

RESEARCH ARTICLE

Synthesis, Characterization, Antioxidant and Antibacterial Studies for New Schiff Base Complexes derived from 4-Bromo-O-toluidine

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ABSTRACT

In this article, new Schiff base ligand LH-prepared Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II), Hg(II), Pd(II), and Pt(II) materials were analyzed using spectroscopy (1 Metal: 2 LH). The ligand was identified using techniques such as FTIR, UV-vis, ¹H-¹³C-NMR, and mass spectra, and their complexes were identified using CHN microanalysis, UV-vis and FTIR spectral studies, atomic absorption, chloride content, molar conductivity measurements, and magnetic susceptibility. According to the measurements, the ligand was bound to the divalent metal ions as a bidentate through oxygen and nitrogen atoms. The complexes that were created had microbicide activity against two different bacterial species and one type of fungus. DPPH techniques were being used to test the ligand and its complexes for antioxidant activity.

Keywords: DPPH methods, Schiff base, Magnetic susceptibility, Microbicide.

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INTRODUCTION

Schiff bases are an important class of ligand that coordinate to metal ions via azomethine nitrogen.^{1,2} They are the by-products of the condensation between amine and carbonyl compounds. A Schiff base is a ligand with a carbon double bond and a nitrogen atom connected to an aryl or alkyl group but not to hydrogen.^{3,4} Through the nitrogen's non-bonding electrons, these served as a binding site for the metal ions. The carbonyl group of the aldehyde gives ald-imines, and the carbonyl group of the ketone gives ketoimines. When it comes to neutralizing free radical species, the antioxidants found in ligands and their complexes typically take the form of hydrogen donors or electron donors to the reactive site. The ability of a wide variety of organic complexes to scavenge free radicals can be evaluated with the help of DPPH free radicals and (ABTS+) tests. As a result of the fact that several organic molecules that function as very good antioxidants have already been reported, it is of the utmost importance to understand the mode of action along with the efficacy of those antioxidants.⁶ Mixed ligand complexes of the Schiff base ligand (Z)-2-((4-bromo-2-methylphenyl) imino) methyl)-4-methylphenol with some metal ions were characterized by various spectral techniques in addition to testing the biological activity of the compounds that were prepared.⁷ In this study, we synthesized novel complexes with Schiff base ligands derived from 4-bromo-O-toluidine and characterized them by means of a wide range of spectral methods.

MATERIALS AND METHODS

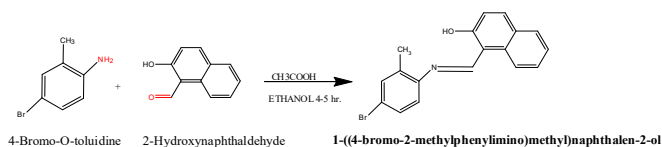
Materials and Physical Measurements

The laboratory's chemicals are of the highest purity and require no further purification; they were purchased from reputable suppliers. Shimadzu UV-160A is utilized for ultraviolet-visible spectra, and an FTIR-8400S spectrophotometer is utilized to validate FTIR spectra on 4000–400 cm⁻¹ in KBr discs. The Stuart Melting Point Kit is used to determine the melting point. All CH Ns of compounds are calculated using Euro (EA 3000). The ¹H-NMR and ¹³C-NMR spectra were obtained at Mashhad University of Medical Sciences in Iran using a Bruker Ultra shield instrument with DMSO as the solvent, operating at 300 MHz for the ¹H-NMR and 75 MHz for the ¹³C-NMR. Iran obtained mass spectra using a GC-MS/MS instrument equipped with an agilent 3200 QTRAP system mass spectroscopy. Atomic absorption method utilising a Shimadzu AA 620G spectrophotometer; Faraday's method measuring magnetic sensitivity utilising a Bruker BM6 instrument.

Preparation of Schiff Base Ligand LH

The Schiff base 1-((4-bromo-2-methylphenylimino)methyl)naphthalen-2-ol was produced by combining an ethanol solution of 4-bromo-O-toluidine (0.186 g, 0.001 mol) with 2-hydroxynaphthaldehyde (0.172 g, 0.001 mol). After being subjected to reflux for 4 to 5 hours, the solution was finally allowed to cool at room temperature, forming a bright

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Scheme 1: The Preparation of the Schiff base ligand (LH)

yellow crystalline solid. In order to obtain the yield of LH (C₁₈H₁₄BrNO), the solid was filtered, washed with hot ethanol, dried at room temperature, and re-crystallized with ethanol. Yield (%): 86. The color is yellow. MP:122°C. Elemental Analysis (%): Calculate: C: 63.49, H: 4.11, O: 4.70, N: 4.11; Found: C: 63.33, H: 4.12, O: 4.53, N: 4.69. M.wt: 340.21. Scheme 1.

Preparation of Complexes

By mixing an ethanol solution of 4-bromo-O-toluidine (0.186 g, 0.001 mol) and 2-hydroxynaphthaldehyde, the Schiff base 1-((4-bromo-2-methylphenylimino)methyl)naphthalen-2-ol was created (0.172 g, 0.001 mol). The resulting solution was subjected to reflux for 4 to 5 hours before being allowed to cool at room temperature. This caused a bright yellow crystalline solid to form. The solid was filtered, washed with hot ethanol, dried at room temperature, and re-crystallized with ethanol to produce the yield of LH (C₁₈H₁₄BrNO). Yield (%): 86. Yellow is the color. M.p:122°C. Analysis of elements (%): Calculated values are C: 63.49, H: 4.11, O: 4.70, and N: 4.11; actual values are C: 63.33, H: 4.12, O: 4.53, and N: 4.69. M.wt: 340.21 (Scheme 1).

Antimicrobial Activity

Using the disc diffusion method, the prepared compounds were evaluated against bacteria such as *Staphylococcus aureus*, *Bacillus Subtilis* as a gram+, *Klebsiella pneumonia*

and *Escherichia coli* as a gram-, and fungi such as *Candida albicans*. The antibacterial and antifungal drugs amoxicillin and metronidazole were used for comparison purposes. This concentration of the solvent DMSO is used to formulate the sample solution. Dishes are incubated at room temperature for 24 hours⁹⁻¹² to determine the amount of bacterial growth. Afterward, the diameter of the inhibition is measured.

Antioxidant Activity

DPPH's (1,1-diphenyl-2-picrylhydrazyl) ability to neutralize free radicals (DPPH). The subject's capacity to recognize DPPH radicals is frequently assessed in studies on antioxidant activity. It is a quick method for ascertaining the radical-scavenging capacity of particular compounds.¹³ The ability of all substances and associations to neutralize free radicals was assessed using varying concentrations of DPPH radicals (25 and 75 ppm). Utilizing a Tecan-PC infinite M200 Pro Plate reader in comparison to a control after an hour of incubation in the dark, absorbance (A) was determined at 517 nm. Then, it was determined how much IC inhibited the DPPH color values. The same experiments were run repeatedly. The following formula¹⁴⁻¹⁷ was used to calculate the antioxidant activity percentage, also known as the DPPH inhibition percentage: Antioxidant percentage is equal to a blank less a sample divided by a blank by 100. (In this case, A stands for the absorbance of the sample, while A stands for the absorbance of the blank).

RESULTS AND DISCUSSION

The results of melting point and the decomposition (dec.) temperature of Schiff base ligand and metal complexes were determined, and the molar absorptivity values of complexes were (6.8–13.2) 1 cm² mol⁻¹ in DMSO solvent, indicating that the complexes did not have an electrolytic nature.¹⁸ Table 1

Table 1: The Physical properties of compounds

Compounds	M.wt g/mole	M.P °C, (dec.)	Color	Elemental analysis % Theoretical (practical)				Cond. Ω ⁻¹
				C	H	N	M	
[Mn(L) ₂ (H ₂ O) ₂]	769.38	185	Pail - brown	56.20	3.93	3.64	7.74	10.2.
				56.13	3.97	3.80	7.69	
[Co(L) ₂ (H ₂ O) ₂]	773.38	195	Brown	55.91	3.91	3.62	7.62	6.8
				55.88	3.89	3.75	7.60	
[Ni(L) ₂ (H ₂ O) ₂]	773.14	(210)	green	55.93	3.91	3.62	7.59	8.9
				55.86	3.87	3.73	7.55	
[Cu(L) ₂ (H ₂ O) ₂]	777.99	(215)	Orange	55.58	3.89	3.60	8.17	9.7
				55.49	3.81	3.69	8.12	
[Zn(L) ₂ (H ₂ O) ₂]	779.83	(220)	Pail- yellow	55.45	3.88	3.59	8.39	11.9
				55.37	3.79	3.62	8.36	
[Cd(L) ₂ (H ₂ O) ₂]	826.85	(190)	yellow	52.29	3.66	3.39	13.60	12.8
				52.19	3.60	3.42	13.55	
[Hg(L) ₂ (H ₂ O) ₂]	915.03	(200)	yellow	47.25	3.30	3.06	21.92	13.2
				47.22	3.28	3.09	21.90	
[Pd(L) ₂]	784.83	(210)	Deep brown	55.09	3.34	3.57	13.56	8.9
				55.07	3.31	3.59	13.54	
[Pt(L) ₂]	873.49	(200)	brown	49.50	3.00	3.21	22.33	12.6
				49.49	2.89	3.25	22.21	

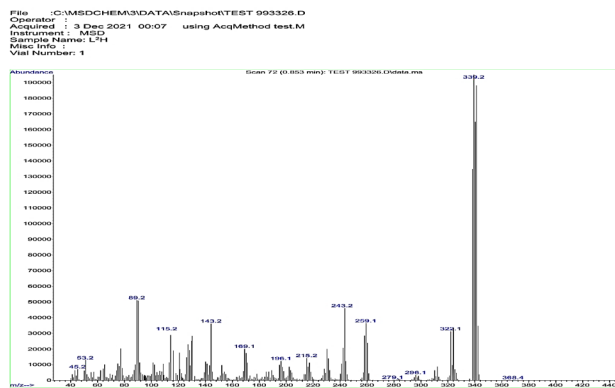
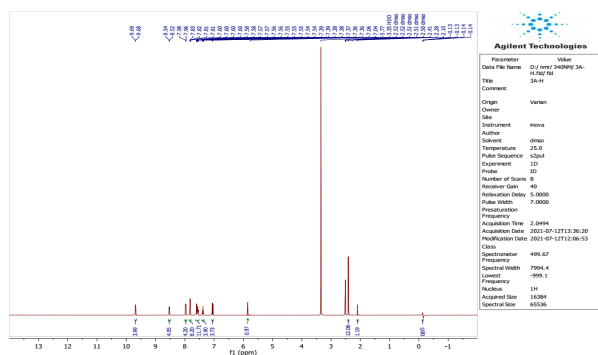


Figure 1: mass spectrum of LH

Figure 2: The ¹H-NMR spectra of LH

presents the results of the microanalysis for CHN and atomic absorption for M%.

Mass, ¹H-NMR and ¹³C-NMR Spectra of the Ligand LH

The results of the initial high-resolution mass spectrometer analysis of the synthesised ligand (LH) are displayed in Figure 1 and Table 2. The observed molecular ion peak for (C₁₈H₁₄BrNO) had a mass-to-charge ratio of m/z = 340.21, which was astonishingly similar to the calculated value. Figure 2 and Table 3 display the ¹H-NMR spectra of the synthesized Schiff base ligand (LH) in d₆-DMSO, respectively.³ The CH₃ group, the solvent DMSO, and the molecule HDO were each responsible for the singlet signal that appeared at 2.41, 2.52, and 3.35 ppm, respectively. These measurements ranged from 2.52 to 3.35 ppm. Multiple signals between 7.04 and 7.98 ppm were attributed to the aromatic protons (9H, m) of phenyl, whereas the singlet signal between 8.54 and 9.69 ppm was attributed to the azomethine proton (HC=N) and OH.¹⁹ As shown in Table 4 and Figure 3, the ¹³C-NMR spectrum of the free ligand LH displayed the following chemical shifts: chemical shift at 11.19 ppm corresponds to the (-CH₃) group; signal at 137.02 ppm corresponds to the (C3); signals at (39.50–40.48) ppm for DMSO; and chemical shifts at range (120.01-130.56) due to aromatic carbon.

Electronic Spectra and Magnetic Susceptibility for Compounds

Three absorption peaks at (412, 429, 470) nm, which correspond to 6A1g(F)4T2g(D), 6A1g(F)4A1g,4Eg(G), and 6A1g(F)

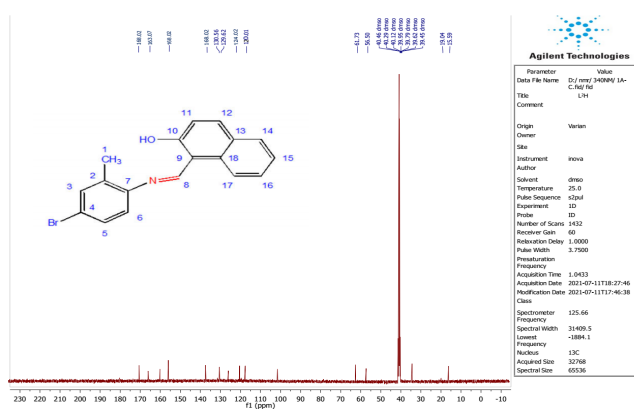
Figure 3: The ¹³C-NMR spectra of LH

Table 2: The Fragmentation Data for the LH

Assignments	Peaks m/z (Abundance %)
[M ⁺] = (C ₁₈ H ₁₄ BrNO)	339.2(100%)
[M ⁺] - CH ₃ = [M ₁ ⁺]	322.1(17 %)
[M ₁ ⁺] - Br + H = [M ₂ ⁺]	243.2(24 %)
[M ₂ ⁺] - (HC=N) - (C ₆ H) = [M ₃ ⁺]	216.2(5 %)
[M ₃ ⁺] - (C ₇ H ₅) = [M ₄ ⁺]	89 (26 %)
[M ₃ ⁺] - (C ₃ H ₂ O) = [M ₅ ⁺]	53(7 %)

Table 3: ¹H-NMR Spectrum Data for Ligand LH

Functional group	δ(ppm)
CH ₃	2.41(3H, m)
DMSO-d ₆	2.50-2.52
HDO	3.35(2H,s)
OH	9.69(H,s)
Ar-H	7.04-7.98 (9H, m)
N=C-H	8.54 (H, s)

Table 4: ¹³C-NMR Spectrum of the Ligand LH

Compound	Functional groups	δ (ppm)
	(CH ₃) group C1	11.19
	C2,C12,C18 for carbon ring	130.56
	C3	137.02
	C4,C11,C17 for Aromatic ring	120.1
	C5,C13,C14 for Aromatic ring	129.62
	C6,C15,C16	124.02
	C7 ring	152.02
	C8 for carbon imine group	155.01
	C8 for (C-OH) group	163.07

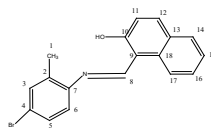


Table 5: Electronic spectra for prepared complexes

Compound	Wave number		ϵ_{max} molar ⁻¹ cm ⁻¹	Assignment	Suggested structure
	nm	cm ⁻¹			
[Mn(L) ₂ (H ₂ O) ₂]	270	37037	1558	Intra ligand	Octahedral
	332	30120	1772	C.T	
	412	24272	417	⁶ A _{1g(F)} → ⁴ T _{2g(D)}	
	429	23310	1791	⁶ A _{1g(F)} → ⁴ A _{1g} , ⁴ E _{g(G)}	
	470	21277	1558	⁶ A _{1g(F)} → ⁴ T _{2g(G)}	
[Co(L) ₂ (H ₂ O) ₂]	270	37037	1367	Intra ligand	Octahedral
	439	22779	385	C.T	
	480	20833	320	⁴ T _{1g(F)} → ⁴ T _{1g(P)}	
	738	13550	27	⁴ T _{1g(F)} → ⁴ A _{2g(F)}	
	802	12468	14	⁴ T _{1g(F)} → ⁴ T _{2g(F)}	
[Ni(L) ₂ (H ₂ O) ₂]	273	36630	1915	Intra ligand	Octahedral
	348	28735	1194	C.T	
	388	25773	1302	³ A _{2g(F)} → ³ T _{1g(P)}	
	799	12516	73	³ A _{2g(F)} → ³ T _{1g(F)}	
	900	11111	78	³ A _{2g(F)} → ³ T _{2g(F)}	
[Cu(L) ₂ (H ₂ O) ₂]	301	33223	2322	Intra ligand	Octahedral
	392	25510	1224	C.T	
	781	12804	31	² E _g → ² T _{2g}	
[Zn(L) ₂ (H ₂ O) ₂]	283	35336	2384	Intra ligand	Octahedral
	347	28818	1500	Intra ligand	
	416	24038	1024	C.T	
[Cd(L) ₂ (H ₂ O) ₂]	272	36765	1858	Intra ligand	Octahedral
	347	28818	750	Intra ligand	
	412	24271	346	Intra ligand	
[Hg(L) ₂ (H ₂ O) ₂]	478	20921	230	C.T	Octahedral
	275	36363	1979	Intra ligand	
	340	29412	720	Intra ligand	
	410	24390	302	C.T	
	458	20619	120	C.T	
[Pd(L) ₂]	272	36765	1904	Intra ligand	Square planner
	315	31746	1292	Intra ligand	
	346	28901	870	C.T	
	412	24272	449	¹ A _{1g} → ¹ B _{1g}	
	769	13004	17	¹ A _{1g} → ¹ A _{2g}	
[Pt(L) ₂]	273	36630	2021	Intra ligand	Square planner
	346	28901	1482	Intra ligand	
	412	24272	899	C.T	
	443	22573	402	¹ A _{1g} → ¹ T _{2g}	
	496	20161	254	¹ A _{1g} → ¹ T _{1g}	

4T_{2g}(G), respectively, were visible in the Mn(II) complex's d-d electronic spectrum.²⁴ The Co(II) complex exhibited three peaks in the d-d transition at (480), 732, and 802 nm, which are the octahedral Co complexes 4T_{1g}(F)4T_{1g}(P), 4T_{1g}(F)4A_{2g}(F), and 4T_{1g}(F)4T_{2g}(P), respectively (F). The

spectrum of nickel complexes reveals two unique peaks at (388, 799, and 900) nm, which correspond to 3A_{2g}(F)3T_{1g}(P), 3A_{2g}(F)3T_{1g}(F), and 3A_{2g}(F)3T_{2g}(F), respectively.²⁵ The electronic spectra of the Cu complex exhibited a single absorption peak at 781 nm, which conforms to the formula

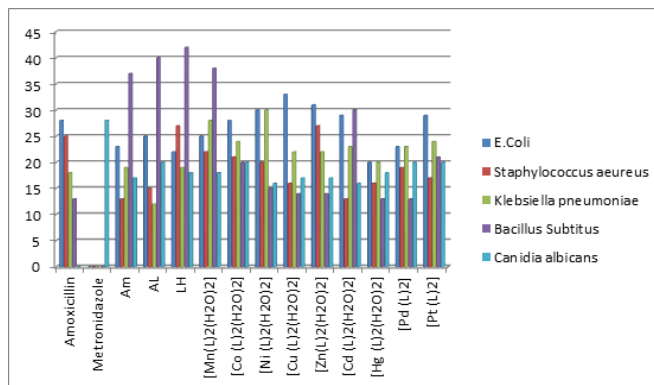
Table 6: FTIR spectra data for the ligand (LH) complexes

Compound	ν (O-H)	ν (C-H) arom.	ν (C-H) alph.	ν (C=N) imine	ν (C=C)	ν (C-O) phenolic	ν (M-N)	ν (M-O)	Other Band (H ₂ O)
LH	3444	3059	2924	1620	1597	1292	-	-	-
[Mn(L) ₂ (H ₂ O) ₂]	-	3089	2954	1651	1631	1257	551	459	3410 763
[Co(L) ₂ (H ₂ O) ₂]	-	3089	2981	1631	1589	1226	551	459	3406 752
[Ni(L) ₂ (H ₂ O) ₂]	-	3082	2978 2931	1628	1604	1253	536	466	3402 763
[Cu(L) ₂ (H ₂ O) ₂]	-	3047	2981 2920 2858	1616	1600 1577	1249	570	462	3444 779
[Zn(L) ₂ (H ₂ O) ₂]	-	3070	2978 2927	1616	1589	1249	524	455	3414 763
[Cd(L) ₂ (H ₂ O) ₂]	-	3074	2978 2873	1600	1573	1249	551	439	3410 763
[Hg(L) ₂ (H ₂ O) ₂]	-	3024	2974 2927	1631	1685	1253	551	459	3406 763
[Pd(L) ₂]	-	3043	2981 2920 2850	1616	1600 1577 1535	1242	551	466	-
[Pt(L) ₂]	-	3082	2931 2897	1651	1627 1585	1249	567	451	-

Table 7: Antimicrobial activity data for the LH and its complexes

Compounds	<i>E. coli</i>	<i>S. aureus</i>	<i>K. pneumoniae</i>	<i>B. subtilis</i>	<i>C. albicans</i>
Amoxicillin	28	25	18	13	
Metronidazole	--	--	--	--	28
Am	23	13	19	37	17
AL	25	15	12	40	20
LH	22	27	19	42	18
[Mn(L) ₂ (H ₂ O) ₂]	25	22	28	38	18
[Co(L) ₂ (H ₂ O) ₂]	28	21	24	20	20
[Ni(L) ₂ (H ₂ O) ₂]	30	20	30	15	16
[Cu(L) ₂ (H ₂ O) ₂]	33	16	22	14	17
[Zn(L) ₂ (H ₂ O) ₂]	31	27	22	14	17
[Cd(L) ₂ (H ₂ O) ₂]	29	13	23	30	16
[Hg(L) ₂ (H ₂ O) ₂]	20	16	20	13	18
[Pd(L) ₂]	23	19	23	13	20
[Pt(L) ₂]	29	17	24	21	20

2Eg2T2g for deformed octahedral Cu complexes.²⁶ In the electronic spectra of Zn, Cd, and Hg complexes, charge transfer peaks at (416), 478, and 458 nm, respectively. For square planner Pd(II) and Pt(II) complexes, respectively, the spectrum of Pd and Pt complexes peaks at 412, 769 nm and 443,496 nm due to (1A₁g1B₁g and 1A₁g1A₂g), (1A₁g1T₂g, and 1A₁g1T₁g), respectively. Mn(II), Co(II), Ni(II), and Cu(II) complexes have measured (eff) effective magnetic susceptibility values of (3.53), 3.35, 2.85, and 1.73 BM, respectively. These values are typical for high-spin octahedral complexes Table 5.²⁴

**Figure 4:** The inhibition zone values for antimicrobial activity

FTIR Spectra for Ligand and Their Complexes

The conclusions that can be drawn from observations of the spectrum of Schiff base Table 6 displayed a band with a frequency of 1620 cm⁻¹ that corresponds to the (HC=N-) stretching vibration. In addition, it exhibited a band with a frequency of 3444 cm⁻¹, which corresponds to the stretching vibration of (O-H).²⁵ Deprotonation occurred when the metal complexes were coordinated to the metal (II) ion, as evidenced by the disappearance of the broad band in the spectra of the metal complexes.²¹ The shifting of the bands of the (C=N) stretching vibration in the range of 1616–1651 cm⁻¹ in the spectra of the metal complexes indicated complexation. The spectra of the metal complexes exhibited the formation of new bands at (524–570) cm⁻¹ and (439–466) cm⁻¹. Both the (M-N) and (M-O) stretching vibrations have been attributed

Table 8: The Inhibition% and IC₅₀ for the LH and its complexes

Compounds	Conc. (µg/ mL)	Inhibition%	IC ₅₀
Vit C.	25	50.7	36.3
	75	74.8	
LH	25	25.9	79.5
	75	43.8	
Mn	25	3.2	---
	75	2.4	
Co	25	1.8	938.1
	75	3.8	
Ni	25	2.0	---
	75	0.0	
Cu	25	1.5	117.1
	75	3.1	
Zn	25	1.8	618.8
	75	6.1	
Cd	25	1.1	114.2
	75	3.3	
Hg	25	13.5	---
	75	9.8	
Pd	25	1.2	597.4
	75	6.6	
Pt	25	4.3	224.4
	75	17.1	

to these recently discovered bands.²⁶ These newly discovered bands also indicate that the Schiff base ligand has somehow coordinated to the metal(II) ion. It is feasible to conclude that (LH) is a neutral bidentate ligand that binds to metal ions through the Schiff base nitrogen and phenolic oxygen. This is possible to accomplish. Experimentation is one possible way to determine the answer to this issue. The spectra of the complexes revealed bands in the range of (752–779) cm⁻¹, and it was found that these bands belonged to (OH). According to,²⁷ which explains the important bands and their assignments for each of the produced complexes, these bands represent coordination between water and metal. Furthermore,²⁷ says that each complex is assigned major bands.

APPLICATIONS

Antimicrobial Activity

In order to determine the efficacy of potential antimicrobial agents, the antibacterial and antifungal activity of Am (4-bromo-O-toluidine), Al (2-Hydroxynaphthaldehyde), Schiff base LH, and its metal(II) complexes was investigated. This was done in order to evaluate potential antimicrobial agents. *Staphylococcus aureus*, *Klebsiella pneumonia*, *Escherichia coli*, and *Bacillus subtilis* were some of the bacteria and fungi that were examined and tested. The findings make it abundantly clear that LH and the complexes it formed possessed a high level of biological activity. This lends credence to the

hypothesis that chelation with a number of biologically active metals results in an increase in the activity of the synthetic compounds against a wide variety of microorganisms Table 7.²⁸

Antioxidant activity

The ability of newly synthesized Schiff bases ligands and their complexes to scavenge free radicals can be evaluated using the DPPH assays method, and the results are displayed in Table 8 and Figure 4, respectively. The DPPH radical is frequently utilized for the purpose of rapidly evaluating antioxidant activity due to the fact that it is both stable and straightforward. The production of stable DPPH decreased the intensity of the DPPH band, which demonstrated the capability of metal complexes and ascorbic acid (a form of vitamin C that was used as a standard) to scavenge free radicals.²⁹ A lower absorbance and a lower IC₅₀ value³⁰ are both indicators that the antioxidant activity has increased. There was found to be no scavenging activity with the complexes of Mn, Ni, or Hg when the IC₅₀ values of the test compounds were observed.

CONCLUSION

In this work, the coordination complexes derived from the Schiff base ligand were prepared, and spectroscopic methods diagnosed them. The results showed that the complexes took the octahedral shape, except for the palladium and platinum complexes, which took the square planar shape.

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