

ORIGINAL RESEARCH ARTICLE

Design, synthesis, spectroscopic characterization, and theoretical studies of Mn(II), Co(II), Ni(II), and Cd(II) Schiff base complexes derived from 4-Hydroxycoumarin, Glycine, and Metformin

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ABSTRACT

In this work, the synthesis, characterisation and preparation of several new Mn(II), Co(II), Ni(II) and Cd(II) complexes with Schiff base ligands prepared from 4-hydroxycoumarin. The ligands and their respective metal complexes were synthesised in 1:1:1 metal to ligand molar ratio and characterised by elemental analysis, Fourier-transform infrared spectroscopy (FT-IR), UV-Visible spectroscopy, molar conductivity measurements, magnetic susceptibility studies and atomic absorption spectroscopy. In addition, theoretical calculations were carried out using GaussView05/Gaussian09 software with the semi-empirical PM6 method to support the experimental findings. FT-IR spectral analysis revealed that the coumarin-based Schiff base ligand acts as a bidentate chelating agent through the azomethine nitrogen (C=N) and the carboxylate oxygen (COO⁻) atoms, resulting in the formation of stable five-membered chelate rings with the metal ions. Shifting of N-H and C=N vibrational bands suggested the involvement of amine and imine nitrogen atoms in the coordination of the metal, and supported the complexation of metformin. The electronic spectra and magnetic moment data were in support of the proposed coordination geometries around the metal centres. Molar conductivity measurements in DMSO indicate that most the complexes behave as electrolytes. The theoretical results were in good agreement with the experimental data, providing deeper insight into the electronic structure and stability of the synthesized complexes. In conclusion the results proved the efficiency of coumarone, and metformin based Schiff base ligands in the field of coordination chemistry and suggested that their divalent metal complexes may be promising in future applications in the field of chemical and materials sciences.

Keywords: schiff base; coumarin; transition metal complexes; spectroscopic characterization; theoretical calculations

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1. Introduction

Schiff bases are compounds containing the azomethine functional group (RC=N) and are usually prepared by condensation of primary amines with carbonyl compounds. These compounds have been the subject of continuous attention in coordination and medicinal chemistry since their first discovery by the German chemist Hugo Schiff in 1864^[1,2]. These compounds have been a subject of constant interest in coordination and medicinal chemistry. Schiff bases are considered as excellent chelating ligands due to their synthetic versatility, ease of preparation and the electron donating ability of the imine group (C=N) which can strongly coordinate with transition metal ions^[3-5]. In recent years Schiff base ligands have increasingly gained importance due to their broad applications. They

have very important role in the design of metal complexes with catalytic, optical, magnetic and biological properties^[6-9]. Schiff bases with donor atoms such as nitrogen, oxygen and sulphur are of particular interest because of their important biological functions such as antibacterial, anticancer, anti-inflammatory and antioxidant activity^[10-12]. Moreover, their structural flexibility allows the design of multidentate ligands able to form stable complexes with a wide variety of transition metals, which are valuable in catalysis, drug design and material sciences^[13-15]. The rapid growth of computational chemistry has made theoretical studies very important for the understanding of electronic and structural properties of Schiff base complexes. GaussView05 in combination with Gaussian09, software is used present powerful tools for modelling molecular structures, predicting reactivity and comparing theoretical results with experimental findings. These computational methods for the study of electronic transitions, molecular orbitals and thermodynamic parameters of Schiff base metal complexes are very useful^[16]. Therefore the present effort is directed to the synthesis and characterisation of new transition metal complexes of Schiff bases obtained from modified glycine and to study their characteristics experimentally and theoretically. Such studies not only provide insight into the coordination behavior of Schiff bases but also pave the way for their application in modern chemistry, medicine, and materials science.

2. Materials and Method

All materials used in this investigation were obtained from sigma / Aldrich and utilised as received. They include; 4- hydroxyl coumerin, Glycine and Other amines, metals transition [Mn(II), Co(II), Ni(II), Cd(II)] Chloride, Ethanol (C₂H₅OH), DMSO and NaOH %10. Elemental analysis was performed using EA – Elemental Analyser, electronic spectra were obtained using a Varian UV-visible spectrophotometer, molar conductivity measurements were conducted using a WTW56 apparatus, FTIR spectra were recorded using a Shimadzu spectrophotometer in the Chemistry Department at the College of Science. Atomic absorption was performed using a Schimadzu model 6809. Magnetic susceptibility measurements were carried out using the Faraday method with a balance magnetic susceptibility model MSB – MKI in the Chemistry Department at the College of Science, Al-Nahrain University. A theoretical study were also conducted using the program ((GaussView05 / Gaussian09w way (Semi- empirical / PM6) to measure the spectra of infrared spectra of UV-Visible to ligand and complexes.

3. Synthesis of 4-hydroxy-2-oxocoumarin -3-carbaldehyde

(Vilsmeier–Haack Formylation Method)**

The Vilsmeier reagent was generated by adding phosphorus oxychloride (POCl₃) (6.0 mL, 0.065 mol) dropwise with continuous stirring for 15 min to a mixture of N, N-dimethylformamide (DMF) (10 mL, excess) that was chilled to 0–5 °C in an ice bath. The reaction mixture was kept at this temperature for a further 20 min. Then, with vigorous stirring, 4-hydroxycoumarin (2.00 g, 0.012 mol) was added slowly to the cold reaction mixture. After complete addition, the mixture was slowly brought to room temperature, then heated under reflux at 70–80 °C for 6–8 h, to achieve double formylation at the activated aromatic positions (3). After completion of the reaction the reaction mixture was cooled to room temperature and carefully poured onto crushed ice (100 g) with stirring. The resulting mixture was neutralized slowly using a cold aqueous solution of sodium acetate (10%) until pH ≈ 6 – 7. A yellow precipitate formed immediately. The solid product was filtered, washed thoroughly with cold distilled water to remove residual inorganic salts, and dried under vacuum. The crude product was recrystallized from hot ethanol to afford 4-hydroxy-2-oxocoumarin -3-carbaldehyde as a yellow crystalline solid, Yield: 65–75%. The synthetic route used for the preparation, of the 4-hydroxy-2-oxocoumarin -3-carbaldehyde is illustrated in **Figure 1**.

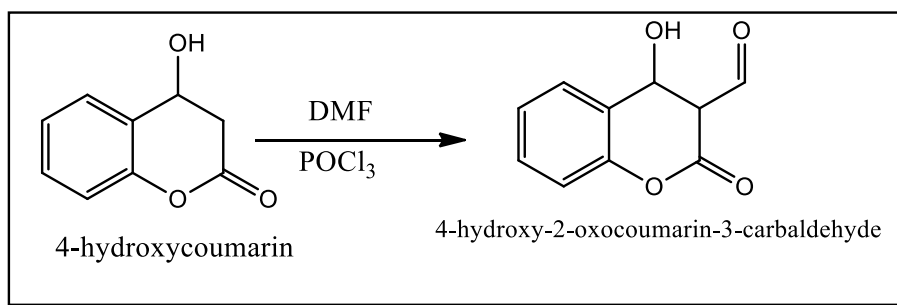


Figure 1. Synthesis of the 4-hydroxy-2-oxocoumarin -3-carbaldehyde.

3.1. Synthesis of the ligand(L1)

Dissolve 0.01 mol of 4-hydroxy-3-formylcoumarin in 30 ml of ethanol. Add 0.01 mol glycine and stir the mixture. Add 2-3 drops of glacial acetic acid (AcOH). Heat the mixture under reflux for two to four hours. The Schiff base ligand is obtained as a yellow precipitate. Wash the filtered product with ethanol (cold). Recrystallize the product from hot ethyl alcohol and vacuum dry, The synthetic route used for the preparation the Schiff base ligand is illustrated in **Figure 2**.

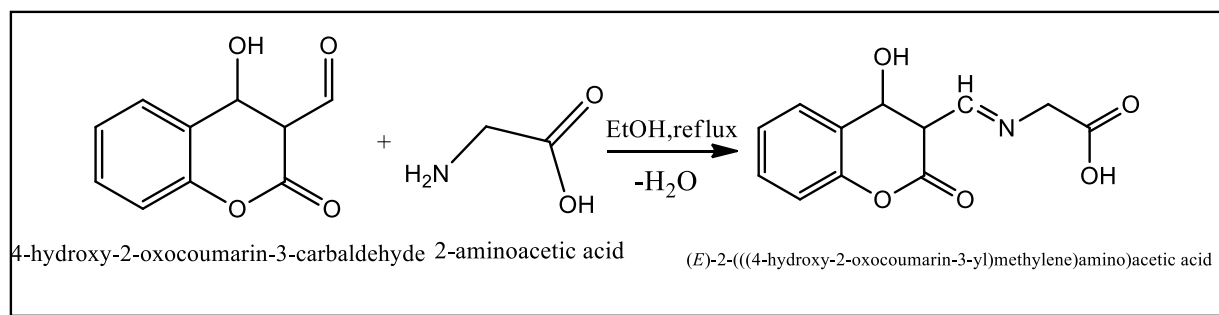


Figure 2. Synthesis of the ligand(L₁).

3.2. Synthesis of Complexes

A hot ethanolic solution (10 mL, 78 °C) of the metal(II) salt [MnCl₂·4H₂O (1 mmol, 0.197 g), CoCl₂·6H₂O (1 mmol, 0.249 g), NiCl₂·6H₂O (1 mmol, 0.237 g) or CdCl₂·2H₂O (1 mmol, 0.231 g)] was added dropwise to a solution of the Schiff base ligand(L₁) (2 mmol, 0.516 g), and (2 mmol) of Metformin ligand(L₂) in ethanol (10 mL). The reactions were performed in a 1:1:1 molar ratio of metal(II) to ligand. The pH of the solution was adjusted to ~7 by 10% aqueous ammonia. The mixture was then refluxed on a water bath for 2-3 h. The reaction was followed to completion, then the mixture was cooled to room temperature and the solid product obtained was filtered off and washed extensively with distilled water, then with hot ethanol and dried under vacuum over CaCl₂. The synthetic route used for the preparation of the mixed ligand and its metal complexes is illustrated in **Figure 3**.

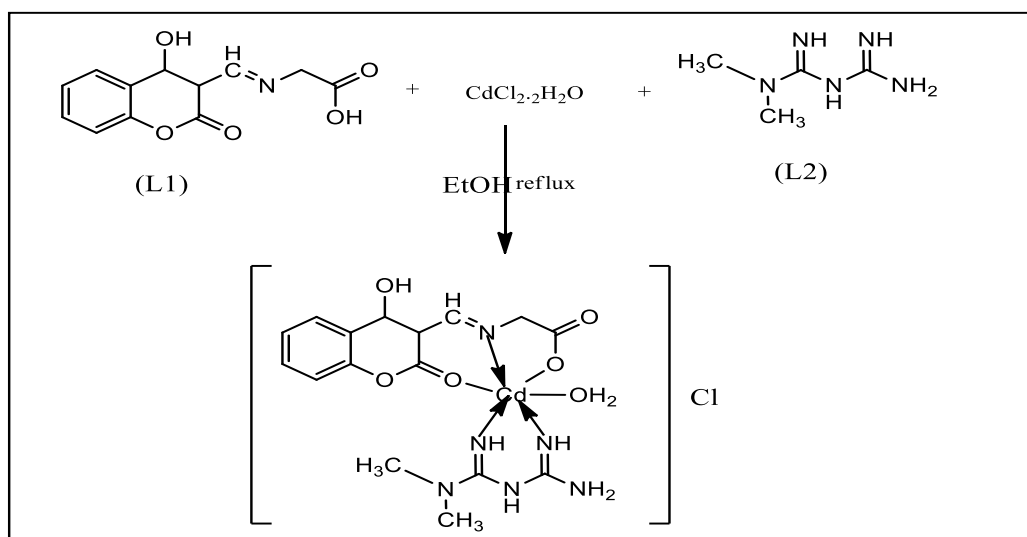


Figure 3. Synthesis of the Complexes.

4. Discussions and Results

4.1. Characterisation of ligands and their metal complexes:

The ligands and its metal complexes were insoluble in water but soluble in organic solvents including DMSO, DMF and Ethanol. Conductivity values ($32\text{--}39.9\text{ cm}^2\text{ohm}^{-1}\text{ mol}^{-1}$) in DMSO revealed that the complexes are electrolytes^[17,18]. The metal percentage in complexes analytical and some of physical data of ligands and complexes are given in **Table 1**.

Table 1. Analytical and some physical data of ligands and their complexes.

Molecular formula	Color	M.wt* <i>C</i>	M.P °C	Conductivity $\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}$ In DMSO	%Metal calc.(found)
(L1) $\text{C}_{12}\text{H}_{11}\text{NO}_5$	Yellow	274	68-70	-----	-----
(L2) $\text{C}_4\text{H}_{11}\text{N}_5$	White	129	221	-----	-----
$[\text{Mn}(\text{L}_1)(\text{L}_2)(\text{H}_2\text{O})_2]\text{Cl}$	Pale yellow	547	286	32	10.05(9.87)
$[\text{Co}(\text{L}_1)(\text{L}_2)(\text{H}_2\text{O})_2]\text{Cl}$	Redish brown	551	265	39.2	10.7(10.99)
$[\text{Ni}(\text{L}_1)(\text{L}_2)(\text{H}_2\text{O})_2]\text{Cl}$	Light green	551	290*	29	10.7(9.889)
$[\text{Cd}(\text{L}_1)(\text{L}_2)(\text{H}_2\text{O})]\text{Cl}$	White	604	>300*	39.9	18.54(17.89)

4.2. Infra-red spectra

The FTIR spectrum of the synthesized compound shows a characteristic absorption band at $\sim 1630\text{ cm}^{-1}$, attributed to the azomethine ($\text{C}=\text{N}$) stretching vibration, confirming the formation of the Schiff base^[19,20]. The strong absorption bands observed at ~ 1730 and $\sim 1685\text{ cm}^{-1}$ correspond to the lactone carbonyl group of the coumarin moiety and the carboxylic acid carbonyl group, respectively^[21–23]. The ligand acts as a bidentate chelating agent through the azomethine nitrogen and the carboxylate oxygen atoms, forming stable five-membered chelate rings with metal ions. The FT-IR spectrum of the metformin ligand displayed characteristic vibrational bands associated with its functional groups. The asymmetric and symmetric N–H stretching vibrations of the primary amine groups were observed as sharp absorption bands in the region around 3366 and $3289\text{--}3290\text{ cm}^{-1}$ ^[24], respectively, consistent with previous spectroscopic studies on metformin and its derivatives. These bands are generally attributed to the $-\text{NH}_2$ and $-\text{NH}$ vibrational modes of the biguanide structure of metformin^[25]. In addition, the $\text{C}=\text{N}$ stretching vibration, characteristic of the imine functionality present in the biguanide moiety, was detected in the region of $1583\text{--}1626\text{ cm}^{-1}$, in

accordance with the reported data for metformin and its complexes^[26]. The mid-infrared region also exhibited C–N stretching vibrations of aliphatic amine groups, consistent with literature assignments for metformin. The coordination to metal ions caused significant variations in the frequency and intensity of the N–H and C=N bands as compared to free ligand. The observed shifts indicate the involvement of the nitrogen atoms of the amine and imine groups in the coordination to the metal centres, which leads to perturbations in the bond strength and the electron density around these functional groups. The changes of these spectra are accepted as diagnostic evidences for metal-nitrogen interaction in coordination compounds^[27]. The FT-IR bands (cm⁻¹) of the ligands and their metal complexes data of the synthesized ligands and their metal complexes are summarized in **Table 2**, As shown in **Figures 4** and **5**. The expected structure is shown in **Figure 6**.

Table 2. FT-IR bands (cm⁻¹) of the ligands and their metal complexes.

Functional Group	Free Ligand (cm ⁻¹)	Mn(II) Complex (cm ⁻¹)	Co(II) Complex (cm ⁻¹)	Ni(II) Complex (cm ⁻¹)	Cd(II) Complex (cm ⁻¹)	Assignment
v(O–H) phenolic	3400–3450	— / shifted	— / shifted	— / shifted	— / shifted	Hydrogen bonding / coordination
v(N–H)	3300–3150	3250–3180	3260–3190	3270–3200	3280–3210	Amine stretching
v(C=N) azomethine	1620–1640	1590–1610	1595–1615	1600–1620	1590–1605	Coordination through N
v(C=O) lactone	1700–1725	1660–1685	1665–1690	1670–1695	1660–1680	Coordination through O
v(M–N)	—	520–560	515–550	525–560	510–545	Metal–nitrogen bond
v(M–O)	—	430–480	435–485	440–490	425–470	Metal–oxygen bond

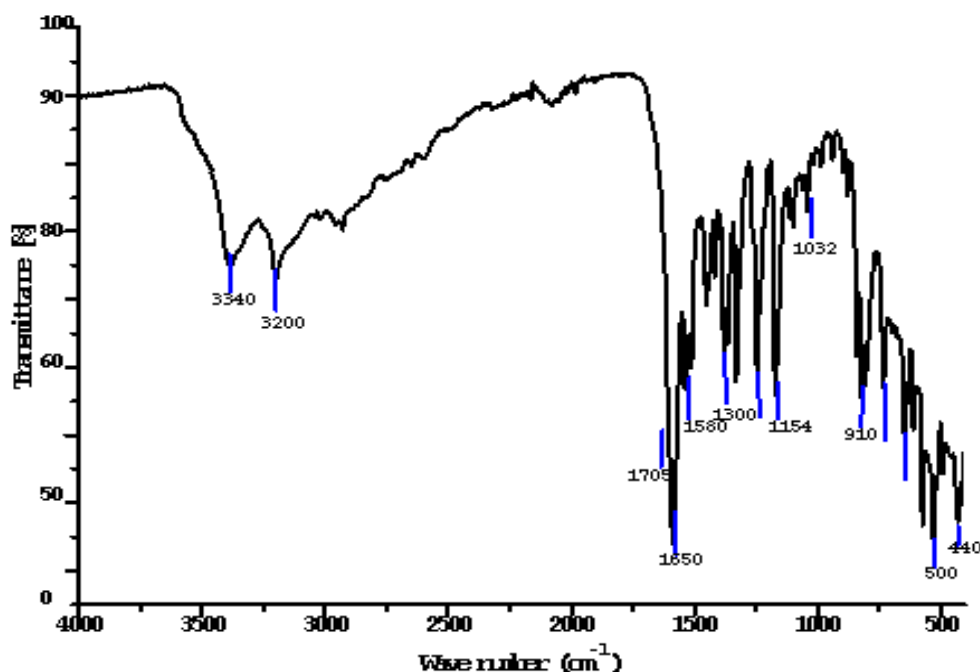


Figure 4. FTIR- spectrum of the ligand(L₁).

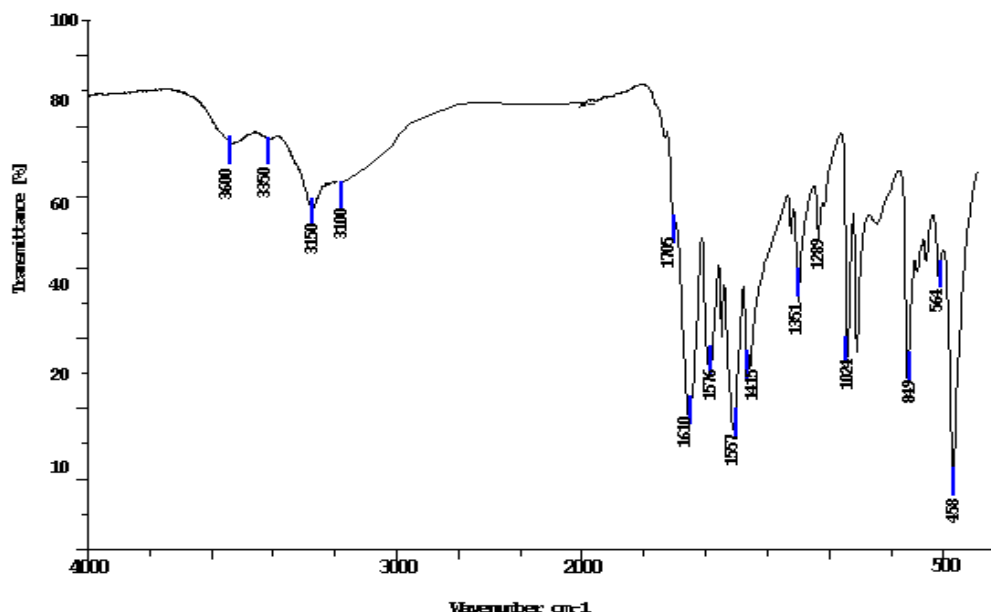


Figure 5. FTIR- spectrum of $[\text{Cd}(\text{L}_1)(\text{L}_2)(\text{H}_2\text{O})]\text{Cl}$.

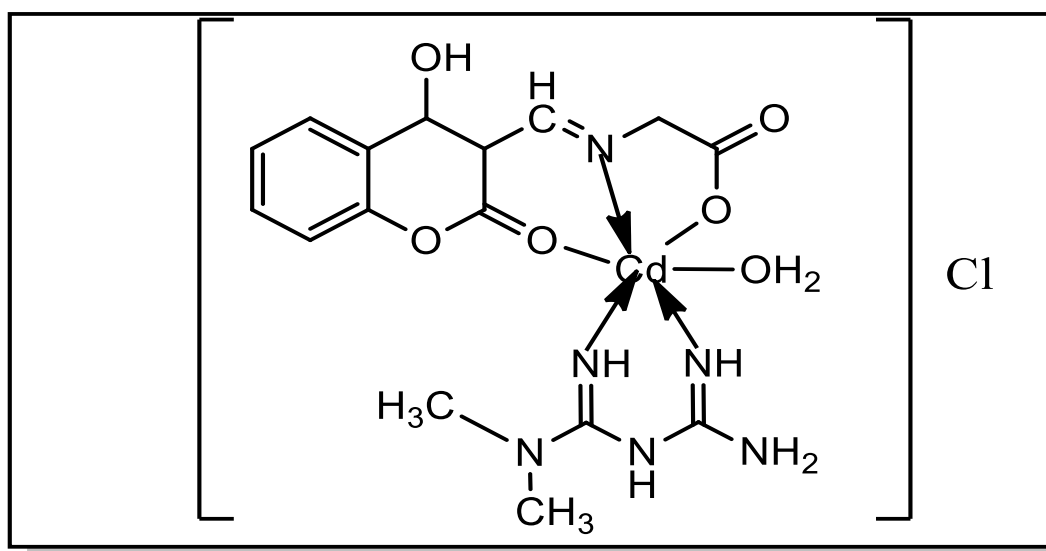


Figure 6. Structure of complex Cd (II).

4.3. Electronic spectra

The electronic absorption spectrum of the free Schiff base ligand is characterised by intense bands in the near UV region which can be assigned to intra-ligand $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions of aromatic rings and azomethine ($\text{C}=\text{N}$) chromophores^[28–30]. These features are characteristic of conjugated Schiff base systems and are attributed to electronic excitation within the ligand framework. The bands usually show bathochromic or hypsochromic shifts on coordination to the metal ions due to redistribution of electron density and changes in conjugation, suggesting effective metal-ligand interaction and chelation through donor atoms^[29–31]. Mn(II) complexes have a high-spin d^5 electronic configuration, which gives rise to weak, spin-forbidden $d-d$ transitions that are generally of low intensity and often obscured by stronger charge-transfer bands. Consequently, the UV–Vis spectra of Mn(II) complexes are dominated by ligand-to-metal charge transfer (LMCT) transitions, where ligand orbitals donate electron density to partially filled metal orbitals^[32–34]. For octahedral Co(II) complexes, several spin-allowed $d-d$ transitions are typically observed,

including ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ and ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$, in agreement with Tanabe–Sugano predictions for d^7 systems^[35–37]. These bands are usually located in the visible and near-infrared regions whereas LMCT transitions are observed at higher energies in the UV region^[38,40]. Magnetic susceptibility data^[30,41] also supports octahedral geometries of these complexes. The characteristic transitions for nickel(II) complexes in octahedral environments (d^8 configuration) are usually attributed to ${}^3A_{2g} \rightarrow {}^3T_{2g}(F)$, ${}^3A_{2g} \rightarrow {}^3T_{1g}(F)$ and ${}^3A_{2g} \rightarrow {}^3T_{1g}(P)$. These transitions can be explained using Tanabe–Sugano diagrams and are observed in the visible region^[42]. In addition, LMCT transitions may contribute to the higher-energy absorption bands^[43]. In the case of Cd(II) complexes, which possess a d^{10} electronic configuration, d–d transitions are not expected due to complete filling of the d orbitals. Therefore, their electronic spectra are dominated by intra-ligand $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions, as well as charge-transfer bands such as LMCT, which occur at higher energies in the UV region and reflect strong ligand contributions^[42]. Overall, the observed UV–Vis spectral features of the synthesized complexes, including ligand band shifts, emergence of charge-transfer transitions, and diagnostic d–d bands where applicable, provide strong evidence for the proposed coordination modes and geometries. These spectral characteristics are consistent with the expected behavior of octahedral Mn(II), Co(II), and Ni(II) complexes and the absence of d–d transitions in d^{10} Cd(II) complexes^[42]. The electronic spectral parameters obtained from the UV–Visible measurements are listed in **Table 3** and **Figure 7, 8**.

Table 3. Electronic Spectral Data (UV–Vis) of Ligands and Its Metal Complexes.

Molecular formula	Absorbing bands ν (cm^{-1})	Type of transition
(L ₁)C ₁₂ H ₁₁ NO ₅	37037 , 30769 278 strong , 332 Medium	$\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$
(L ₂) C ₄ H ₁₁ N ₅	25773, 40816	$\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$
[Mn(L ₁)(L ₂)(H ₂ O) ₂]Cl	20833 ,27778 , 34483 510 weak, 345 Medium , 286 strong	${}^4A_{1g} \rightarrow {}^4T_{1g}(G)$, LMCT
[Co(L ₁)(L ₂)(H ₂ O) ₂]Cl	19231, 27027, 33898 540 weak, 360 Medium, 292 strong	${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$, ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$, LMCT
[Ni(L ₁)(L ₂)(H ₂ O) ₂]Cl	16393 ,25974, 33333 560 weak,370 Medium , 295 strong	${}^3A_{1g} \rightarrow {}^3T_{1g}(P)$, ${}^3A_{2g} \rightarrow {}^3T_{2g}(F)$, LMCT
[Cd (L ₁)(L ₂)(H ₂ O)]Cl	29851, 36364 350 weak, 288 strong	$\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$

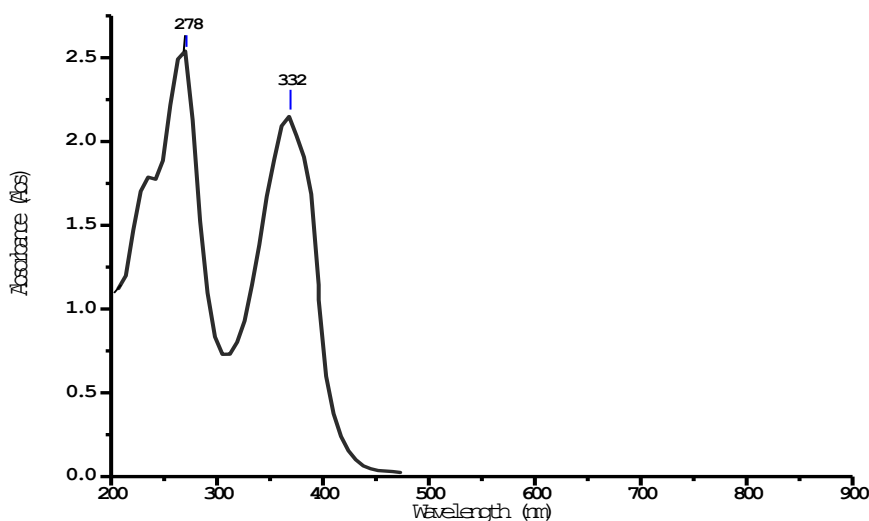


Figure 7. UV – Visb spectrum of Ligand (L₁).

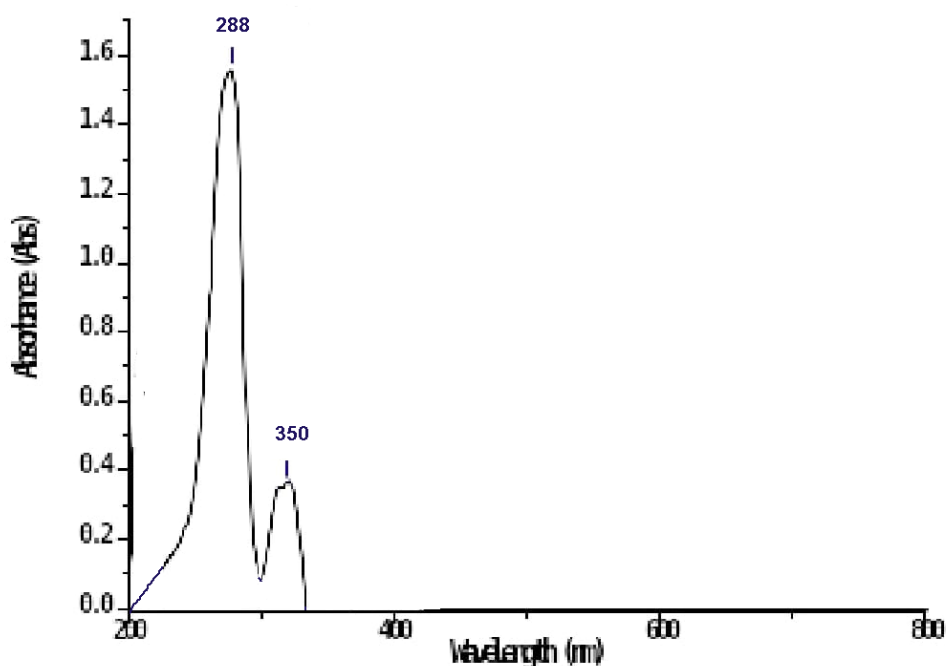


Figure 8. UV- Visb spectrum of $[\text{Cd}(\text{L}_1)(\text{L}_2)(\text{H}_2\text{O})_2]\text{Cl}$.

The molar conductivity values ($32.1\text{--}39.9\text{ }\Omega^{-1}\text{cm}^2\text{ mol}^{-1}$) suggest that all the complexes are 1:1 electrolytes showing that the chloride ions are beyond the coordination sphere^[43–45]. The magnetic moment of the Mn(II) complex is 2.05 B.M. which is in agreement with a high spin d5 configuration and is indicative of an octahedral shape^[44–46]. The result of 4.83 B.M. for the Co(II) complex is typical of high-spin octahedral Co(II) systems with orbital contribution^[48,49]. The magnetic moment of the Ni(II) complex is 2.38 B.M. which is in agreement with two unpaired electrons expected for octahedral d8 complexes^[46]. In contrast, the Cd(II) complex is diamagnetic ($\mu_{\text{eff}} = 0$ B.M.) in accordance with its closed-shell d10 structure^[46]. In general, the conductivity and magnetic measurements are in very good agreement with the hypothesised octahedral geometries and ionic formulations of the complexes^[46]. The magnetic moment and molar conductivity values of the synthesized metal complexes are summarized in **Table 4**.

Table 4. Magnetic Moments and Electrical Conductivity of Complexes.

Molecular formula	Molar conductivity ($\text{ohm}^{-1}\text{ cm}^2\text{ mol}^{-1}$)	μ_{eff} (B.M)	Geometry
$[\text{Mn}(\text{L}_1)(\text{L}_2)(\text{H}_2\text{O})_2]\text{Cl}$	32	2.05	O.h
$[\text{Co}(\text{L}_1)(\text{L}_2)(\text{H}_2\text{O})_2]\text{Cl}$	39.2	4.83	O.h
$[\text{Ni}(\text{L}_1)(\text{L}_2)(\text{H}_2\text{O})_2]\text{Cl}$	29	2.38	O.h
$[\text{Cd}(\text{L}_1)(\text{L}_2)(\text{H}_2\text{O})]\text{Cl}$	39.9	0	O.h

5. Biological Activity

The synthesised ligands and their metal complexes were screened for anti-microbial activity by agar well diffusion method and the results were represented as inhibition zone diameters (mm). The solvent control (DMSO) exhibited no inhibitory effect against any of the tested microorganisms, confirming that the observed biological activity is attributed solely to the ligands and their complexes^[47]. The mixed-ligand metal complexes generally showed higher antimicrobial activity than the free ligands, particularly against *Streptococcus* sp., *Pseudomonas aeruginosa*, *Klebsiella* sp., and *Candida albicans*. In contrast, limited

activity was observed against *E. coli*. Among the compounds tested, the Co(II) mixed ligand complex exhibited the highest and broadest antimicrobial activity indicating the enhancement of biological activity due to complexation. This enhancement can be explained on the basis of the chelation theory which states that the chelation decreases the polarity of the metal ion hence increasing the lipophilic character of the complex which makes it to permeate through the lipid layers of microbial cell membranes and complex with the intracellular molecules^[47]. These findings in general reveal that metal complexation considerably increases the antibacterial activity of Schiff base ligands, confirming their potential as bioactive agents. The antimicrobial activities of the synthesized ligands and their corresponding metal complexes against the tested microorganisms are illustrated in **Figure 9A** and **Figure 9B**.



9A



9B

Figure 9. Antimicrobial activity of the synthesized ligands and their metal complexes against the tested microorganisms using the agar well diffusion method. (A) Antibacterial activity against the tested bacterial strains. (B) Antifungal activity against *Candida albicans*.

6. Conclusion

A new coumarin based ligand and their M(II)-metformin mixed ligand complexes have been synthesised and characterised successfully by different analytical and spectroscopic techniques. The results obtained were in agreement with the suggested structures and coordination modes. The synthesised compounds showed promising biological activity and conductivity behaviour. The experimental results were supported by theoretical calculations and provided interesting information about the electronic properties and stability of the prepared compounds. The results obtained suggest potential applications of the synthesised compounds, biology and material science.

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Conflict of interest

The authors declare that is no conflict of interest regarding the publication of this manuscript.

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