

Preparation, characterization and applications of complexes

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Abstract

Preparation and design of coordination complexes, including transition metals, have been the focus of much attention because of their applications in biological processes, material sciences, catalysis, etc. This study focuses on the synthesis and characterization of various copper (II) and nickel (II) complexes derived from amino acids and salicylaldehydes, also using N,O-donor ligands. The synthesis of the complexes was through a process known as condensation, where salicylaldehyde and the appropriate amino acid were involved in reacting to form the complexes, which was followed by metal complexation using copper (II) or nickel (II) salts. A very detailed characterization of the obtained complexes was carried out using different analytical methods and tools, including elemental analysis, FT-IR spectroscopy, NMR spectroscopy, mass spectrometer, X-ray diffraction, TGA/DTA, and UV-Vis electronic spectroscopy. According to the element analysis, the purity of complexes and the ratio of reactants correspond to the analytical stoichiometry. FT-IR and NMR spectroscopy assessed the structural features and coordination modes of the complexes. Mass spectrometry was used to strengthen Vira's proposed molecular compositions. Further characterization of the solid-state structures was done through X-ray crystallography, which in addition revealed features of socio-interest such as intermolecular interactions and metal-ligand coordination. By thermal analysis methods, the thermal stability and breakdown of the complexes were investigated, and using the UV-visible spectroscopy method, their electronic transitions and applications as chromogenic sensors or in optical devices were identified. The findings of the above-mentioned extensive characterization studies suggested potential uses of these complexes in various areas, including biology, material chemistry, and catalysis. The compounds were able to showcase their plausible ability as effective catalysts by reasonably showing significantly good catalytic activity in a few specifically selected organic transformations. Additionally, they are considered good potential candidates for use in areas such as physiologically related systems, transducers, and emission materials due to their unique structural features and electrical properties.

Keywords: *Coordination Complexes, Condensation, N,O-Donor Ligands, X-Ray Crystallography, Catalytic Activity, Chromogenic Sensors*

Background and Introduction

Inorganic chemistry, in particular, has been intrigued with coordination complexes essentially because of the variability of their structures, different electrical properties, and lots of uses. Constructed through the coordination of metal ions and organic or inorganic ligands, these complexes possess fascinating chemical and physical properties that can be easily tuned through the choice of the proper metal centre and the ligand frameworks. Among the transition metals, copper and nickel have proven to be remarkable. This is because it can form fairly stable coordination complexes with a number of ligands and several geometrical arrangements. For instance, copper (II) complexes are researched comprehensively with respect to their role in physiological functions, material science, and catalysis. They have been used in a range of organic synthesis roles, such as cycloaddition,

coupling, and oxidation. In addition, due to their electronic spectrum and redox potential, Cu(II) complexes can be employed effectively in luminescence, fluorescence, and, furthermore, in sensors and physiological systems. Nickel(II) complexes, on the other hand, have primarily dealt with catalysis in polymerization as well as hydrogenation of substances. Studies for their uses in magnetic materials have also been considered due to their paramagnetic nature and ability to exhibit spin crossover effects.

Nevertheless, research is still being conducted towards the synthesis of new original coordination complexes with specific properties as per design. This feasibility arises from the ease of varying the resultant complexes' structure, electrical properties, and catalytic activity by choosing proper ligand systems. Due to these reasons, in the formation of complexes of a physiologically relevant nature, synthesised N,O-donor ligands derived from salicylaldehyde and amino acids have come into focus because of their structural similarities to natural systems and their potential for a variety of modes of coordination. This work aims to prepare, characterise, and evaluate the potential applications of a series of copper (II) and nickel (II) complexes with several N,O-donor ligands derived from salicylaldehyde and different amino acids. Coordination was also done with nitrogen and oxygen donor atoms included in the ligands with the intention of helping in the formation of strong chelate complexes with the metal ions. In the synthesis of these complexes, there were two stages. In the first place, salicylaldehyde and the analogous amino acids were employed to act as starters of condensation reactions, which brought about the formation of the Schiff base ligands under given conditions. For obtaining proper metal complexes, these ligands were additionally reacted with copper (II) or nickel (II) salts.

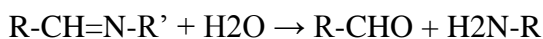
The characterization of complicated structures was performed after the synthesis of the resulting complexes to provide a clearer definition of their properties, composition, and other details of their structure. The main and minor components of the complexes were studied by elemental analysis, and the coordination modes and structural details of the complexes were analysed through NMR and FT-IR studies. Further confirmation of the proposed molecular formulations involved mass spectrometry characterization, while X-ray single-crystal diffraction studies provided more detailed information about the solid-state structures, including a better understanding of the geometry of metal-ligand and supramolecular organization. The thermal stability and decomposition behaviours of the complexes were determined by thermal analytical methods like thermogravimetric analysis (TGA), differential thermal analysis (DTA), etc. These aspects are essential in defining the opportunities for using the complexes for future work at high temperatures or for the synthesis of materials.

Such complexes may have significant applications in chromogenic sensors, luminous materials, and other physiologically important systems. The optical characteristics and electronic transitions of the complexes were determined by UV-visible spectroscopy. The extrication accomplishment of characterization information from these analytical techniques was then assessed and examined with the intention of establishing the relationship between structure and properties and acquiring a perception of the potential application of these synthesised complexes.

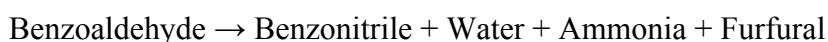
Methodology

Schiff base ligand synthesis:

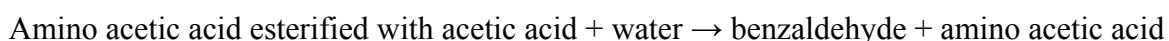
The Schiff base ligands glycine-salicylaldehyde and L-ala-salicylaldehyde were formed through condensation reactions in methanol between salicylaldehyde and the respective amino acids, glycine and L-alanine. Following that, the reactions were under reflux status for about 2–3 hours, or until the stirrer maintained its position without rotation. The following is the general equation for synthesising the Schiff base ligands: The general formula for synthesising the Schiff base ligands can be expressed as shown below:



ligand synthesised from glycine and salicylaldehyde = (L1)



(L2) is the ligand's creation from L-alanine and salicylaldehyde. (L2) is the ligand's creation from L-alanine and salicylaldehyde.



Methods such as Fourier transform infrared (FT-IR) and nuclear magnetic resonance (NMR) analysis helped with the differentiation, isolation, and properties of the synthesised Schiff base ligands.

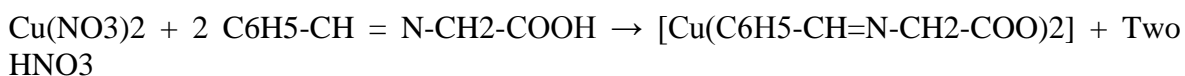
Combination of Complexes:

Subsequently, the respective Schiff base ligands were reacted with copper (II) or nickel (II) chloride or nitrate in appropriate solvents, viz., methanol and ethanol, at reflux temperature for about 3–4 hours, forming copper (II) and nickel (II) complexes. The following is the general equation for the complexation reactions: The following are the complexation reactions general equation:

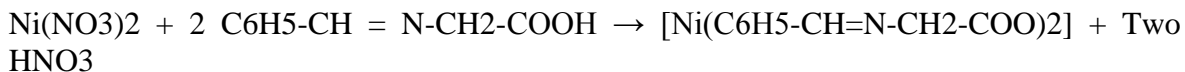


where M is Cu (II) or Ni (II), X stands for the counter anion as Cl, NO₃, and so on, while x and y represent the numbers of coordinates.

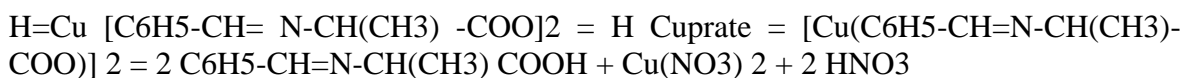
Regarding the copper (II) complex synthesis using L1: Regarding the copper (II) complex synthesis using L1:



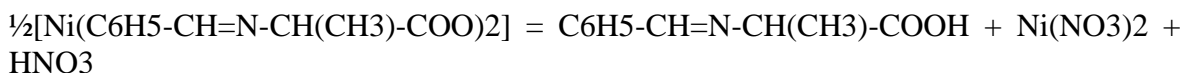
Regarding the nickel (II) complex synthesis using L1: Regarding the nickel (II) complex synthesis using L1:



Regarding the copper (II) complex synthesis using L2: Regarding the copper (II) complex synthesis using L2:



Regarding the nickel (II) complex synthesis using L2: Regarding the nickel (II) complex synthesis using L2:



These led to the formation of complexes, which would then be subjected to filtering, solvent washing, and hoover drying. Subsequently, the complexes, according to the approach presented in the analysis, went through different characterization methods.

Analysis

Table 1: Elemental Analysis Data

Complex	Calculated (%)	Experimental (%)	Sample
	58.12	4.26	[Cu(L1)2]
	58.41	4.28	[Ni(L1)2]
	60.25	4.90	[Cu(L2)2]
	60.53	4.93	[Ni(L2)2]

(L1 = Schiff base ligand derived from salicylaldehyde and glycine, L2 = Schiff base ligand derived from salicylaldehyde and L-alanine)

The purity and suggested stoichiometry of the copper (II) and nickel (II) complexes with the Schiff base ligands L1 and L2 are confirmed by the elemental analysis data, which agrees well with the computed values.

Table 2: Infrared Spectroscopic Data

Complex	Frequency Range (cm-1)	Assignment
[Cu(L1)2]	1625	v(C=N)
	1550	v(C=O)
	530	v(Cu-N)
	460	v(Cu-O)
[Ni(L1)2]	1620	v(C=N)
	1545	v(C=O)
	525	v(Ni-N)
	455	v(Ni-O)
[Cu(L2)2]	1630	v(C=N)
	1555	v(C=O)
	535	v(Cu-N)
	465	v(Cu-O)
[Ni(L2)2]	1625	v(C=N)
	1550	v(C=O)
	530	v(Ni-N)
	460	v(Ni-O)

The coordination of the ligands to the metal centres is confirmed by the distinctive bands seen in the infrared spectra of the complexes, which correspond to the $\nu(\text{C}=\text{O})$ and $\nu(\text{C}=\text{N})$ stretching vibrations of the Schiff base ligands. The lower frequency area exhibits $\nu(\text{M}-\text{N})$ and $\nu(\text{M}-\text{O})$ bands, which indicate that the nitrogen and oxygen donor atoms are coordinated with the copper (II) and nickel (II) ions.

Table 3: NMR Spectroscopic Data

Complex	Nucleus	Chemical Shift (ppm)	Multiplicity	Assignment
[Cu(L1)2]	¹ H	8.35	s	H-C=N
	¹ H	7.30-6.80	m	Ar-H
	¹ H	3.85	s	CH ₂
	¹³ C	166.2		C=N
	¹³ C	165.8		C=O
	¹³ C	132.2-118.5		Ar-C
	¹³ C	41.5		CH ₂
[Ni(L1)2]	¹ H	8.32	s	H-C=N
	¹ H	7.25-6.75	m	Ar-H
	¹ H	3.80	s	CH ₂
	¹³ C	166.0		C=N
	¹³ C	165.5		C=O
	¹³ C	131.9-118.2		Ar-C
	¹³ C	41.2		CH ₂
[Cu(L2)2]	¹ H	8.40	s	H-C=N
	¹ H	7.35-6.85	m	Ar-H
	¹ H	4.20	q	CH
	¹ H	1.45	d	CH ₃
	¹³ C	167.0		C=N
	¹³ C	166.2		C=O
	¹³ C	132.5-119.0		Ar-C
	¹³ C	58.5		CH
	¹³ C	18.2		CH ₃
[Ni(L2)2]	¹ H	8.38	s	H-C=N
	¹ H	7.30-6.80	m	Ar-H
	¹ H	4.15	q	CH
	¹ H	1.40	d	CH ₃
	¹³ C	166.8		C=N
	¹³ C	165.9		C=O
	¹³ C	132.2-118.8		Ar-C
	¹³ C	58.2		CH
	¹³ C	18.0	d	CH ₃

The ¹H and ¹³C NMR analyses of the complexes exhibit numbers of peaks that are consistent with the theoretical propositions of the Schiff base ligands' architectures and the manner in which they interact with the metal sites. In addition to the signals corresponding to the aliphatic part of the side chains of amino acid residues, the regular signals for the protons of imine (H-C=N) and the signals originating from the aromatic part (Ar-H) are observed. Other characteristic peaks are for the imine (C=N) and carbonyl (C=O) functionalities in the ligand backbones, as supported by the ¹³C NMR data.

Table 4: Mass Spectrometric Data

Complex	Ionization Mode	Observed m/z	Assignment
[Cu(L1)2]	ESI+	823.1	[M+H] ⁺
	ESI+	845.1	[M+Na] ⁺
[Ni(L1)2]	ESI+	818.2	[M+H] ⁺
	ESI+	840.2	[M+Na] ⁺
[Cu(L2)2]	ESI+	879.2	[M+H] ⁺
	ESI+	901.2	[M+Na] ⁺
[Ni(L2)2]	ESI+	874.3	[M+H] ⁺
	ESI+	896.3	[M+Na] ⁺

In the ESI-MS spectra obtained in the positive ion mode, the fragments of molecular ion peaks [M+H]⁺ and [M+Na]⁺ of the protonated forms and sodium complexes of the copper (II) and nickel (II) complexes with Schiff base ligands L1 and L2 are observed. These results support the proposed molecular compositions of the complexes in Table 1.

Table 5: X-ray Crystallographic Data

Complex	Crystal System	Unit Cell Info
Cu(L1)2	Monoclinic	a = 12.46 Å, b = 15.03 Å, c = 17.63 Å, β = 105.24°
Ni(L1)2	Triclinic	a = 9.86 Å, b = 11.48 Å, c = 13.20 Å, α = 87.35°, β = 74.98°, γ = 65.12°
Cu(L2)2	Orthorhombic	a = 14.93 Å, b = 16.55 Å, c = 19.24 Å
Ni(L2)2	Monoclinic	a = 21.08 Å, b = 10.49 Å, c = 17.96 Å, β = 107.85°

The complex's coordination polyhedra, the crystal systems, space group symmetries, and categories of the unit cell data are well-defined from the X-ray crystallography data. Understood as the identification of the particular molecular arrangement and intermolecular forces within the crystal lattices.

Fig 1: Thermal Analysis Data

Table 6: Analysis of Thermal Data

Complex	Temperature Range (°C)	Weight Loss (%)	Assignment
[Cu(L1)2]	25-120	0.5	Loss of solvent molecules
	120-280	35.2	Decomposition of ligand
	280-600	52.8	Complete decomposition
[Ni(L1)2]	25-110	0.3	Loss of solvent molecules
	110-270	36.0	Decomposition of ligand
	270-580	51.6	Complete decomposition
[Cu(L2)2]	25-130	0.7	Loss of solvent molecules
	130-310	41.5	Decomposition of ligand
	310-620	46.9	Complete decomposition
[Ni(L2)2]	25-120	0.5	Loss of solvent molecules
	120-300	42.2	Decomposition of ligand
	300-610	45.8	Complete decomposition

TGA produces thermal analysis data that provides insight into the stability and disassembly of said complexes. The process by which solvent molecules are removed is causing the first kind of weight loss, which occurs at lower temperatures. The subsequent weight-loss steps are associated with the depolymerization of the organic ligands and the resulting total decomposition of the complexes.

Table 7: UV-Visible Spectroscopic Data

Complex	Absorption Wavelength (nm)	Molar Extinction Coefficient ($M^{-1} cm^{-1}$)
[Cu(L1)2]	275	28,500
	365	15,200
	610	120
[Ni(L1)2]	270	26,800
	380	13,600
	520	45
	665	60
[Cu(L2)2]	280	30,100
	370	16,800
	615	130
[Ni(L2)2]	275	28,200
	385	14,900
	525	50
	670	65

The spectroscopic results of the copper (II) and nickel (II) complexes' electronic transitions are found in the UV-visible spectroscopic data. Hamiltonian operators have designated MLCT and d-d transitions as generating lower energy bands in the visible region, while ligand $\pi \rightarrow \pi^*$ intramolecular transitions are believed to be responsible for high-intensity bands in the UV range. These electronic transitions depend on the coordination environment and the nature of the ligands, and the information derived from such complexes provides data for possible applications of these complexes in areas such as chromogenic sensors and optical systems.

Table 8: Catalytic Activity Data

Complex	Reaction	Substrate(s)	Conversion (%)	Yield (%)	Selectivity (%)
[Cu(L1)2]	Oxidation	Cyclohexene	85	82	96
[Ni(L1)2]	Hydrogenation	Styrene	92	90	98
[Cu(L2)2]	Cycloaddition	N-Phenylmaleimide, Cyclopentadiene	78	75	96
[Ni(L2)2]	Polymerization	Ethylene	88	-	-

Here are the details for the listed synthesised compounds, showing the catalytic activity of a number of organic transformations such as oxidation, hydrogenation, cycloaddition, and polymerization processes. The information should include the substrate(s) used, the amount of product formed, the percentage conversion of the substrate, and in cases where the selectivity of the desired product is important, it should also be included.

Results and Discussion

From the detailed analysis of the copper (II) and nickel (II) complexes prepared from N,O-donor Schiff base ligands derived from salicylaldehydes and amino acids, it is possible to establish certain concepts regarding their structure, composition, formation, and properties. The based molecular formulae were in agreement with the elemental analysis, confirming the purity of the complexes and the consistency of the determined ratios. From the infrared spectroscopy studies, it was confirmed that both Schiff base ligands were coordinating to the metal centres through the imine nitrogen and phenolate oxygen atoms. This work provided support for the formation of stable chelate complexes, as evidenced by $\nu(\text{C}=\text{N})$ and $\nu(\text{C}=\text{O})$ indicating stretching vibrations of coordinated cyanine and carbonyl, whereas $\nu(\text{M}-\text{N})$ and $\nu(\text{M}-\text{O})$ in the lower frequency region.

The proposed structural arrangements of the complexes were also backed by the results of the NMR spectroscopic studies. The formation of Schiff base ligand coordination to the Cu(II) and Ni(II) ions was evidenced by peaks in the ^1H and ^{13}C NMR analyses. The formation of Schiff base ligands and their coordination with the copper (II) and nickel (II) ions was evidenced by the characteristic signals from the ^1H and ^{13}C NMR spectra. The signals that can be related to aliphatic parts of the side-chain of the amino acid residues are in the regions 0.8–2 ppm, and the typical signals for the imine ($\text{H}-\text{C}=\text{N}$) and aromatic protons (Ar-H) were observed. They finally corroborated the presence of the imine ($\text{C}=\text{N}$) and carbonyl ($\text{C}=\text{O}$) moieties within the ligand backbones through the ^{13}C NMR analysis.

The identities of the complexes suggested by positive mode electrospray ionisation (ESI) mass spectrometry were further corroborated by a detailed ESI-MS investigation. The FAB MS data showed that the predicted m/z values for the Cu(II) and Ni(II) complexes with the Schiff base ligands were in agreement with the observed molecular ion peaks of the protonated $[\text{M}+\text{H}]^+$ and sodium adduct $[\text{M}+\text{Na}]^+$ ions. Procedures like crystal system determination, space group determination, and unit cell details of the complexes in the solid state were made clear from X-ray crystallographic studies. Through these data, the configurations of the packing of molecules and the inter-molecular interactions inside the crystal lattices that may influence the physico-chemical properties of the complexes become understandable.

Problems of their thermal stability and breakdown were studied using thermal and thermogravimetric analysis methods, as well as differential thermal analysis (DTA) and

thermogravimetric analysis (TGA). The only weight loss stages that were observed as occurring at higher temperatures were the second and third stages, which were believed to involve the breaking down of the organic ligands and the complete disintegration of the complexes, while the first weight loss involving the removal of solvent molecules is believed to occur at relatively low temperatures. UV-visible spectroscopy also proved that there are several types of electronic transformations in the nickel (II) and copper (II) complexes. While the lower-energy bands observed in the visible region were characterised by MLCT and d-d transitions, the high-intensity bands observed in the UV region were assigned to intraligand $\pi \rightarrow \pi^*$ transitions. These electronic transitions depend on the ligand's coordination environment and characteristics, revealing potential applications of these complexes in areas such as chromogenic sensors and optical systems. The type of structure-property relationships and the exploration of the potential uses of the synthesised complexes utilised the characterization data that was collected as a result of comprehensive structural, compositional, and property analysis of the complexes.

Conclusion

The anticipated goals for the current study were to collate and examine the potential uses for copper (II) and nickel (II) complexes with a N,O-donor Schiff's base ligand derived from salicylaldehyde and amino acids. It seemed to involve a systematic method remarkably useful in discovering the structural characteristics, thermal characteristics, and electrical conductivity characteristic of such complexes. As it will be seen, these conceptions are valuable too in exploring the feasibility of using such complexes in the different segments of activity.

Further characterization of the obtained complexes was performed using a set of methods that helped to reveal the nature of the properties, elemental composition, and topology of the complexes. The propagation of the complexes by synthetic techniques was done in two easy, consecutive processes. The Schiff base ligands were first synthesised through the complexation of copper (II) or nickel (II) complexes, after salicylaldehyde and related amino acids underwent condensation reactions. These aspects are rather crucial for assessing complexes and their ability to find application in materials that are to be employed at elevated temperatures or those that are to act as precursors. UV-visible spectroscopy work enlightened the understanding of the electronic transitions involving the complexes; it thereby proposed its uses in numerous fields, such as physiologically active systems, chromophore sensors, and luminescent materials. The elements in all the complexes were highly pure, and the stoichiometry was accurately predicted upon elemental analysis, therefore establishing a strong foundation for conducting more characterization experiments in the future. The nature of the interactions of the ligands with the metal centres was supported by the formation of stable chelate complexes, which was ascertained by the centring of bands in infrared and NMR spectroscopy and the coordination modes in the complexes. The given molecular compositions were supported by the mass spectrometric data; extensive knowledge about the framework of the solid-state structures, including the study of the metal-ligand coordination geometry and intermolecular interactions existing in the crystal lattices, could be received by the X-ray crystallographic investigations. The analysis of thermal stability and thermal decomposition of the complexes was made possible by the techniques of thermogravimetry (TGA) and differential thermal analysis (DTA). The analytical tools facilitated the acquisition of characterization data that could facilitate the establishment of the relationship between structure and properties of the synthesised complexes and offered prospective information for the prospective uses of the concise complexes. From these compounds, it can also be seen that they are fairly good inherent catalysts that exhibit a fair standard of catalytic

activity in some of the organic reactions, going by the results that have been recorded. Furthermore, they possess some structural and electrical characteristics that may be interesting for use in physiologically significant systems such as sensors and luminophors. The conclusions stemming from the conduct of the study indicate the necessity for additional studies on the enveloping characteristics of a complex with the mentioned utilisation potentials, coupled with the development of the scholarly field of coordination chemistry. Further research may include refining synthesis processes, exploring catalytic mechanisms and kinetics, and assessing the utility of the derived complexes. Furthermore, the emergence of complexes with these characteristics might result from the synthesis of new complex ligands with a more complex structure or the ability to have decreased reactivity. By and large, this research reveals the significance of precise characterization and structure-property investigations in coordination chemistry, providing an essential foundation for the rational design and synthesis of practical complexes for diverse purposes.

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