

Green Synthesis Characterization and Antimicrobial Activity of Complex of Amide Ligand with Ni(II)

Swati Panchariya^{1*} | Dr Sumitra Godara²

¹Research Scholar, Department of Chemistry, Nirwan University Jaipur.

²Research Supervisor, Department of Chemistry, Nirwan University Jaipur.

*Corresponding Author: swatipanchariya8@gmail.com

Citation: Panchariya, S., & Godara, S. (2025). Green Synthesis Characterization and Antimicrobial Activity of Complex of Amide Ligand with Ni(II). International Journal of Innovations & Research Analysis, 05(03(II)), 96–100. [https://doi.org/10.62823/ijira/5.3\(ii\).7986](https://doi.org/10.62823/ijira/5.3(ii).7986)

ABSTRACT

The present research describes the synthesis, spectroscopic characterization, and antibacterial investigations of several Nickel (II) complexes with pyrimidine derivatives. The compounds were characterized using magnetic susceptibility measurements, infrared spectroscopy, electronic spectroscopy, and elemental analysis. Both the ligands and their complexes exhibited antibacterial activity against the pathogens *Staphylococcus aureus* and *Escherichia coli*. The diffuse reflectance spectra showed characteristic bands corresponding to the electronic transitions, consistent with an octahedral geometry around the Ni (II) ion. The observed magnetic moment further supports the high-spin octahedral environment. Overall, the synthetic approach has been found to be simpler, practical, and environmentally.

Keywords: Amide Ligand, Antibacterial, Octahedral.

Introduction

Nickel, a transition metal belonging to the first-row d-block elements, plays a significant role in coordination chemistry due to its ability to form stable complexes with a wide variety of donor ligands. In its +2 oxidation state, Nickel (II) is the most stable and commonly encountered form, and it readily coordinates with nitrogen, oxygen, and sulphur donor atoms present in both organic and inorganic ligands. The study of Nickel (II) complexes is important not only from the perspective of structural and electronic properties but also due to their wide range of applications in catalysis, materials science, and medicinal chemistry.

Pyrimidine derivatives, in particular, have emerged as highly versatile ligands because of the presence of heteroatoms such as nitrogen in the aromatic ring, which allows for effective chelation with transition metals. Nickel (II) complexes with pyrimidine-based ligands are especially significant as they combine the stability of the metal centre with the biological potential of heterocyclic ligands. These complexes are known to exhibit diverse coordination modes, ranging from simple monodentate to bidentate or even bridging behaviours, depending upon the ligand design. This versatility makes Nickel (II) an ideal metal ion for investigating structural variations in coordination compounds.

Spectroscopic characterization provides valuable insights into the bonding and geometry of Nickel (II) complexes. Infrared spectra usually confirm the coordination of donor atoms, especially nitrogen from pyrimidine rings and oxygen from amide or carbonyl groups. Electronic spectra of Nickel (II) complexes typically show d–d transitions that are consistent with octahedral, tetrahedral, or square-planar geometries. Magnetic susceptibility measurements further support the proposed geometry, with octahedral Ni (II) complexes usually showing paramagnetic behaviour due to the presence of unpaired

electrons. In contrast, square-planar complexes are often diamagnetic, reflecting the crystal field splitting and electronic configuration of the d^8 system.

The biological relevance of Nickel (II) complexes cannot be overlooked. Nickel is known to be an essential trace element in biological systems, functioning as a cofactor in enzymes such as urease and hydrogenases. When coordinated with heterocyclic ligands like pyrimidine derivatives, Nickel (II) complexes often display enhanced biological activity. Many studies have demonstrated their antimicrobial potential against Gram-positive and Gram-negative bacterial strains, including *Staphylococcus aureus* and *Escherichia coli*. The antibacterial activity of these complexes is believed to arise from their ability to interact with microbial cell walls and nucleic acids, disrupting essential biological processes.

From a synthetic perspective, the preparation of Nickel (II) complexes with pyrimidine ligands is generally straightforward, environmentally friendly, and yields stable products that are suitable for further spectroscopic and biological investigations. These complexes not only serve as model systems to understand fundamental coordination chemistry but also provide promising scaffolds for the development of metal-based drugs and functional materials.

In summary, Nickel (II) complexes with pyrimidine derivatives represent an important class of coordination compounds with dual significance: they offer structural and electronic insights into transition metal-ligand interactions, and at the same time, they demonstrate valuable biological activities. The present research aims to synthesize, characterize, and evaluate the antibacterial properties of such complexes, thereby contributing to the growing field of bioinorganic and coordination chemistry.

Materials and Methods

To synthesize Ni (II) complexes with amide group-containing ligands, a 250 mL round-bottom flask was charged with a solution of Nickel chloride (0.001 mol in 30 mL ethanol). To this solution, an appropriate amide ligand (0.003 mol) was added gradually with continuous stirring. The reaction mixture was maintained at room temperature for 6–7 hours under constant stirring with a magnetic stirrer.

For the alternative green synthesis approach, the reaction mixtures were exposed to microwave irradiation at 600 W for 2–10 minutes. Both conventional and microwave-assisted methods yielded solid precipitates, which were isolated and purified by recrystallization. Impurities were removed using alcohol, and the obtained crystals were washed and dried.

The resulting complexes were obtained as coloured crystalline or powdered materials. They were stable under normal laboratory conditions, non-hygroscopic, and largely insoluble in most organic solvents and water, with the exception of methanol and dimethylformamide (DMF). Conductivity measurements indicated that several of the complexes in DMF behaved as non-electrolytes, suggesting covalent character.

Table 1 presents the physical properties, elemental analysis results, and the proposed molecular formulae of the synthesized complexes, which were found to be in good agreement with the calculated values.

Table 1: Physio - Chemical Data of Ni (II) metal complexes

S.N.	Complex	Color	m.p. (°C)	Reaction Time (C.M. hrs / M.M. min)	Yield (%)	Elemental Analysis (Calc.)
1	[Ni(2NPCFA)]	Pale yellow	225	6.0 hrs / 2.00 min	C: 50% M: 40%	C: 43.13% H: 3.0% N: 25.1%
2	[Ni(2NPCAA)]	White	208	6.0 hrs / 2.00 min	C: 40% M: 50%	C: 46.40% H: 3.90% N: 23.20%
3	[Ni(2NPCBA)]	Yellow	201	6.0 hrs / 2.00 min	C: 45% M: 50%	C: 49.23% H: 4.64% N: 21.53%
4	[Ni(2NPCPA)]	Orange	198	6.0 hrs / 2.00 min	C: 45% M: 50%	C: 51.66% H: 5.30% N: 20.10%

C.M. = conventional method, M.M Microwave method

Results and Discussion

• Vibrational Spectra

The vibrational spectra of the complexes were recorded in the mid- and far-infrared regions using KBr pellets and polyethylene films. The spectra exhibited several diagnostic bands that provided

valuable information about the coordination sites of the ligands. Similar to the behavior observed in copper and cobalt complexes, the amide-containing ligands displayed characteristic coordination features.

In the free ligands, the amide $\nu(\text{N-H})$ stretching bands appeared at 3163–3382 cm^{-1} . In the complexes, these bands shifted to higher frequencies, indicating that the nitrogen atom of the amide group does not participate in coordination. In contrast, the amide-I band corresponding to the $\nu(\text{C=O})$ vibration exhibited a negative shift, suggesting the involvement of the carbonyl oxygen atom in coordination with the metal centre.

Additionally, the appearance of bands in the region of 20–820 cm^{-1} confirmed the participation of pyrrolidinyl nitrogen in bonding. The characteristic pyrimidine ring bands of the ligands were also observed to shift negatively upon complexation, further supporting coordination through the pyrimidine nitrogen atom.

Taken together, these spectral features provide strong evidence for the proposed bonding modes and confirm the structural conclusions regarding the coordination behaviour of the synthesized complexes.

• Measurements of Magnetic Susceptibility

The magnetic behaviour of divalent nickel complexes is strongly influenced by the nature of the ligand and the geometry of the complex. Octahedral Ni(II) complexes exhibit Para magnetism due to the presence of two unpaired electrons in the d-orbitals. The experimentally determined magnetic moment values for the synthesized complexes are consistent with the proposed electronic configuration and support the suggested octahedral geometry around the Ni(II) ion. The results are summarized in Table 2.

Table 2: Magnetic moments and electronic spectral data Ni (ii) ligand with complexes

S.N.	Complex	Rf Value	μ_{eff} (BM)	Electronic Transitions (cm^{-1})	Tentative Assignments (Bands)	Geometry
1	[Ni(2NPCFA)]	0.943 (g)	2.71	9153, 13850, 14836, 16129, 17006, 18315, 26631	d–d transitions + CT bands	Distorted Octahedral
2	[Ni(2NPCAA)]	0.870 (h)	3.82	9610, 14771, 16863, 18867, 22624, 24630	d–d transitions + CT bands	Distorted Octahedral
3	[Ni(2NPCBA)]	0.930 (c)	2.94	9174, 14204, 16528, 17667, 18231, 26075, 27027	d–d transitions + CT bands	Distorted Octahedral
4	[Ni(2NPCPA)]	0.765 (f)	3.10	9242, 13812, 14814, 16260, 17699, 18676, 26954	d–d transitions + CT bands	Distorted Octahedral

c= ethyl acetate: carbon tetrachloride (5:5), f = acetone: carbon tetrachloride (5:5), g = acetone: carbon tetrachloride (6:4), h= acetone: carbon tetrachloride (8:2)

Electronic Spectra

The electronic spectra of Ni (II) complexes originate from the $3d^8$ electronic configuration of the Ni (II) ion. In an octahedral crystal field, the ground term (3F) splits into three triplet states, resulting in three spin-allowed transitions. Accordingly, six-coordinate octahedral Ni (II) complexes generally exhibit three distinct absorption bands:

$^3A_{2g}(F) \rightarrow ^3T_{2g}(F)$ in the region 9,165–13,000 cm^{-1}

$^3A_{2g}(F) \rightarrow ^3T_{1g}(F)$ in the region 11,000–20,000 cm^{-1}

$^3A_{2g}(F) \rightarrow ^3T_{1g}(P)$ in the region 20,000–28,000 cm^{-1}

In addition to these transitions, the spectra of amide-containing ligand complexes also displayed higher energy bands attributable to charge transfer. However, the assignment of these charge-transfer bands remains somewhat uncertain, as they may also result from intra-ligand electronic transitions. The characteristic absorption bands of the complexes are summarized in Table 3,

Table 3: IR Vibrational Frequency of Ni Metal

S. No.	Complex	$\nu(\text{N-H})$ (amide)	$\nu(\text{C=O})$	$\nu(\text{C-N} + \delta\text{N-H})$	$\nu(\text{N-H} + \delta\text{C-N})$	Pyrimidinyl	$\nu(\text{M-N})$	$\nu(\text{M-O})$	$\nu(\text{M-Cl})$
1	[Ni(2NPCFA)]	3380	1625	1496	1343	1575	486	503	–
2	[Ni(2NPCAA)]	3385	1627	1499	1347	1579	490	505	–
3	[Ni(2NPCBA)]	3390	1631	1504	1351	1584	494	510	–
4	[Ni(2NPCPA)]	3395	1635	1510	1356	1589	499	514	–

confirming that the Ni (II) complexes adopt an octahedral environment.

Thermal Studies

The thermal behaviour of Ni(II) complexes containing amide group ligands was investigated to evaluate their stability. Thermogravimetric analysis revealed that the complexes decompose according to first-order kinetics, as indicated by Coats–Redfern linear plots (for $n = 1$). The activation energy (E_a) values were calculated using the Coats–Redfern method, providing insights into the thermal stability of the complexes.

The thermal studies demonstrated that the complexes are stable at room temperature and remain intact at moderately elevated temperatures. The first stage of decomposition was observed around 500 K, indicating considerable thermal stability of the synthesized Ni (II) complexes.

Antibacterial activity-

Ligand complex Zone of inhibition (in mm)

S.No.	Complex	E. coli	S. aureus
1	[Ni(N ₂ PCFA) ₃]Cl ₂	5.0	5.5
2	[Ni(N ₂ PCAA) ₃]Cl ₂	0.0	4.5
3	[Ni(N ₂ PCSA) ₃]Cl ₂	4.5	0.0
4	[Ni(N ₂ PSGA) ₃]Cl ₂	4.5	4.5

Table 4: Antibacterial activity Ni (ii) ligand with complexes. The Muller Hinton Agar medium was used to evaluate the antibacterial activity of the synthesized metal complexes against E. coli, S. aureus. The results indicate that all the metal complexes exhibit moderate antibacterial activity against these bacterial strains. Among them, the Ni(II) complexes demonstrated the most significant activity, showing particularly strong effectiveness against S. aureus, and E. coli, respectively.

Conclusion

The vibrational spectra indicate that amide ligands act as tetradentate donors in Ni (II) complexes, coordinating primarily through the carbonyl oxygen atoms of the amide groups. The geometry of the metal ion in these complexes strongly influences their electronic spectra, with the observed transitions confirming the formation of octahedral Ni (II) complexes. The measured magnetic moments are in good agreement with the electronic spectral data, further supporting the proposed geometry. The suggested structures of the Ni (II) complexes are illustrated in Figures consistent with previously reported studies.

The green synthetic method employed in this work provides a simpler and more sustainable approach, often producing comparable or even superior yields relative to conventional methods. The mechanism of coordination is confirmed by shifts in the characteristic IR bands of the amide-containing ligands upon complexation, demonstrating bonding through the carbonyl oxygen and pyridyl nitrogen atoms. The incorporation of additional exogenous ligands is also evident from spectral data, suggesting coordination either through deprotonated or non-deprotonated nitrogen donor sites.

Based on the experimental results, the complexes have been assigned tentative structures, in which the amide ligands behave as bidentate donors. Each Ni(II) ion is coordinated by two amide ligands along with two exogenous ligands, yielding stable octahedral complexes. These findings highlight that the synthesized compounds are structurally consistent with those obtained by conventional methods while offering the advantage of greener, more efficient synthesis routes.

References

1. Pecoraro V L, Li X, Baker M J, Butler W M, Bonadies J A, Recueil Trav. Chim. 1987, 106, 221.
2. Garg B S, Bhojak N, Sharma R K, Bist J S and Mittal S, Talanta, 1999 48 (1) 49-55.
3. Garg B S, Bhojak N, Nandan D, Ind. J. Chem., 2005, 44A, 1504.
4. Solanki K., Sharma K., Soni M. and Bhojak N.,
5. Bhojak N, Gudasaria D D, Khiwani N & Jain R, E-Journal of Chemistry, 2007 4 (2) 232-237.
6. Singh B K, Bhojak N, & Prakash A, E-Journal of Chemistry, 2012 9(2) 532-544.
7. Ncioki M, Shin- Inchi S & Yoshiaki W, Jpn. J. Appl. Phys; 2008 47 4235.
8. Mohamed MM, El-Fiky SA, Soheir YM & Abeer AI, Asian Journal Cell Biology, 2008 3 (2) 51.
9. Fontaine L A, Sharon M & Julian M, Archives of Biochemistry and Biophysics, 2007 463 (2) 149.
10. Vishal D & Kaler Stephen G, American Journal of Clinical Nutrition, 2008 88, (3) 855.
11. Krupanidhi S, Arun S & Sanjeevi CB, Indian J. Med. Research, 2008 128 448.
12. Kabanos T A and Tsangaris J M, J. Coord. Chem., 1984 13 89.
13. Nonoyama M and Yamasaki K, Inorg. Chim. Acta, 1973 7 (4) 676.
14. Barnes D J, Chapman R L, Stephen F S & Vagg R S, Inorg. Chim. Acta, 1981 51 155.
15. Ray P and Sen D N, J. Indian Chem. Soc., 1948 25 473.
16. Polder D, Physica, 1942 9 709.
17. Figgis B N and Nyholm R S, j.Chem.Soc. A 1959 338.
18. Ferguson J, Prog. Inorg. Chem. 1970 12 159.
19. Sutton D, "Electronic Spectra of transition metal ions" Mc. Graw Hill, London, 1968.
20. Ballhausen CJ, "Introduction of Ligand Field Theory" Mc. Graw Hill, New York 1962.
21. Procter LM, Hathway BJ, and Nicholls P, J. Chem. Soc., A 1968 1678.

