

Synthesis, Structure, and Biological Activity Studies of New Metal Ion Complexes Based on 3-[(3-Hydroxynaphthalene-2-yl-ethylidene)-hydrazono]-1,3-dihydro-indol-2-one

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Abstract: In the present study, a novel ligand (L) made of 2-hydroxynaphthaldehyde and 3-hydrazone-1,3-dihydro-indole-2-one(3-[(3-hydroxynaphthalen-2-yl-ethylidene)-hydrazono]-1,3-dihydro-indol-2-one). The ligand was characterized by FTIR, UV-vis, mass, ¹H-NMR, ¹³C-NMR, and CHN elemental analysis. New complexes of this ligand were created by treating methanol and a drop of DMF solution of the produced ligand with the hydrated metal salts of Mn(II), Co(II), Ni(II), Cu(II), and Zn(II) in a molar ratio of 2:1 (L:M). As a result, complexes have been emerged and identified FTIR, UV-vis, C.H.N., chloride-containing, molar conductance, magnetic susceptibility, and atomic absorption. The characterization result for each complex indicated complexes with octahedral coordination geometry and tridentates with metal to ligand ratios of 1:2. The biological activities of the new compounds were examined against Gram-negative bacteria (*Escherichia coli*) and Gram-positive bacteria (*Staphylococcus aureus*) giving an acceptable inhibition efficiency.

Keywords: biological activities; isatin; metal complexes; Schiff base; 2-hydroxynaphthaldehyde

■ INTRODUCTION

In coordination chemistry, Schiff bases are widely used because of their high coordination ability to form complexes with a wide variety of metal ions, including those of transition metals [1]. Hydrazine compounds show the advantage that they react with aldehydes and ketones to give pure derivatives [2]. Hydrazine derivatives are often employed in various biological activities. Many hydrazones have been employed as antimicrobial medications, which are commonly used to treat a variety of biological functions [3]. The enormous biological importance of Schiff bases-benzohydrazide derivatives has urged us to synthesize Schiff bases derivatives from benzohydrazide with functionalization of amino group with heterocyclic compounds with anti-inflammatory selective inhibitor, anti-fungal, antibacterial, and anti-inflammatory activities [4-6]. The 2-aminobenzohydrazide has been excessively used as a starting material in the synthesis of some bioactive heterocyclic compounds. Furthermore, the presence of

imine group is critical for understanding how transformation and racemization events occur in biological systems [7-8]. Isatin and 2-hydroxynaphthaldehyde are significant polydentate ligand moieties that are used in coordination chemistry and have many applications in many different fields [9]. The existence of an imine group is also required for understanding transformation and racemization processes in biological systems [10]. A wide range of hydrazone applications and its derivatives, including their antibacterial properties, makes them particularly useful [11]. There are also antitubercular, carbonic anhydrous inhibitors, and anti-inflammatory activities [12]. Since it was discovered that some of these complexes may have potential medical applications, transition-metal complexes of these compounds have been extensively researched. A recent discovery in the subject of bioinorganic chemistry has heightened the attention to macrocyclic complexes combining oxygen and nitrogen atoms [13]. A new Schiff base is

synthesized in this study, and it is used as a ligand to offer sites that may act as donors and form complexes with Mn(II), Co(II), Ni(II), Cu(II), and Zn(II). The ligand and its complexes have been completely characterized.

■ EXPERIMENTAL SECTION

Materials

All chemicals used in this investigation were from well-known international companies. 1*H*-indole-2,3-dione (97%), N₂H₂·H₂O (99%), 2-hydroxynaphthaldehyde (99%), MnCl₂·2H₂O (99%), NiCl₂·6H₂O (99%), CuCl₂·2H₂O (99%), CoCl₂·6H₂O (99%), and ZnCl₂ (98%) were given by BDH and utilized immediately received.

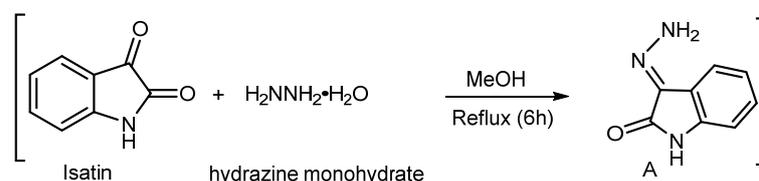
Instrumentation

Infrared spectra of ligands and their complexes were recorded in the range of 4,000–200 cm⁻¹ using a device of Shimadzu FTIR spectrometer with KBr disk for bonds and CsI for complexes. Magnetic sensitivity was measured using John Mathey instrument. Mass100P Shimadzu contribution also determines LC/MS revenue. ¹H and ¹³C-NMR were performed using a Bruker 400 MHz meter while the C.H.N microanalysis was performed using Perkin Elmer automatical instruments model 240B. Minerals were determined using a Shimadzu 680G AA spectrometer.

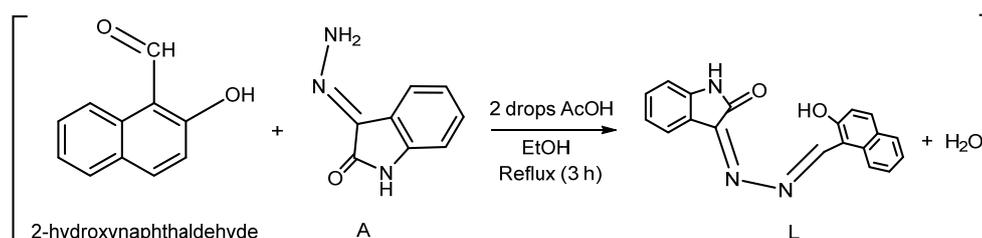
Procedure

Synthesis Schiff base of ligand (L)

Preparation of compound A [14]. In 10 mL of ethanol, isatin (1 g, 0.0067 mol) was dissolved and stirred.



Scheme 1. Preparation of compound A



Scheme 2. The prepared Schiff bases ligand

Aqueous hydrazine (10 g, 0.124 mol) was added dropwise with continuous stirring, followed by refluxing the reaction for 6 h and allowing it to cool to 25 °C. The yellow precipitate was washed with methanol after the generated residue was filtered and dried at 50 °C. The endpoint of the reaction was discovered by TLC (Scheme 1). The compound was characterized by melting point and FTIR.

Preparation of 3-[(3-hydroxynaphthalen-2-yl)ethylidene]hydrazono-1,3-dihydroindol-2-one Schiff base. An amount of 1 g (0.00675 mol) of 2-hydroxynaphthaldehyde was dissolved in 10 mL of ethanol (absolutely 99.9%). Then, 0.93 g (0.0058 mol) of A in 10 mL of methanol was added and followed by 2 drops of glacial acetic acid with continuous stirring until the components were homogenous. The mixture was refluxed for 6 h at 100 °C. The precipitation of light red crystals was completed, and the crystals were filtrated, washed with absolute ethanol, dried for 24 h, and recrystallized with hot absolute ethanol. The recrystallized powder was collected by filtration and then dried. The yield was 75%, and the melting point was 281–283 °C (Scheme 2). The ligand was characterized by FTIR, UV-vis, mass, ¹H-NMR, ¹³C-NMR, and CHN elemental analysis.

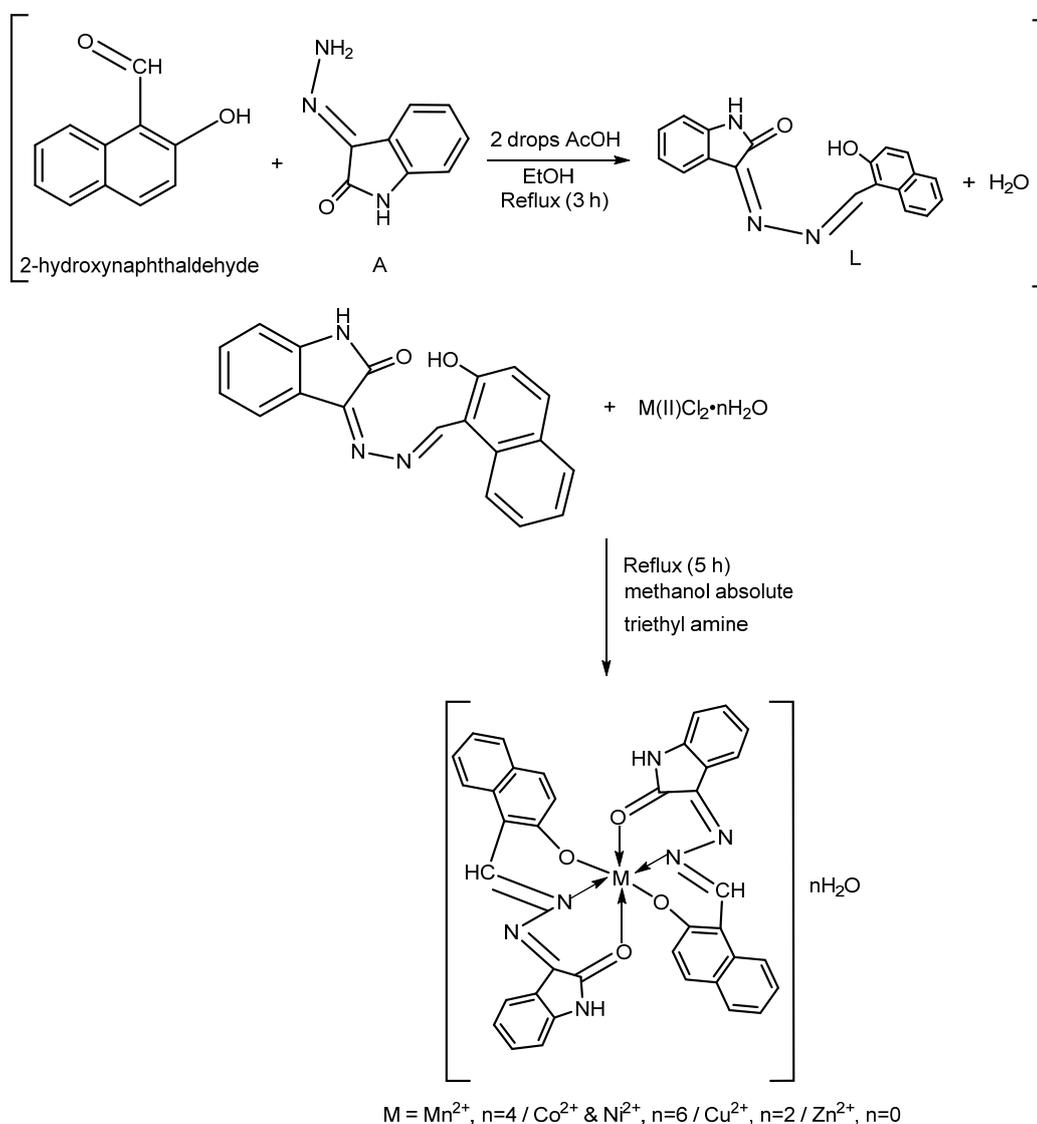
Preparing metal complexes with Schiff bases ligand [15]. As much as 15 mL methanol was added in a 25 mL flask containing 0.635 mg (0.2 mol) of L. One drop of DMF was added as well with warming and slight stirring to complete dissolution. This is followed by adding

0.1 mol salt (0.0627 g of Mn(II) or 0.0754 g of Co(II) or 0.0753 g of Ni(II) or 0.0540 g of Cu(II), or 0.0432 g of Zn(II)), and it was dissolved in 5 mL methanol and one drop of triethylamine. The mixture was refluxed for 5 h until a colored precipitate was formed and the mixture was allowed to cool to 25 °C. The precipitate was filtered and washed with cold water. After that, it was dried with ether. The product was filtered and dried at 50 °C (Scheme 3).

Biological activity

The bioactivity of ligand and complexes has been studied against *S. aureus* and *E. coli* bacteria. The well-spread agar method was used to examine the *in vitro*

antibacterial activity against both strains of bacteria. Gentamicin has been used as an antibiotic. Test and antibiotic samples were prepared in a solution (1 mg/mL) of DMF. Sterilized agar and liquefied were inoculated with a microorganism suspension (1/100 mL from the middle) and poured into a Petri dish to give a depth of about 3 mm. Test and antibiotic samples were placed in the wells. The wells were set up in a hardened medium, and the resulting plates were cooled for 1 h at 5 °C and then incubated at 37 °C for 18 h. Areas of inhibition of bacterial growth resulting from test and antibiotic samples were measured in millimeters.



Scheme 3. Preparation of metal complexes

RESULTS AND DISCUSSION

The tridentate ligand was synthesized in high yield. Table 1 summarizes the physical properties and micro elemental analyses of the generated ligand, as well as its metal complexes. The results and the proposed structural formula are well connected. The match between the calculated and observed findings of the elemental analysis demonstrated the successful formation of ligand and complexes.

Metal Analysis

Complexes were analyzed for metal content. The complexes were digested in conc. HNO₃ and standard solution were obtained by using deionized water. The results were in a good agreement with the proposed formula (Table 1), in accordance, the prepared complexes.

Mass Spectrum of Ligand

In coordination chemistry, mass spectroscopy is increasingly being employed as a powerful structural characterization method [16]. The LC-MS spectrum of Schiff base (Fig. 1) exhibited a molecular ion peak at $m/z = 315.98$, which corresponds to its molecular weight, computed (315.33 g/mol) values.

¹H-NMR Spectrum of Ligand

The chemical surroundings of organic molecules may be determined *via* NMR spectroscopy. Using tetramethylsilane (TMS) as the internal reference standard, the ¹H-NMR of the ligand L in dimethyl sulfoxide (DMSO-*d*₆), which in turn gave a signal δ at 2.32 ppm, as shown in Fig. S1 and Table 2. The ¹H-NMR spectra revealed all of the required peaks to confirm

Table 1. The physical characteristics of produced compounds and elemental microanalysis of C, H, N, and M

Compound	M.W.	Color	M.P (°C)	Analysis (calculated)			
				%C	%H	%N	%M
L (C ₁₉ H ₁₃ N ₃ O ₂)	315.33	Light red	226–228	71.54 (72.37)	4.35 (4.16)	12.67 (13.33)	-----
[MnL ₂].3H ₂ O	737.63	Pale red	>300	62.43 (61.88)	3.78 (4.10)	10.72 (11.39)	7.98 (7.45)
[CoL ₂].2H ₂ O	723.61	Dark red	>300	63.65 (63.08)	4.12 (3.90)	10.86 (11.61)	7.72 (8.14)
[NiL ₂].2H ₂ O	723.37	Green orange	Dec.	62.15 (63.10)	4.14 (3.90)	10.94 (11.62)	8.75 (8.11)
[CuL ₂].3H ₂ O	746.24	Dark red	Dec.	61.87 (61.16)	3.81 (4.05)	10.59 (11.26)	7.88 (8.52)
[ZnL ₂].H ₂ O	712.04	Pale red	>300	63.66 (64.10)	3.39 (3.68)	12.15 (11.80)	8.79 (9.18)

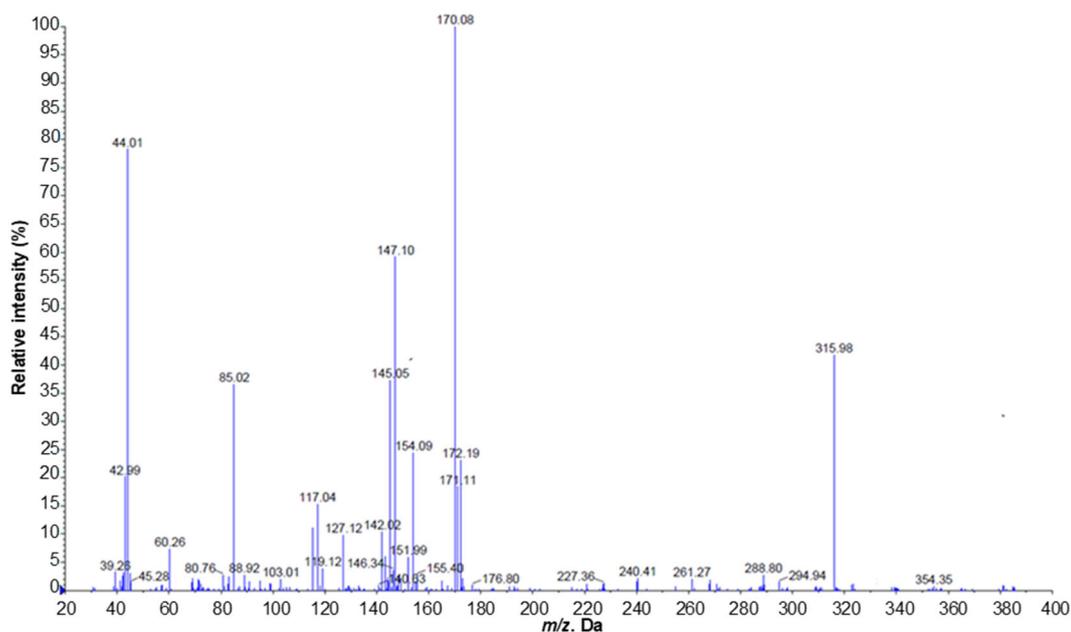


Fig 1. LC-MS spectra of Schiff base ligand

Table 2. ¹H-NMR spectra for L and the chemical shift in ppm

Compound	¹ H-NMR	δ ppm
L (C ₁₉ H ₁₃ N ₃ O ₂)	Singlet (1H) OH	13.900
	Singlet (1H) NH	11.070
	Singlet (1H) HC=N	9.800
	Singlet (4H) indole ring	7.678–8.480
	Multiple (6H) naphthalene ring	6.933–7.631
	Solvent+H ₂ O	2.523–3.365

chemical structure of the synthesized ligand, including the following signals: singlet signal at δ 13.90 ppm belongs to Ar–OH [17]. The singlet signal at δ 11.07 ppm belongs to the N–H group [18], doublet signal at δ 9.8 ppm belongs to azomethine proton N=C–H, and multiple signals at δ 8.480–6.93 ppm belong to ten aromatic protons.

¹³C-NMR Spectrum of Ligand

Using ¹³C-NMR spectroscopy, the chemical composition of the produced ligand was identified. Hence, as shown in Fig. S2, the ¹³C-NMR spectrum exhibited the necessary peaks at the appropriate habitats to illustrate the chemical structure of the created ligand. The ¹³C-NMR was studied using DMSO-*d*₆ as the solvent. The spectrum of the ligand showed δ 108–128 ppm belong to C₁–C₁₀ of the aromatic ring, C=O at 144.7 ppm, C₁₁–C₁₈ at 129–162.9 ppm, and C₁₉ (C=N) at 165.76 ppm [14].

FTIR Spectra of Schiff Base Ligand and Its Complexes

The FTIR spectrum (Fig. S3) of newly obtained ligand displayed a discernible absorption band at 1607 cm⁻¹, which contributed to azomethine formation. Additionally, the lack of an asymmetrical NH₂ group absorption band can be significant evidence for L production. This can be a strong indicator that the amino group in A and the carbonyl group of the 2-hydroxynaphthaldehyde combine to produce the ligand.

It should be noted that the lack of the C=O absorption band of 2-hydroxynaphthaldehyde might potentially assist ligand synthesis *via* this group. Other absorption bands were found at 1712, 3201, and 3417 cm⁻¹, which correspond to the stretching vibrational modes of the following functional groups: C=O of amide, N–H amine, and O–H hydroxyl, respectively [19]. When the infrared spectra of the complexes were compared to the spectrum of the ligand, a considerable change was discovered as bands vanished or moved to other frequencies, and new bands were developed that were not present in the L ligand spectrum. The modifications are summarized below: the coordination of the metal ions to the nitrogen azomethine causes a shift-high in the frequency of C=N value [20]. The new bands indicated conversion of the C=O group in ligand from 1712 cm⁻¹ into new bands. The frequency of the hydroxyl group in 3417 cm⁻¹ for the L that demonstrates how the metal ions coordinated to L through oxygen as it faded or shrunk and transformed into larger bands, which is an indicator of the oxygen group connection with metal ion bands. Meanwhile, new ranges appeared in the 430–578 cm⁻¹ range (Table 2). These bands apply to the formation of M–O and M–N bonds to confirm the development of complexes [20]. All FTIR spectra are shown in Fig. S4–S8, and metal complexes data are listed in Table 3.

Table 3. Infrared spectrum of the prepared ligand and metal complexes

Compound	H ₂ O	O–H	C=O	C=N	M–O	M–N
L (C ₁₄ H ₁₀ N ₄ O)	----	3417	1712	1607		
[MnL ₂].3H ₂ O	3444		1719	1623	578	457
[CoL ₂].2H ₂ O	3484		1723	1625	576	459
[NiL ₂].2H ₂ O	3446		1724	1620	587	447
[CuL ₂].3H ₂ O	3447	----	1719	1617	559	459
[ZnL ₂].H ₂ O	3421	----	1679	1614	501	430

UV-vis Spectroscopy Measurements: Ligand and Its Complexes

Figs. S9-S14 show the UV-vis spectra in the range of 200–1100 nm for the prepared ligand and its complexes after they were dissolved in DMF. The ligand's electronic spectra displayed significant absorption at 305, 344, and 437 nm that are related to $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$, and charge transfer (CT), respectively [21]. Changes in the placement of absorption bands in the complex spectra were detected, indicating metal ion coordination *via* the azomethine and oxygen functional groups [19-20]. The Co(II) complex spectrum showed four bands at 385, 527, 705, and 931 nm, which represented the permitted transitions and had a magnetic moment of 4.79 MB. This corresponds to the magnetic moment of the hexagonal complexes found inside the octahedron (C.T, ${}^4T_{1g(F)} \rightarrow {}^4A_{2g(F)}$, ${}^4T_{1g(F)} \rightarrow {}^4T_{2g(F)}$ and ${}^4T_{1g(F)} \rightarrow {}^4T_{2g(F)}$). The Ni(II) complex's electronic spectrum had four peaks at 445, 478, 627, and 935 nm, which represent four transitions (C.T, ${}^3A_{2g(F)} \rightarrow {}^3T_{1g(P)}$, ${}^3A_{2g(F)} \rightarrow {}^3T_{2g(F)}$ and ${}^3A_{2g(F)} \rightarrow {}^3T_{2g(F)}$), respectively [22]. The complex magnetic moment value of Ni(II) was 3.05 B.M., confirming its high spin octahedral geometry. The electronic spectrum of Cu(II) complex showed two peaks and two bands at 490 and 710 nm, resulting from the combined two transitions of C.T and ${}^2E_g \rightarrow {}^2T_{2g}$. It has a magnetic moment of 1.73 BM, which confirms its high spin octahedral geometry. We do not expect transitions to occur in Zn(II) and Mn(II) complexes since their outer shells are stable during saturation or semi-saturation, respectively, and the observed bands are mentioned in Table 4.

Molar Conductance

Conductivity of the prepared complexes was recorded as DMF solutions are within the range 4.6–36 $\text{cm}^2 \text{Ohm}^{-1} \text{mol}^{-1}$ (Table 4) after allowing the solution to equilibrium at 25 °C, which indicates the nonelectrolyte nature of these complexes [23-24]. The results of the molar conductivity measurements showed they are consistent with the proposed formulas for the complexes because they don't contain an anion outside the sphere of complexes.

Study of Antibacterial Activity

These species were investigated due to their relevance in the field of medicine, and the drilling method (agar well diffusion) was employed to examine the antibacterial activity of chemical compounds. Given that the experiment was conducted in aerobic conditions at 37 °C, the results indicated that the synthesized ligand and its components were physiologically effective. Drilling was employed to expose each agar active compound's agar bacteria to two distinct types of agar bacteria (one type Gram-negative and one type Gram-positive), which were used with DMF solvent at concentrations of 110–3 M and displayed varying efficacy to the negative and positive stain-bacteria of the compounds [25-26]. The data are shown in Table 5.

It came to light that the findings of ligand efficacy and its complexes were very effective against bacteria compared with antibiotic gentamicin [27-29], but only complex $[\text{MnL}_2] \cdot 3\text{H}_2\text{O}$ is ineffective against gram-positive bacteria (*S. aureus*).

Table 4. UV-vis measurements of ligand, magnetic moment, and molar conductivity of metal complexes

Compound	Conductivity DMF ($\text{cm}^2 \text{ohm}^{-1} \text{mol}^{-1}$)	M.p. (°C)	Magnetic moment (B.M)	Color	Abs. bonds (nm) (Assigned transition)
L ($\text{C}_{14}\text{H}_{10}\text{N}_4\text{O}$)	---	281–283	----	Light red	305 ($\pi \rightarrow \pi^*$), 344 ($n \rightarrow \pi^*$), 437 (CT)
$[\text{MnL}_2] \cdot 3\text{H}_2\text{O}$	7.6	>300	5.43	Pale red	365 ($n \rightarrow \pi^*$), 488 (CT)
$[\text{CoL}_2] \cdot 2\text{H}_2\text{O}$	36.0	>300	4.79	Dark red	385 (CT), 527 (${}^4T_{1g(F)} \rightarrow {}^4T_{1g(P)}$), 705 (${}^4T_{1g(F)} \rightarrow {}^4A_{2g(F)}$), 931 (${}^4T_{1g(F)} \rightarrow {}^4T_{2g(F)}$)
$[\text{NiL}_2] \cdot 2\text{H}_2\text{O}$	4.6	Dec.	3.05	Green orange	445 (CT), 478 (${}^3A_{2g(F)} \rightarrow {}^3T_{1g(P)}$), 627 (${}^3A_{2g(F)} \rightarrow {}^3T_{2g(F)}$), 935 (${}^3A_{2g(F)} \rightarrow {}^3T_{2g(F)}$)
$[\text{CuL}_2] \cdot 3\text{H}_2\text{O}$	15.0	Dec.	1.73	Dark red	490 (CT), 710 (${}^2E_g \rightarrow {}^2T_{2g}$)
$[\text{ZnL}_2] \cdot \text{H}_2\text{O}$	14.1	>300	Dia	Pale red	518 (CT)

Table 5. The antibacterial activity of produced Schiff bases, ligands, and complexes

Sample	<i>S. aureus</i> (mm)	<i>E. coli</i> (mm)
DMF	–	–
L (C ₁₄ H ₁₀ N ₄ O)	20	15
[MnL ₂].3H ₂ O	8	12
[CoL ₂].2H ₂ O	13	16
[NiL ₂].2H ₂ O	16	15
[CuL ₂].3H ₂ O	24	30
[ZnL ₂].H ₂ O	18	22

■ CONCLUSION

A hexadentate ligand attaches to the central ion *via* the oxygen of hydroxynaphthaldehyde, oxygen of C=O, and azo-methine nitrogen. Analyses of the spectral data from this experiment indicated that all compounds generated had a 2:1 L:M ratio, which is congruent with a mononuclear structure. Spectral and elemental investigations, as well as conductivity and magnetic moment, revealed that all complexes in the DMF solution were non-electrolytes for octahedral structures. The biological activity of each compound against two bacteria. When evaluated, *E. coli* and *S. aureus* all demonstrated effective inhibition while the manganese compound had no inhibitory effects on the bacteria.

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■ SUPPORTING INFORMATION

S1. ¹H-NMR spectrum of Schiff base ligand, S2. ¹³C-NMR spectrum of Schiff base ligand, S3. FTIR spectrum of Schiff base ligand, S4. FTIR spectrum of Mn(II) complex, S5. FTIR spectrum of Co(II) complex, S6. FTIR spectrum of Ni(II) complex, S7. FTIR spectrum of Cu(II) complex, S8. FTIR spectrum of Zn(II) complex, S9. UV-vis spectrum of Schiff base ligand, S10. UV-vis spectrum of Mn(II) complex, S11. UV-vis spectrum of Co(II) complex, S12. UV-vis spectrum of Ni(II) complex, S13. UV-vis spectrum of Cu(II) complex, S14. UV-vis spectrum of Zn(II) complex.

■ CONFLICT OF INTEREST

The authors declare no conflict of interest.

■ AUTHOR CONTRIBUTIONS

Naser Shaalan designed and supervised the project. Safa Sami performed the experiment, wrote, and analyzed. The findings were discussed by both authors, and they both contributed equally to the final version of the paper.

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