and subsequent coordination of the phenolic oxygen to the metal ion. This is further supported by the downward shift of υ C-O (phenolic) (Osowole1, 2018) compared to the free ligand. Upon complexation, the υ (C=N) (Osowole, 2013) band shifts to lower wave numbers relative to the free ligand, indicating that the nitrogen of the azomethine group is coordinated to the metal ion. The υ C-N band shifts to lower wave numbers relative to the free ligand. The IR spectra of the metal chelates show new bands in the range of 500-600 cm^{-1} and 400-500 cm^{-1}, which can be assigned to the υ M-O and M-N vibrations, respectively (Sakhare, 2015). The IR spectrum of Ni(II) shows

a strong band in the 3050-3600 cm^{-1} region, indicating the presence of coordinated water in these metal complexes. The presence of coordinated water is further confirmed by the appearance of a nonligand band in the 830-840 cm^{-1} region, which can be assigned to the rocking mode of water. The presence of coordinated water is also evidenced and supported by TG/DTA analysis of these complexes. Therefore, it is concluded that coordination occurs via the phenolic oxygen and azomethine nitrogen of the ligand molecules in Table 3 below.

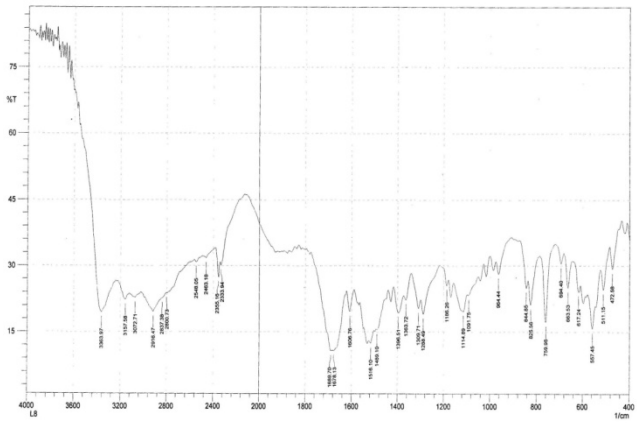

Fig. 2. Infrared Spectra of Ligand L

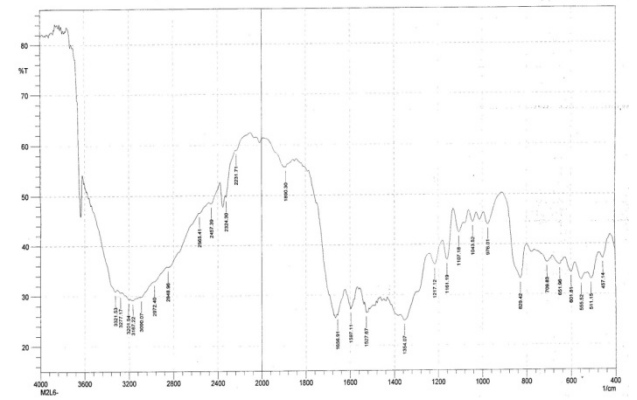

Fig. 3. Infrared Spectra of Ni(II) Complex of Ligand L

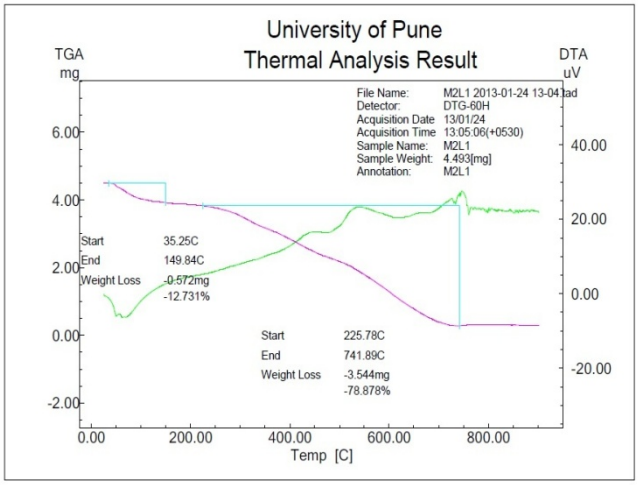

Fig. 4. TGA-DTA Curve of Ni (II) Complex of Ligand L_1

Molar conductance measurements: Conductivity measurements of the complexes were recorded for (10^{-3} M) and the sample solutions were in room temperature (DMso). The molar conductivity values of the complexes are shown in (Table 4). From the results, it was concluded that the Ni(II) complexes of ligand (L_1) have molar conductivity values in the range of (10.5-22.4 Ω^{-1} mol^{-1} cm^2). This suggests that the complexes are non-ionic and therefore can be considered as non-electrolytes (Usharani, 2013).

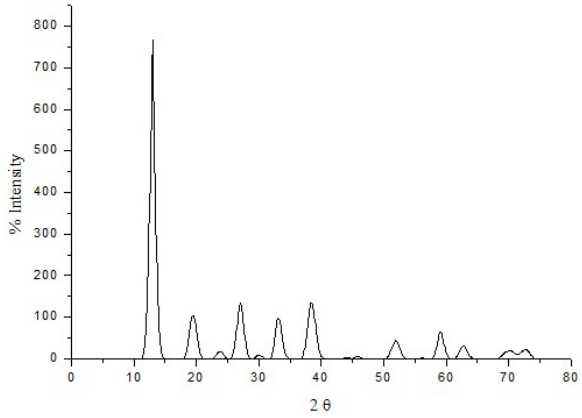

Fig. 5 X-ray Diffractogram of Ni (II) complex of L_1

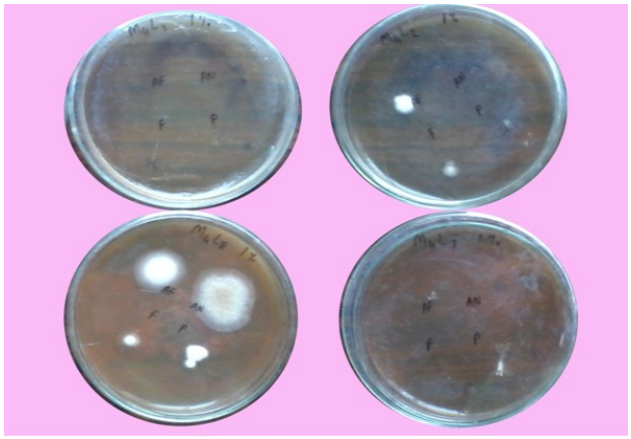

Fig. 6. Antifungal Activity of Ni(II) with L_1, 1%

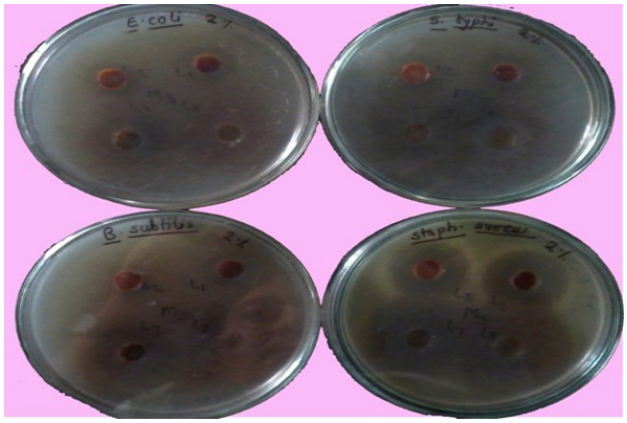

Fig: 7 Antibacterial Activity ofNi(II) with L_1, 2%

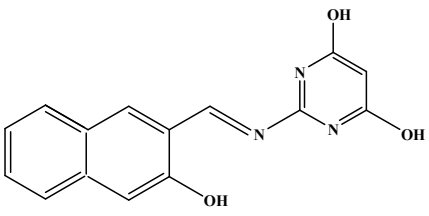

Fig. 8. Structure of Ligand L_1

Table 3. Salient features of IR spectral data of ligands & Metal complex

Bond vibrational modes	O-H Free Stretching(ν)	C = N AzomethineStretching(ν)	C = C Aromatic ring stretching(ν)	C - N Aryl azomethine stretch (ν)	C - O Enolic stretching (ν)	M-O	M-N
L	3342	1684	1656	1550	1268	--	--
Ni-L	3335.55	1683.23	1651.21	1560.22	1260.31	561.30	451.23

Table 4. The kinetic and thermodynamic parameters for decomposition of metal complexes

Complex	Step	Decomp. Temp. (^{0}C)	n	Ea (kJmole^{-1})	Z (S^{-1})	ΔS ($JK^{-1}mole^{-1}$)	ΔG (kJmole^{-1})	Correl -ation coefficient
Ni-L_1	I	433	0.92	10.32	1.26×10^4	-173.67	25.09	0.980

Table 4. Indexed X-ray Diffraction Data of Ni(II) Complex of Ligand L_1

Peak No.	2θ (observed)	2θ (calculated)	d (observed)	d (calculated)	Miller indices of Planes			Relative intensities (%)
					h	k	l	
1	13.266	13.236	6.66851	6.68394	1	1	0	100.00
2	18.497	18.508	4.79289	4.79	2	0	0	6.22
3	21.253	21.223	4.17715	4.18295	2	0	2	2.08
4	27.158	27.162	3.28086	3.28041	2	2	1	11.18
5	33.683	33.705	2.65875	2.65702	1	2	5	3.02
6	36.635	36.606	2.45096	2.45286	0	0	7	1.89
7	39.199	39.154	2.29637	2.2989	4	1	1	1.74
8	42	41.991	2.14947	2.1499	4	1	3	3.25
9	46.288	46.318	1.95983	1.95862	2	0	8	2.62
10	50.374	50.361	1.81002	1.81045	4	2	5	2.06
11	66.07	66.079	1.413	1.41283	5	1	8	1.57

Unit cell data and crystal lattice parameter
a (A^0) = 9.58 Volume(V) = 1534.67862 $(A^0)^3$
b (A^0) = 9.33 Density(obs.) = 1.087 gcm^{-3}
c (A^0) = 17.17 Density(cal.) = 1.076 gcm^{-3}
α = 90.00 Z = 1
β = 90.00 Crystal system = Orthorhombic
γ = 90.00 Standard deviation (%) = 0.052 Porosity = 1.02%

Table 5. Antifungal activity of ligands

Test Compound	Antifungal Growth							
	Aspergillusniger		*Penicilliumchrysogenum*		*Fusarium moneliforme*		*Aspergillus flavus*	
	1%	2%	1%	2%	1%	2%	1%	2%
L_1	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
Ni-L_1	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
+ve control	+ve	+ve	+ve	+ve	+ve	+ve	+ve	+ve
-ve control (Griseofulvin)	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve

Ligand& Metal : +ve – Growth (Antifungal Activity absent) -ve - Growth (Antifungal Activity present)
RG - Reduced Growth (More than 50% reduction in growth observed)

Table 6. Antibacterial activity of ligands and their metal complexes

Test Compound	Diameter of inhibition zone (mm)							
	E. coli		*Salmonella typhi*		*Staphylococcu saureus*		*Bacillus subtlis*	
	1%	2%	1%	2%	1%	2%	1%	2%
L_1	12mm	16mm	11mm	14mm	13mm	18mm	14mm	19mm
Ni-L_1	13mm	17mm	13mm	18mm	17mm	21mm	14mm	18mm
DMSO	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
Penicillin	14mm	14mm	18mm	18mm	31mm	31mm	19mm	19mm

Ligand & Metal: - ve - No Antibacterial Activity; Zone of inhibition - --mm

Magnetic susceptibility: The effective magnetic moment values of metal complexes are summarized in (Table 1). The values were measured at room temperature. The range of (μ_{eff}) for the complex (Ni(L_1)) is (2.81 to 3.97 B.M). This value is within the range of the octahedral geometry (Sakhare, 2025).

Thermogravimetric analysis: Thermal decomposition studies of the complexes were carried out to confirm the information obtained from the IR spectroscopy studies and to determine the presence of water molecules in these complexes and their decomposition pattern. Simultaneous TGA/DTA analysis of Ni(II) was studied from ambient temperature to 10,000 °C in nitrogen atmosphere using α-Al_2O_3 as a reference. Analysis of the thermograms of the complexes revealed that the Ni(II) complexes (Figure 4) exhibit a two-stage decomposition. The initial weight loss is 6.68 0% between temperatures. 55-2300 °C may be related to the loss of two adjusted waters (calculated 6.02%). The anhydrous compound is not stable at high temperatures. In the range of 230-650 °C, it decomposes rapidly with a mass loss of 79.73%, which corresponds to the decomposition of the complex in the second stage (calculated 80.71%). The decomposition is complete with the formation of a stable residue of the metal oxide NiO (13.13% (calculated) was observed). 13.29%). Kinetic and thermodynamic values, i.e., activation energy (Ea), frequency coefficient (Z), entropy

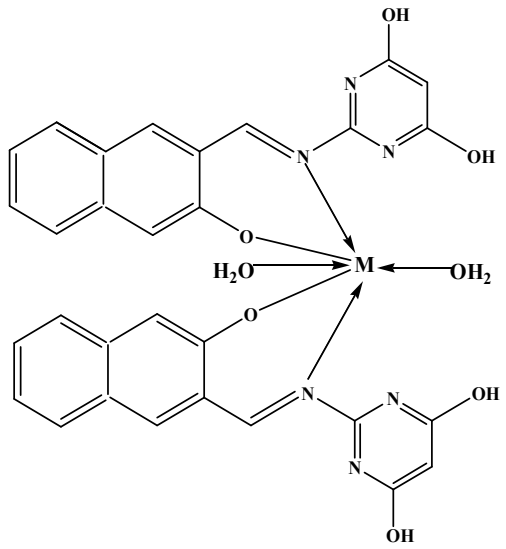

Fig. 9. The proposed Structure of the Metal complexes [When M= Ni (II)]

change (-ΔS), and free energy change (ΔG) for the nonisothermal decomposition of h.complexes, were determined using the Horowitz-Metzger method (33). The values are shown in Table 3. The calculated values of the given activation energies of the complexes are relatively low, indicating the autocatalytic effect of metal ions on the thermal decomposition of the complexes. The negative values of the activation entropy indicate that the activated complexes are more ordered, rather than slower in reaction. The stronger order may be due to the polarization of bonds in the activated state, which may occur through charge transfer transitions (Sakhare, 2015).

Electronic Spectra: The electronic spectrum of the Schiff base ligand (L_1) shows absorption bands at (42555 cm^{-1} and 27028 cm^{-1}), which are due to the transitions ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$), respectively. The Ni(II) complex shows a band at (42555 cm^{-1}) due to the transition ($\pi \rightarrow \pi^*$). The band at (23256 cm^{-1}) is due to charge transfer (C.T) and the band at (14815 cm^{-1}) is due to the transition $^3A_{2g} \rightarrow {}^3T_{2g}$. The complex has an octahedral geometry (Avaji, 2006).

X – Ray Diffraction Studies of Metal Complexes: Ni(II) complexes of ligands L_1 were selected for powder X-ray diffraction studies (Figure 5). The X-ray powder data of all major peaks were indexed individually using a trial and error method. The unit cell data, crystal lattice parameters, and data obtained after indexing of the powder data are shown in Table 4. The Ni(II) complex of ligand L_1 shows eleven reflections with a maximum at $2\theta = 9.77°$, corresponding to a d-value of 4.54 Å. The unit cell values of the lattice parameters are a = 9.58 Å, b = 9.33 Å, c = 17.17 Å, $\alpha = \beta = 90^o$ $\gamma = 90^o$, and the unit cell volume V = 1534.67862 $(Å)^3$.

BIOLOGICAL ACTIVITY

Antibacterial activity & Antifungal Activity: The antifungal and antibacterial activities of the ligands and metal complexes were tested in vitro against fungi such as *Aspergillus niger, Penicillium chrysogenum, Fusarium moneriforme, Aspergillus flavus,* and bacteria such as *Escherichia coli, Bacillus subtilis, Staphylococcus aureus, and Bacillus subtilis,* using paper disc and plate methods (Sakhare, 2022). The compounds were tested at concentrations of 1% and 2% in DMSO and compared with known antibiotics, namely *griseofulvin* and *penicillin* (Tables 5 and 6). Tables 5 and 6 show that the inhibition by the metal chelates is higher than that by the ligands, a result that is in good agreement with previous findings of comparable activity of the free ligands and their complexes (Sakhare, 2015; Sakhare, 2015). This increase in activity of the metal chelates is due to the increased lipophilicity of the metal ions in the complexes. The increase in activity with concentration is due to the effect of the metal ions on normal cellular processes. The action of the compounds may involve the formation of hydrogen bonds with active sites on cellular components, resulting in the disruption of normal cellular processes (Sakhare, 2025).

CONCLUSION

In light of the above discussion, we have proposed an octahedral geometry for the Ni(II) complex. Based on the physicochemical and spectral data discussed above, it can be hypothesized that the ligand behaves like a dibasic bidentate NO, coordinating through the phenolic oxygen and the imino nitrogen, as shown in Figure 9. The complex is biologically active and exhibits enhanced antibacterial activity compared to the free ligand. Thermal studies indicate the thermal stability of the complex. X-ray studies suggest a monoclinic crystal system for the Ni(II) complex.

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