Research Article

Synthesis And Characterization of Indole-Based Glycylglycine Schiff Base Metal Complexes

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Abstract

Schiff base metal complexes have attracted significant attention in bioinorganic and medicinal chemistry because of their versatile coordination behavior, structural diversity, and potential biological applications. The present study focuses on the synthesis and characterization of two tridentate Schiff base ligands derived from glycylglycine and their corresponding transition metal complexes. The ligands were prepared through condensation reactions involving glycylglycine with indole-3-carboxaldehyde (SF1) and cumin aldehyde (SF2). Subsequently, complexes of these ligands with cobalt(II), nickel(II), copper(II), and zinc(II) ions were synthesized and subjected to comprehensive physicochemical characterization. The structures of the synthesized ligands and metal complexes were investigated using appropriate analytical and spectroscopic techniques. Spectral data indicated the involvement of azomethine nitrogen, peptide nitrogen, and carboxyl ate oxygen atoms in metal coordination, supporting the proposed tridentate binding mode of the ligands. Molar conductance measurements provided information regarding the electrolytic nature of the complexes. Furthermore, electron spin resonance (ESR) spectroscopy and magnetic susceptibility measurements supported a square-planar geometry for the copper(II) complex. Thermo gravimetric analysis revealed multistep thermal decomposition of the synthesized compounds, ultimately resulting in the formation of stable metal oxide residues. X-ray diffraction (XRD) analysis confirmed the crystalline or nanocrystalline nature of the investigated materials, with estimated crystallite sizes ranging from 31 to 67 nm. Overall, the findings demonstrate that the synthesized glycylglycine-based Schiff base ligands exhibit versatile coordination properties toward selected transition metal ions, producing complexes with distinct structural and physicochemical characteristics. The combined spectroscopic, magnetic, thermal, and diffraction results provide valuable insights into their coordination behavior and solid-state properties. These findings establish a foundation for further investigation of the biological activities and pharmacological potential of the synthesized compounds. Additional biological evaluation will be necessary to determine their efficacy, safety, and suitability for potential medicinal applications.

Keywords: Indole-Based Schiff Bases, Glycylglycine Metal Complexes, Transition Metal Coordination, Spectroscopic Characterization, Biological Activity

1. Introduction

Schiff bases are widely recognized as important ligands because they can be prepared easily through the condensation of primary amines with carbonyl compounds such as aldehyde or ketones. These compounds were first reported by the German chemist and Nobel laureate Hugo Schiff in 1864. Their ability to interact with transition-metal ions makes them an important class of compounds in coordination chemistry. Schiff base ligands can coordinate with a wide range of metal ions, mainly through the nitrogen atom of their azomethine group. Their simple preparation, easy availability, structural flexibility, and useful electronic properties have led to extensive research in coordination chemistry. In recent years, Schiff base complexes have attracted significant attention because of their applications in areas such as analytical chemistry, organic synthesis, metallurgy, metal refining, electroplating, and photography [1].

Schiff bases also represent a versatile group of ligands due to their straightforward synthesis, adaptable structures, and strong ability to bind metal ions. The characteristic azomethine (–C=N–) group plays a central role in their coordination with transition metals. Metal complexes of Schiff bases have been extensively investigated for their diverse biological properties, including antibacterial, antioxidant, anti-inflammatory, and anticancer activities. These characteristics make Schiff base metal complexes promising candidates for further research in medicinal and pharmaceutical applications.

Transition metals are key elements in many biological systems and play a critical role in enzymatic and metabolic processes. Metal cations may bind with nitrogen and oxygen donor atoms found in proteins and nucleic acids, affecting the architecture and functionality of biological macromolecules. The potential of transition metal complexes to imitate metalloenzymes and facilitate DNA breakage has made them interesting candidates in the creation of metallodrugs [2].

Indole derivatives are extensively found in natural substances and medications, demonstrating a variety of biological functions including antimicrobial, analgesic, antioxidant, and anticancer properties. The integration of indole groups into Schiff base ligands amplifies biological efficacy and augments metal-binding capability. Moreover, glycylglycine-derived ligands provide many coordination sites that promote the formation of stable metal complexes with improved lipophilicity and membrane permeability [3].

The complexation of metals often amplifies the biological efficacy of Schiff bases via chelation, which diminishes the polarity of the metal ion and elevates the lipophilicity of the complex. This facilitates permeation across biological membranes and augments engagement with bimolecular targets including DNA and proteins. Copper complexes among transition metals are notably appealing because of their redox characteristics and capacity to produce reactive oxygen species, which can trigger oxidative stress and apoptosis in cancerous cells.

Considering these factors, the current research sought to synthesize indole-derived glycylglycine Schiff base ligands along with their Co(II), Ni(II), Cu(II), and Zn(II) complexes, and to characterize them through diverse spectroscopic and analytical methods [3,4].

2. Material And Methods

2.1 Synthesis of Schiff base ligands

Schiff bases have become important chelating ligands in the coordination chemistry of both main-group and transition metals. Their stability under different redox conditions and the moderate Lewis basicity of their imines groups contribute to their widespread use. The physical and biological properties of Schiff bases are strongly influenced by intermolecular hydrogen bonding and proton-transfer processes. These compounds also offer useful possibilities for introducing chirality into molecules, modifying the electronic behavior of metal centers, and improving the solubility and stability of homogeneous as well as heterogeneous catalysts.

Transition metals are essential for many biological processes and contribute to the structure and activity of several important biomolecules. They can interact with oxygen- and nitrogen-containing groups of proteins in different coordination arrangements. In bioinorganic chemistry, Schiff base metal complexes are therefore extensively studied as synthetic models of the metal-binding sites found in metalloenzymes and metalloproteins.

Indole and its derivatives are widely utilized in the preparation of pharmaceuticals, fragrances, dyes, and agricultural chemicals. In addition, tridentate and tetra dentate Schiff base complexes have attracted increasing attention because of their potential to model both synthetic and naturally occurring oxygen-transport systems. Copper complexes containing tridentate Schiff base ligands, especially dimeric complexes, have been investigated as useful bioinorganic models [5].

In recent years, considerable research has focused on indole-based compounds because many of them exhibit valuable biological activities, including antibacterial and analgesic effects. Another important area of research is the development of metal-based drugs for cancer treatment. The ability of certain metal complexes to interact with and cleave DNA is considered an important factor in their potential chemotherapeutic activity. Because of their redox properties, several transition-metal complexes have demonstrated the ability to promote DNA cleavage. Various Schiff base metal-complex systems have also been reported in earlier studies [6].

Schiff bases are generally synthesized through a condensation reaction between primary amines and aldehydes or ketones, resulting in the formation of an imine (–C=N–) linkage. This reaction typically proceeds via nucleophilic addition of the amine to the carbonyl group followed by dehydration [7].

2.2 Synthesis of Schiff Base Ligand SF1 (Indole-3-carboxaldehyde and Glycylglycine)

Glycylglycine (1 m mol) was dissolved in 20 mL of methanol containing KOH (1 m mol). A methanolic solution of indole-3-carboxaldehyde (1 m mol in 10 mL methanol) was added slowly to the mixture with continuous stirring. The reaction mixture was refluxed for approximately 2 hours. After completion, the yellow solution was cooled to room temperature and concentrated under reduced pressure using a rotary evaporator. Addition of anhydrous diethyl ether produced a yellow precipitate. The resulting solid was collected and purified by recrystallization from ethanol.

2.3 Synthesis of Schiff Base Ligands SF2 (Cuminaldehyde and Glycylglycine)

Glycylglycine (10 m mol) and KOH pellets (10 m mol) were dissolved in 50 mL of methanol in a 100 mL round-bottom flask with continuous stirring. A methanolic solution of cuminaldehyde (10 m mol in 50 mL methanol) was added gradually to the reaction mixture. The mixture was stirred vigorously at room temperature for 15 minutes and then refluxed at 70–80 °C for 8 hours. After reflux, the solution was concentrated to approximately one-third of its original volume using a rotary evaporator. The concentrated solution was poured into diethyl ether, resulting in the formation of a yellow precipitate. The precipitate was separated by vacuum filtration, washed several times with anhydrous ether, and dried under vacuum over anhydrous calcium chloride. The Schiff base ligand was obtained with an isolated yield of approximately 72% [8].

2.4 Preparation of Schiff Base Metal Complexes

Individual Schiff base ligands (SF1 and SF2) were dissolved in 10 mL of methanol. A metabolic solution containing 1 m mol of Co(II), Ni(II), or Cu(II) nitrate was then added gradually with continuous stirring. The resulting reaction mixture was stirred for 2 hours and allowed to stand in air at room temperature. The precipitated metal complex was collected by filtration, washed thoroughly with cold ethanol and diethyl ether to remove unreached materials, and finally dried under vacuum over anhydrous calcium chloride [9].

2.5 Characterization and Physical Measurements

The synthesized Schiff base ligands and their metal complexes were characterized using various analytical and spectroscopic techniques. Elemental composition was determined using a Perkin–Elmer elemental analyzer, while mass spectra were recorded on a JEOL SX 102/DA-600 mass spectrometer. The ^1H and ^13C NMR spectra of the Schiff base ligands were obtained using a JEOL GSX 400 FT-NMR spectrometer.

The molar conductance of the complexes was measured in metabolic solutions (10^{-3} M) using a calibrated digital conductivity meter. FTIR spectra were recorded using KBr pellets over the range of 4000–400 cm^{-1} with a JASCO FT/IR-410 spectrometer. UV–Visible spectra were obtained using a Perkin Elmer Lambda-25 spectrophotometer. Magnetic susceptibility measurements were performed at room temperature using a Gouy balance, with diamagnetic corrections calculated using Pascal's constants [10,11].

The X-band ESR spectrum of the Cu(II) complex was recorded using a Varian E112 spectrometer, with TCNE used as the reference. Electrochemical behavior was investigated by cyclic voltammetry using a BAS CV-50 W electrochemical analyzer. A three-electrode arrangement consisting of an Ag/AgCl reference electrode, platinum auxiliary electrode, and glassy-carbon working electrode was employed, with tetrabutylammonium perchlorate serving as the supporting electrolyte.

Thermal stability was examined using an SDT Q600/V8.3 Build 101 thermal analyzer. The instrument was calibrated with pure metal standards, and approximately 6–8 mg of each sample was analyzed at a heating rate of 20 °C/min under a nitrogen flow of 20 mL/min. Powder X-ray diffraction patterns were recorded using a Rigaku Dmax diffractometer with Cu Kα radiation (λ = 1.5406 Å). Surface morphology of the synthesized compounds was examined by scanning electron microscopy using a Hitachi SEM instrument [12,18].

3 RESULTS

3.1 Synthesis and Characterization of the Ligand

The synthesized Schiff base ligand was obtained as a pale-yellow solid and showed good solubility in most organic solvents. Its composition was confirmed through elemental analysis and various spectroscopic techniques. The experimental elemental analysis values were in close agreement with the calculated theoretical values, supporting the proposed molecular composition.

The FTIR spectrum of indal-glygly showed a characteristic azomethine (C=N) stretching band at 1618 cm^{-1}. The asymmetric and symmetric stretching vibrations of the carboxylate group [ν_asym(COO) and ν_sym(COO)] appeared at 1587 and 1384 cm^{-1}, respectively. A band at 1532 cm^{-1} was assigned to the amide group of the potassium salt of the Schiff base. Importantly, the N–H stretching band of glycylglycine at approximately 3100 cm^{-1} and the C=O stretching band of indole-3-carboxaldehyde at around 1700 cm^{-1} were absent in the final product. These spectral changes confirm the condensation reaction between indole-3-carboxaldehyde and glycylglycine and support the formation of the indal-glygly Schiff base. The UV–Visible spectrum showed an absorption maximum at 307 nm, which can be attributed to the π–π* transition associated with the azomethine chromophore.

The ^1H NMR spectrum further supported the proposed structure of indal-glygly. A signal at approximately 8.2 ppm was assigned to the azomethine proton (–HC=N–), while the amide proton (–CONH–) appeared at about 7.6 ppm. The indole N–H proton produced a signal near 10.3 ppm. The aromatic protons appeared as a multiplet in the region of 7.4–8.0 ppm, whereas the two aliphatic methylene protons ($–CH_2–$) gave a multiplet between 3.8 and 4.2 ppm.

The FAB mass spectrum recorded at room temperature showed a molecular ion/base peak at m/z 259, corresponding to the molecular composition $C_{13}H_{12}N_3O_3^+$. Additional fragment ions were observed at m/z 143 and 116, which were assigned to $C_{11}H_9N_2O^+$ and $C_9H_7N_2^+$, respectively. Overall, the elemental and spectroscopic findings support the proposed structure of the indal-glygly Schiff base ligand. The ligand contains several potential donor sites and can coordinate with metal ions through the azomethine nitrogen, peptide nitrogen/oxygen, and carboxylate oxygen atoms.

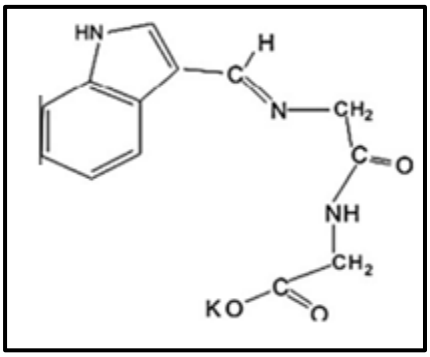

Figure 1. Proposed structure of Schiff base ligand SF1

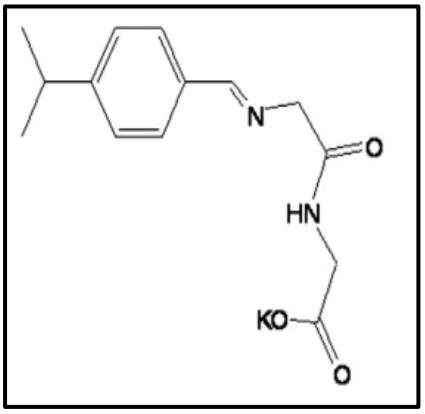

Figure 2. Proposed structure of Schiff base ligand SF2

3.1 Synthesis and characterization of metal complexes

The synthesized metal complexes were found to be stable under normal atmospheric conditions and showed good stability in air. They were readily soluble in DMSO but exhibited poor solubility in most common organic solvents. The analytical data presented in Table 1 confirmed a consistent 1:1 metal-to-ligand ratio for all the complexes. The proposed general formula of the complexes is $[ML(H_2O)]NO_3$, where L represents the Schiff base indole-3-carboxaldehyde-glycylglycine (indal-glygly), while M corresponds to Co(II), Ni(II), or Cu(II).

Molar conductivity measurements were performed in 10^{-3} M DMSO solutions at room temperature to investigate the ionic nature of the complexes. The relatively high conductivity values indicated that all the synthesized complexes behave as 1:1 electrolytes in solution. This observation suggests that the nitrate ion remains outside the coordination sphere and does not directly participate in bonding with the metal center.

Table 1. Physical data of Schiff base ligand and its complexes

Compound	Formula	Colour
SF1	KC13H12N3O	Yellow
Co SF1	CoC13H14N4O7	Pink
Ni SF1	NiC13H14N4O7	Green
Cu SF1	CuC13H14N4O7	Blue
Zn SF1	ZnC13H14N4O7	Yellow
SF2	KC14H17N2O3	Yellow
Co SF2	CoC14H22N2O6	Brown
Ni SF2	NiC14H22N2O6	Light green
Cu SF2	CuC14H22N2O6	Deep green
Zn SF1	ZnC14H22N2O6	Light yellow

Table 2. Analytical data of Schiff base ligand and its complexes

Compound	Analytical (%) calculated (found)			
	Carbon	Hydrogen	Nitrogen	Metal
SF1	52.51 (52.35)	4.07 (4.05)	14.13 (14.08)	-
Co SF1	39.31 (39.71)	3.55 (3.20)	14.11 (14.55)	14.84 (14.54)
Ni SF1	39.33 (39.69)	3.55 (3.18)	14.11 (14.76)	14.78 (14.48)
Cu SF1	38.86 (38.64)	3.51 (3.16)	13.94 (13.67)	15.81 (15.91)
Zn SF1	39.12 (39.34)	3.56 (3.24)	14.02 (13.78)	15.92 (15.99)
SF2	55.87 (55.98)	5.65 (5.70)	9.45 (9.33)	-
Co SF2	45.21 (45.05)	6.01 (5.94)	7.43 (7.51)	15.63 (15.79)
Ni SF2	44.93 (45.08)	6.01 (5.94)	7.42 (7.51)	15.68 (15.73)
Cu SF2	44.39 (44.50)	5.68 (5.87)	7.56 (7.41)	16.74 (16.82)
Zn SF2	44.73 (44.28)	5.48 (5.84)	7.66 (7.38)	17.01 (17.22)

Table 3. Molar conductance of Schiff base ligand and its complexes

Compound	Molar conductance (Ω^{-1}, cm^2 mol^{-1})
SF1	-
Co SF1	69.81
Ni SF1	70.72
Cu SF1	73.32
Zn SF1	74.08
SF2	-
Co SF2	15.32
Ni SF2	12.24
Cu SF2	17.32
Zn SF1	10.23

3.2 Solubility, Stability, and Molar Conductance

The Schiff base ligands showed sensitivity to air and were soluble in several commonly used organic solvents. Among the synthesized complexes, the CuL_3H_2O complex was relatively sensitive to air, hygroscopic in nature, and soluble in water, DMF, and DMSO. In contrast, the other metal complexes were stable under normal atmospheric and moisture conditions and showed good solubility in acetonitrile, DMF, and DMSO. Molar conductivity measurements performed in DMSO at a concentration of 10^{-3} M indicated that the synthesized complexes behave as non-electrolytes, suggesting that no ions are released through dissociation in solution.

3.3 Mass Spectral Studies

The electron-ionization mass spectrum of Schiff base ligand SF2 showed distinct molecular ion peaks at m/z 301.26 and 292.87, with relative intensities of 23% and 18%, respectively. These values were consistent with the proposed molecular compositions of the ligands. The FAB mass spectra of the CoSF2, NiSF2, CuSF2, and ZnLSF2 complexes displayed molecular ion peaks at m/z 375.46, 373.31, 378.50, and 382.27, respectively, which correspond closely to their expected molecular masses.

Similarly, the SF1, NiSF2, CuSF2, and ZnSF2 complexes exhibited molecular ion peaks at m/z 366.13, 368.59, 407.11, and 390.78, respectively. The observed molecular ion peaks provided evidence for the proposed compositions of the complexes. The mass spectral data of the synthesized compounds further suggested that the complexes exist predominantly as monomeric species, supporting a 1:1 metal-to-ligand stoichiometry.

4. Infrared Spectral Studies

4.1 SF1 and Its Metal Complexes

The FTIR spectrum of the SF1 Schiff base showed a characteristic azomethine (C=N) stretching band at 1618 cm^{-1}. After coordination with metal ions, this band shifted toward lower frequencies, indicating the involvement of the azomethine nitrogen in bonding with the metal center. The peptide band of the free ligand appeared at approximately 1532 cm^{-1} and also shifted to lower wavenumbers after complex formation. This change suggests coordination through the peptide nitrogen atom.

The asymmetric carboxylate stretching vibration shifted toward higher frequencies, whereas the corresponding symmetric vibration moved toward lower frequencies after complexation. The difference between these two carboxylate stretching frequencies (Δν) was greater than 200 cm^{-1}, indicating that the carboxylate group coordinates to the metal ion in a monodentate manner through one oxygen atom.

Broad absorption bands observed around 3389–3415 cm^{-1} in the complexes were assigned to O–H stretching vibrations of coordinated water molecules. This assignment was further supported by the thermal analysis results. Additional bands in the region of 1361–1371 cm^{-1} were attributed to the presence of uncoordinated nitrate ions. New bands appearing at approximately 519–534 cm^{-1} and 417–449 cm^{-1} were assigned to metal–oxygen [ν(M–O)] and metal–nitrogen [ν(M–N)] vibrations, respectively.

Based on these spectral changes, SF1 acts as a tridentate Schiff base ligand. It coordinates with the Co(II), Ni(II), and Cu(II) ions through the azomethine nitrogen, peptide nitrogen, and carboxylate oxygen donor atoms. A water molecule occupies the remaining coordination position around the metal center.

4.2 SF2 and Its Metal Complexes

The FTIR spectrum of SF2 displayed a strong band at approximately 1665 cm^{-1}, corresponding to the azomethine (C=N) stretching vibration. Following complex formation, this band shifted toward lower frequencies, confirming coordination of the azomethine nitrogen with the metal ion.

The free Schiff base showed characteristic N–H stretching vibrations associated with the peptide group in the region of 3300–1520 cm^{-1}. These bands disappeared after complexation, indicating deprotonation of the peptide nitrogen and its involvement in metal coordination. A broad band near 3400 cm^{-1} appeared in the complexes and was attributed to the O–H stretching vibration of coordinated water. The band observed around 800 cm^{-1} also supported the presence of coordinated water molecules.

The asymmetric stretching vibration of the carboxylate group appeared near 1608 cm^{-1}, while the symmetric carboxylate stretching band of the free ligand was observed around 1385 cm^{-1}. Upon coordination, these bands shifted to lower frequencies, indicating interaction between the carboxylate oxygen and the metal center. The difference between the asymmetric and symmetric carboxylate stretching frequencies was approximately 200 cm^{-1} or greater, supporting monodentate coordination of the carboxylate group.

Furthermore, new absorption bands in the ranges of 520–540 cm^{-1} and 420–440 cm^{-1} were observed in the spectra of the metal complexes. These bands were assigned to metal–oxygen and metal–nitrogen stretching vibrations, respectively. Overall, the FTIR findings confirm that SF2 coordinates with the metal ions through the azomethine nitrogen, deprotonated peptide nitrogen, and carboxylate oxygen donor sites

4.3 Electron Spin Resonance (ESR) Studies

The ESR spectra of the Cu(II) complex were recorded in DMSO at room temperature (300 K) and at 77 K. At 300 K, the spectrum showed a broad and intense signal in the high-field region, which can be attributed to the rapid tumbling of molecules in solution and the resulting isotropic behavior. When the sample was examined in the frozen state at 77 K, the spectrum became more resolved, displaying four distinct signals in the low-field region along with a strong signal at high field.

The magnetic moment of the Cu(II)–SF1 (indal-glygly) complex was found to be 1.81 BM, indicating the presence of one unpaired electron and supporting the mononuclear nature of the complex. The absence of a half-field signal around 1600 G, which would normally arise from an ms=±2 transition, further indicates that there is no significant Cu–Cu interaction. The observed g-values suggest that the unpaired electron is mainly located in the dx2−y2 orbital, which is consistent with a square-planar arrangement around the Cu(II) center. The calculated G value of approximately 3.5 indicates considerable anisotropy in the copper environment. Overall, the ESR results support the presence of a square-planar Cu(II) complex in which the unpaired electron occupies the dx2−y2 orbital.

4.4 Thermal Analysis

Thermogravimetric and differential thermal analyses were used to investigate the thermal stability and decomposition behavior of the synthesized complexes.

For the Cu(II)–SF1 complex, thermal decomposition occurred in three major stages. The first stage appeared between 120 and 135 °C and was associated with the removal of coordinated water. The observed mass loss was 4.45%, which was close to the calculated value of 4.37%. The second decomposition step occurred between 210 and 235 °C and was attributed to melting accompanied by partial degradation of the ligand, resulting in an experimental weight loss of approximately 17% compared with the calculated value of 17.76%. The final major decomposition stage occurred between 310 and 335 °C and involved extensive degradation of the organic ligand, producing a weight loss of about 70%, close to the theoretical value of 70.21%. At temperatures above 400 °C, the remaining residue corresponded to the respective metal oxide.

The CoL_3H_2O, NiL_3H_2O, CuL_3H_2O, and ZnL_3H_2O complexes derived from SF2 displayed broadly similar thermal decomposition patterns, generally involving two principal stages. The first stage occurred approximately between 160 and 290 °C, with endothermic DTA signals appearing in the 160–200 °C region. This step was mainly related to the removal of coordinated water molecules. The final decomposition stage occurred between approximately 360 and 630 °C and involved the breakdown and removal of the organic ligand, ultimately leaving the corresponding metal oxide as the final residue.

For the $CoL_1{\cdot}3H_2O$ and $NiL_1{\cdot}3H_2O$ complexes, weight losses of 14.52% and 14.57% were recorded, compared with theoretical values of 14.77% and 14.78%, respectively. These losses occurred within the ranges of 150–225 °C and 154–216 °C and were assigned to the elimination of three coordinated water molecules. Endothermic DTA peaks around 220–225 °C further supported the dehydration process. Subsequent decomposition of the organic ligand occurred between 346–589 °C for the cobalt complex and 337–600 °C for the nickel complex. The corresponding DTA maxima appeared at approximately 430 and 437 °C, respectively. The experimental weight losses were 68.85% and 68.78%, which were close to the calculated values of 69.10% and 69.15%. Above these temperatures, the TG curves became nearly horizontal, indicating the formation of stable metal oxide residues.

The $CuL_1{\cdot}5H_2O$ and $ZnL_1{\cdot}4H_2O$ complexes showed three distinct stages of thermal degradation. The initial stage occurred between 52–120 °C for $CuL_1{\cdot}5H_2O$ and 80–120 °C for $ZnL_1{\cdot}4H_2O$. Endothermic DTA peaks were observed at approximately 133 and 78 °C, respectively. This initial mass loss was attributed to the removal of lattice water molecules. The second stage began at approximately 147 °C and continued to 276 °C for $CuL_1{\cdot}5H_2O$, whereas $ZnL_1{\cdot}4H_2O$ decomposed within the range of 165–247 °C. DTA peaks around 250 and 231 °C, respectively, were associated with the removal of coordinated water molecules. The final decomposition stage extended from 367–640 °C for $CuL_1{\cdot}5H_2O$ and 355–602 °C for $ZnL_1{\cdot}4H_2O$, with DTA peaks at approximately 427 and 466 °C. This stage corresponded to the degradation of the organic ligand, ultimately yielding anhydrous metal oxides.

The RuL_4H_2O and $RuL_1{\cdot}5H_2O$ complexes also exhibited characteristic thermal decomposition patterns. The initial weight losses were 4.07% and 8.23%, respectively, occurring between 60–98 °C and 60–110 °C. These values were close to the calculated losses of 4.15% and 8.11% and were attributed to the removal of lattice water molecules. Endothermic DTA peaks appeared near 102 and 106 °C, supporting this assignment. The second decomposition stage occurred between 153–266 °C for RuL_4H_2O and 180–276 °C for $RuL_1{\cdot}5H_2O$, with DTA peaks at approximately 197 and 188 °C, respectively. This stage was mainly associated with the loss of coordinated water molecules. Further heating produced additional mass loss due to decomposition of the remaining complex, accompanied by both endothermic and exothermic thermal events.

Table 4. Thermogravimetric data of Schiff base metal complexes.

Compound	Temperature range (C)	Loss in weight found (calc.) (%)	DSC peak (ºC)	Assignments
Co SF1	50–136	9.2 (9.17)	149	H2O
	136–285	8.89 (8.86)	181	Ligand
	287–397	81.68 (81.43)	381	CoO
Ni SF1	50–141	6.6 (6.17)	117	H2O
	141–246	16.27 (16.76)	159	Ligand
	246–400	77.07 (77.21)	384	NiO
Cu SF1	50–164	5.5 (5.17)	134	H2O
	164–260	17 (17.76)	250	Ligand
	260–400	77 (77.13)	391	CuO
ZnSF1	100–190	7.5 (7.12)	139	H2O
	180–290	18 (18.61)	262	Ligand
	290–450	79 (79.21)	398	ZnO
Co SF2	160–286	14.02(14.47),	195	3H2O
	365–610	65.23(65.44),	511	Ligand
	>610	20.71(20.09)	617	CoO
Ni SF2	176–280	14.13(14.48),	178	3H2O
	368–630	65.13(65.49)	540	Ligand
	>630	20.68(20.02)	610	NiO
Cu SF2	188–293	14.51(14.29,	175	3H2O
	360–622	65.32(65.65)	549	Ligand
	>622	21.69(21.05)	623	CuO
Zn SF2	185–282,	14.38(14.22),	167	3H2O
	365–616,	64.02(64.33),	362	Ligand
	>616	21.86(21.43)	534	ZnO

4.5 X-ray Diffraction (XRD) Studies

The XRD patterns of the synthesized Co(II), Ni(II), and Cu(II) complexes are presented in Figures 6(a–c). All three complexes showed sharp and well-defined diffraction peaks, confirming their crystalline nature. The diffraction patterns of the metal complexes were clearly different from that of the free Schiff base ligand, indicating changes in the structural arrangement following metal coordination.

The crystalline behavior of the metal–Schiff base complexes can be attributed to the ordered arrangement of the metal and ligand components within the complexes. The average crystallite size was estimated from the full width at half maximum (FWHM) of the characteristic diffraction peaks using the Scherrer equation. The calculated average crystallite sizes were approximately 31, 40, and 67 nm for the Co(II), Ni(II), and Cu(II) complexes, respectively. These values indicate that the synthesized metal–Schiff base complexes possess a nanocrystalline structure.

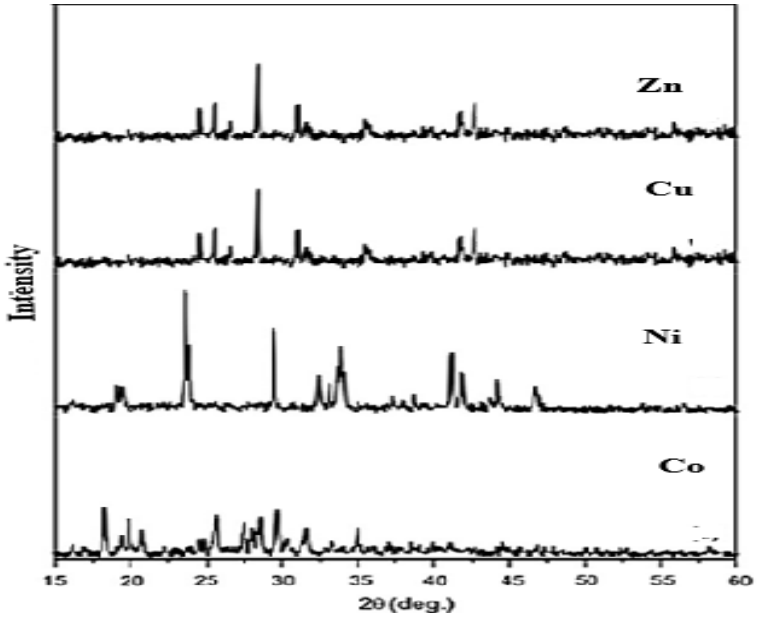

Figure 3. XRD pattern of (a) Co(II), (b) Ni(II) and (c) Cu(II) Schiff base complexes SF1

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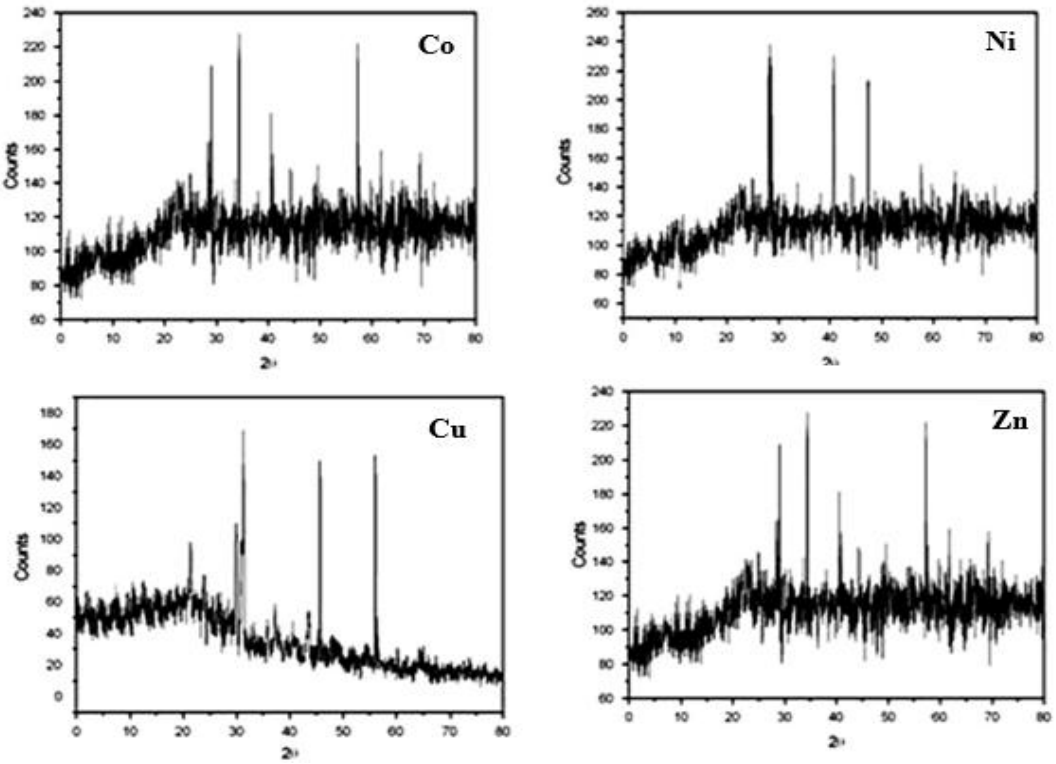

Figure 4. XRD pattern of (a) Co(II), (b) Ni(II) and (c) Cu(II) Schiff base complexes SF2.

Scanning Electron Microscopy (SEM) Studies

The surface morphology of the synthesized Co(II), Ni(II), Cu(II), and Zn(II) complexes of SF1 was examined using scanning electron microscopy. The SEM images revealed noticeable differences in particle morphology among the complexes. The Co(II) and Ni(II) complexes mainly showed platelet-like structures, whereas the Cu(II) complex displayed a characteristic cauliflower-like morphology. The particle dimensions of the Co(II), Ni(II), and Cu(II)–indal-glygly complexes were generally within the micrometer range. However, some particles smaller than 100 nm were also observed. These smaller particles tended to aggregate and form larger clusters. The SEM observations, together with the crystallite-size results obtained from XRD, indicate that the complexes possess polycrystalline structures consisting of nanosized grains with varying degrees of agglomeration.

Powder XRD patterns of the Schiff base metal complexes were recorded over a 2θ range of 0–80°. The $[CoL(H_2O)_3]$, $[CoL_1(H_2O)_3]$, and $[CuL_1(H_2O)]_2H_2O$ complexes exhibited strong and well-defined diffraction peaks, confirming their crystalline nature. In contrast, the remaining complexes showed relatively broad or poorly defined diffraction patterns, suggesting a predominantly amorphous structure. The average crystallite sizes were calculated using the Scherrer equation. The $[CoL(H_2O)_3]$, $[CoL_1(H_2O)_3]$, and $[CuL_1(H_2O)]_2H_2O$ complexes showed average crystallite sizes of approximately 48, 61, and 42 nm, respectively, confirming their nanocrystalline character.

SEM analysis of the $[CoL_1(H_2O)_3]$, $[CoL(H_2O)_3]$, $[CuL_1(H_2O)]_2H_2O$, and $[RuL_1(H_2O)]_2H_2O$ complexes, presented in Fig. 8a–d, further demonstrated their nanoscale morphology. The $[CuL_1(H_2O)]_2H_2O$ complex exhibited a well-developed surface with a distinctive nanorod-like morphology. The grains in the $[CoL(H_2O)_3]$ complex were comparatively well defined and showed a relatively uniform distribution. The grain-size observations from SEM were consistent with the XRD findings, confirming the presence of polycrystalline materials containing nanosized grains.

The SEM images of $[CoL_1(H_2O)_3]$ and $[RuL_1(H_2O)]_2H_2O$ revealed irregularly shaped agglomerates with submicron dimensions. Other complexes showed rough and irregular surfaces resembling fractured particles, along with smaller grains distributed within the micrometer range. Overall, the SEM and XRD results demonstrate that the synthesized Schiff base metal complexes possess diverse surface morphologies, crystallinity, and nanoscale structural features depending on the metal ion and coordination environment.

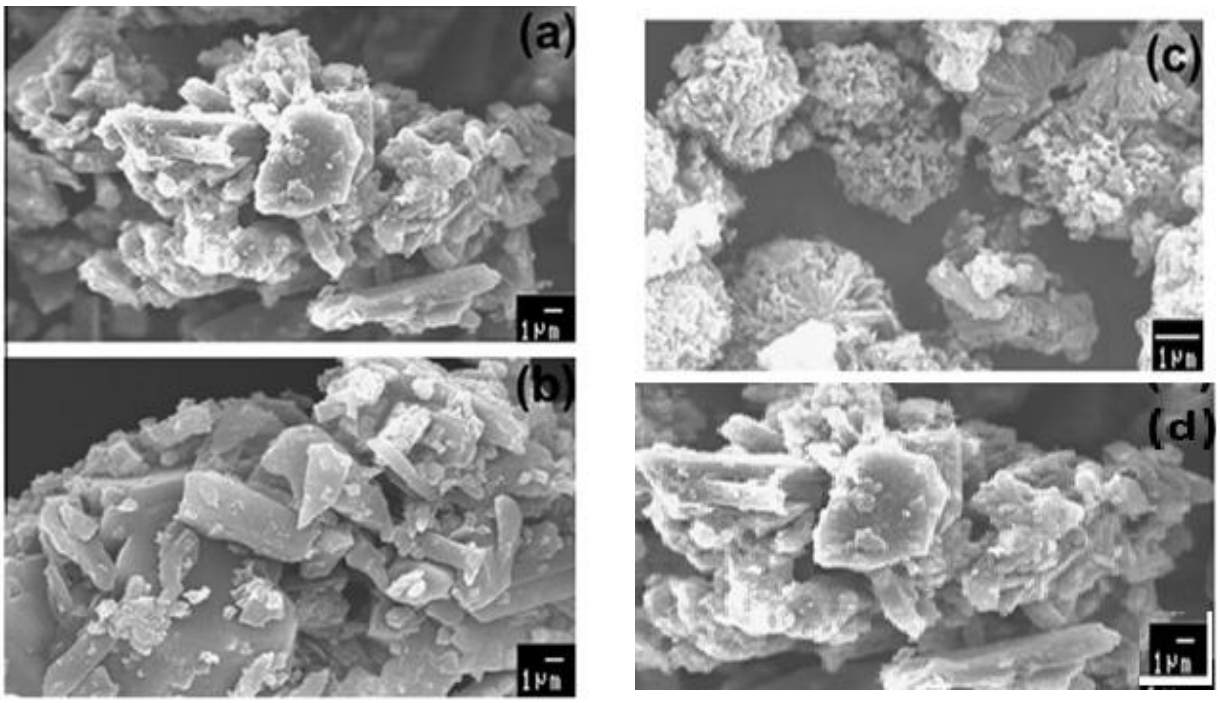

Figure 5. SEM images of (a) Co(II), (b) Ni(II) and (c) Cu(II) (d) Zn Schiff base complexes of SF1

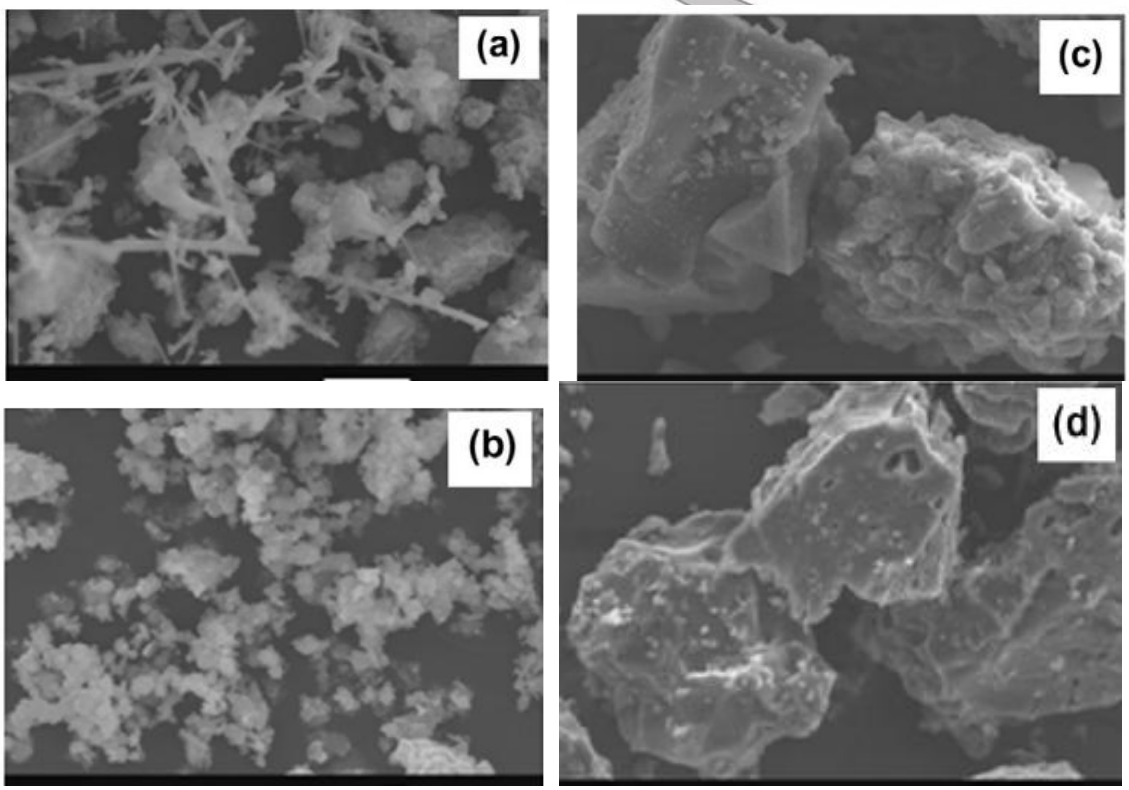

Figure 6. SEM images of (a) Co(II), (b) Ni(II) and (c) Cu(II) (d) Zn Schiff base complexes SF1

5. DISCUSSION

The current investigation effectively produced indole-derived glycylglycine Schiff base metal complexes, with data validating their successful creation using several physicochemical and spectroscopic methods. FTIR examination revealed the vanishing of the aldehyde carbonyl peak and the emergence of the azomethine (–CH=N–) band, therefore validating the synthesis of the Schiff base ligand. Alterations in the FTIR peaks subsequent to metal complexation, coupled with the emergence of M–N and M–O bands, suggested that the ligand successfully coordinated with the metal ions.

The UV–Visible spectra provided further evidence for complex formation by indicating alterations in electronic transitions after metal binding. Investigations into magnetic susceptibility confirmed the anticipated structures of the metal complexes, however molar conductance assessments revealed that the majority of complexes functioned as non-electrolytes. Thermal examination indicated that the metal complexes exhibited more stability than the uncoordinated ligand, indicating that chelation enhanced the compounds' stability. Elemental analysis further validated the suggested composition and purity of the produced complexes.

The improved stability of the metal complexes may be attributed to chelation, which enhances electron delocalization and fortifies metal–ligand interactions. Indole derivatives possess antimicrobial, antioxidant, anti-inflammatory, and anticancer attributes, and their biological efficacy may be enhanced through complexation with metal ions. The characterisation findings validated the effective synthesis of stable indole-derived glycylglycine Schiff base metal complexes and indicate that these substances have significant promise for forthcoming pharmacological and medicinal chemistry research.

6. CONCLUSION

This research successfully synthesized two Schiff base ligands derived from glycylglycine, which were used to create Co(II), Ni(II), Cu(II), and Zn(II) metal complexes. Elemental and spectroscopic evaluations validated the effective synthesis of the ligands and their interaction dynamics with transition metal ions. Infrared spectral analyses demonstrated that the ligands

function as tridentate chelators, coordinating via azomethine nitrogen, peptide nitrogen, and carboxylate oxygen atoms. Molar conductance assessments indicated the electrolytic characteristics of the complexes, whilst ESR and magnetic analyses corroborated a square planar configuration for the Cu(II) complex.

Thermal analysis revealed the existence of coordinated and lattice water molecules and validated the thermal stability of the complexes, culminating in decomposition to metal oxides. Powder XRD investigations demonstrated the crystalline and nanocrystalline characteristics of the complexes, while SEM study showed a variety of surface morphologies and particle dimensions ranging from nano to micro. The integrated physicochemical, spectroscopic, and thermal findings robustly support the suggested configurations of the produced complexes.

The effective synthesis and comprehensive characterization of these Schiff base metal complexes establish a robust basis for forthcoming studies on their biological and pharmacological uses, especially in the creation of prospective metallodrugs and functional nanomaterials.

- **CONFLICT OF INTEREST**

  The authors confirm they have no conflict of interest.

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