RESEARCH ARTICLE

Preparation, Characterization, Antioxidant and Antibacterial Studies of New Metal (II) Complexes with Schiff Base for 3-amino-1-phenyl-2-pyrazoline-5-one

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ABSTRACT

A new ligand complexes have been synthesis from reaction of metal ions of Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II), Hg(II), Pd(II) and Pt(II) with schiff base LH. 5-[(2-Hydroxy-naphthalen-1-ylmethylene)-amino]-2-phenyl-2,4-dihydro-pyrazol-3-one, this ligand was characterized by Fourier transform infrared (FTIR), UV-vis, ^{1}H, $^{13}CNMR$, and mass spectra. All complexes were characterized by techniques micro analysis C.H.N, UV-vis and FTIR spectral studies, atomic absorption, chloride content, molar conductivity measurements and magnetic susceptibility. The ligand acts as bidentate, coordination through nitrogen atom from azomethin group and deprotonated phenolic oxygen atom. The spectroscopic and analytical measurements showed that the geometric shape of the prepared complexes is octahedral, while the square planar of the palladium and platinum complexes. The ligand and its complexes were having been screened for their antimicrobial activities and antioxidant.

Keywords: Antioxidant study, Conductivity, Transition metal.

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INTRODUCTION

Schiff bases are the condensation products of amine and carbonyl compounds and are important ligand classes that coordinate to metal ions via azomethine nitrogen.[1,2] Schiff base is a ligand containing a carbon double bond in which the nitrogen atom is connected to an aryl or alkyl group but not hydrogen.[3,4] The carbonyl group of the aldehyde gives aldimines while that of Ketone gives ketoimines,[5] and these provide binding site for the metal ions through nonbonding electrons of the nitrogen. Particularly, range of transition metal complexes of azomethine ligands derived from the reaction of 3-amino-1-phenyl-2-pyrazoline-5-one,[6-9] Herein, we report the synthesis schiff base complexes 5-[(2-Hydroxy-naphthalen-1-ylmethylene)-amino]-2-phenyl-2,4-dihydro-pyrazol-3-one and characterization by spectrophotometric methods in addition to estimating the biological activity and studying the antioxidants of the prepared compounds.

MATERIALS AND PHYSICAL MEASUREMENTS

Chemicals used in the laboratory are the highest purity that does not need any further purity and have been purchased from distinguished sources. The device used to measure the melting point is by Stuart Melting Point Kit; the CHN for all compounds is measured by Euro (EA 3000); ultraviolet-visible spectra are performed on a Shimadzu UV-160A; the FTIR spectra are verified via FTIR-8400S Spectrophotometer on 4000–400 cm^{-1} in KBr discs; The ^{1}H, ^{13}C-NMR spectra were performed in Bruker Ultra shield 300MHZ for ^{1}H-NMR and 75MHZ for ^{13}C-NMR respectively, and using Dimethyl sulfoxide (DMSO) as solvent, Mashhad University of Medical Sciences, Iran. Mass spectra were obtained by LC-MS/MS using agilest 3200 QTRAP system mass spectroscopy Mashhad University of Medical Sciences, Iran. Atomic absorption method by means of AA 620G Shimadzu spectrophotometer; magnetic sensitivity was measured using a Faraday's method using Bruker BM6 instrument.

Preparation of Schiff Base Ligand LH

The schiff base was prepared by mixing ethanol solution of 3-amino-1-phenyl-2-pyrazoline-5-one (0.175 g, 0.001 mol) with that of ethanol solution of 2-hydroxynaphthaldehyde (0.172 g, 0.001 mol). The resulting solution was refluxed for 4 to 5 hours, then cooled at room temperature; the bright yellow crystalline solid was formed on cooling. Then the solid was filtered, washed with cold ethanol, dried at room temperature and re-crystallized with ethanol to obtain yield LH ($C_{20}H_{15}N_3O_2$):

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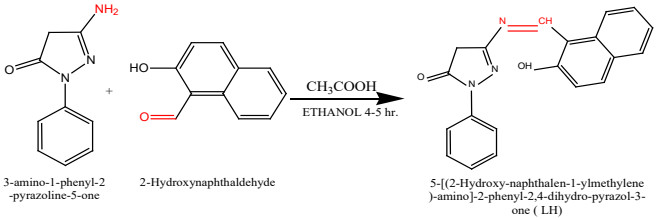

3-amino-1-phenyl-2 -pyrazoline-5-one

2-Hydroxynaphthaldehyde

5-[(2-Hydroxy-naphthalen-1-ylmethylene)-amino]-2-phenyl-2,4-dihydro-pyrazol-3-one (LH)

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

Yield (%): 88. Color: Yellow. M.p:124 °C. Elemental analysis (%): Calcd.: C: 72.94, H:4.59, O: 9.72, N: 12.76; found: C: 69.08, H: 5.30, O: 12.69, N: 12.99. MS (m/z): Calcd. for LH $[M + H]^+$: 329, found 328. Scheme 1.

Preparation of Complexes

A solution of ligand (0.560 g, 2 mmol) and NaOH (2 mmole) in 10 mL absolute ethanol, was added with stirred to a solution consisting of same solvent 10 mL and metal (II) chloride (1 mmol) such as Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II), Hg (II), Pd (II) and Pt (II). The product mixture is stirred for sixty minutes and, then, the result is filtered and dried through anhydrous $CaCl_2$.

Antimicrobial Activity

The prepared compounds were tested against *Staphylococcus aeureus*, *Bacillus subtitus* as a gram$^+$, *Klebsiella pneumonia* and *Escherichia coli* as a gram-, and fungi such as *Canidia albicans* by disc diffusion technique, were used for comparison amoxicillin as an antibacterial and metronidazole as an antifungal. The sample solution is prepared from the concentration of 0.001M in DMSO as a solvent. The dishes are incubated during 24 hours at room temperature then the diameter of the inhibition is measured and this indicates the growth of bacteria.[10-13]

Antioxidant Activity

Radical scavenging of 1,1-Diphenyl-2-picrylhydrazyl (DPPH). The assessment of DPPH radical scanning activity is a standard test in studies of antioxidant activity. It is a rapid technique to assay the radical scavenging activity of specific compounds.[14] All compounds' free radical scavenging effects and associations were evaluated with DPPH radicals at different concentrations 25 and 75 ppm. After 1-hour of incubation in the dark, the absorbance (A) was recorded against a blank at 517 nm on a Tecan-PC infinite M200 Pro Plate reader and IC inhibition of DPPH color values were calculated. Experiments were duplicated. DPPH inhibition percentage (Antioxidant Activity%) was calculated using the following formula:[15,16] Antioxidant% = A blank – A sample/A blank× 100 [Where A blank is the absorbance of the blank and A sample is the absorbance of sample].

RESULTS AND DISCUSSION

The results of melting point and the decomposition (dec.) temperature of schiff base ligand and metal complexes were determined, molar absorptivity values of complexes were (5.7–11.9) Ω^{-1} cm^2 mol^{-1} in DMSO solvent indicating non

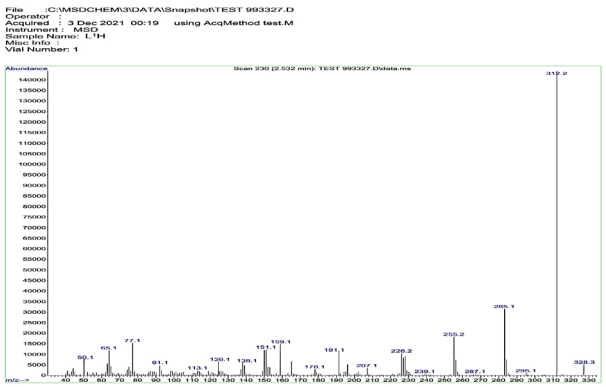

Figure 1: The mass spectrum of ligand (LH).

electrolytic nature of the complexes.[17] Microanalysis CHN and atomic absorption M% were shown in Table 1.

Maas Spectroscopy for (LH) Ligand

The spectrum for (LH) Figure 1, suggested the presence of additional peaks at m/z 328.35 (4%), 312.2 (100%), 285.1 (21%), 255.2 (13%), 191.1 (8%), 126.1 (7%), 77 (11%) and 50 (5%) that assigned to the molecular ion ,$[M- (O)]^+$, $[M- (HCN)]^+$, $[M-C_3N_2]^+$, $[C_6H_5]^+$and $[C_5H_5]^+$, respectively.

^{1}H-NMR and ^{13}C-NMR Spectra of (LH) Ligand

The spectrum of the Schiff base ligand in $DMSO^{-6}$ is shown in Figure 2. The singlet signal in δ 2.50, 3.35 ppm and 2.28 ppm may be attributed to the solvent and $–CH_2$ protons group. The multiple signals ranged between δ (7.04–7.98) ppm were assigned to the aromatic protons, and the singlet signal at δ 9.68 and 10.69 ppm was due to the azomethine and OH protons.[18] The ^{13}C-NMR spectrum Figure 3, shows chemical shift at (34.20 and 155) ppm refers to C2 and C1, the 120.4 ppm may be a signed to (C5,9), (128.70, 126.40, 126.30, 127.7) ppm attributed to C6, C8, C19, C17 and C16. The C10 at 163.7 ppm and 168 ppm at C3.[19]

FTIR Spectra of the Schiff base and Complexes

The values obtained in the spectra of the schiff base showed a band at 1654 cm^{-1} which is assigned to U (HC=N-) stretching vibration and another band at 3209 cm^{-1} which is assigned

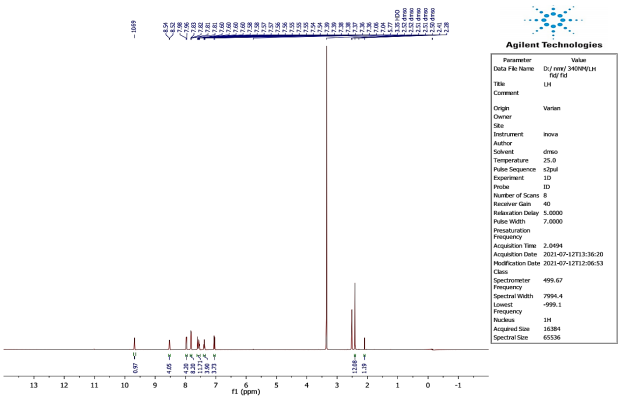

Figure 2: The ^{1}H-NMR of ligand (LH).

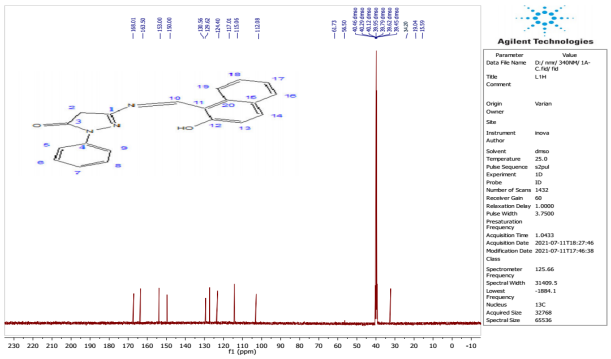

Figure 3: The ^{13}C-NMR of ligand (LH).

U (O-H) stretching vibration Table 2.[20] The broadband disappeared in the spectra of the metal complexes, indicating deprotonating on coordination to the metal (II) ion.[21] The shifting of the bands of U (C=N) stretching vibration in the spectra of the metal complexes in the region (1616–1651) cm^{-1} indicated the complexation has taken place. The new bands appeared in the spectra of the metal complexes at 501–574 and 412–470 cm^{-1} which are assigned to U (M-N) and U (M-O) stretching vibration, respectively,[22] also indicating the coordination of the schiff base ligand to the metal(II) ion. It can be concluded that (LH) binds to the metal ions through Schiff base nitrogen and phenolic oxygen and the LH behaves as neutral bidentate ligand. The spectra of complexes showed the appearance of bands in the range 748–763 cm^{-1} attributed to υ(OH), these bands confirm the coordination of the water with metal.[23] Table 2 describes the important bands and assignment for all prepared complexes.

Magnetic Moment and Electronic Spectra for Complexes

The values of measured magnetic susceptibility and effective magnetic moment (μeff) for the Mn(II), Co(II), Ni(II), Cu(II) complexes are shown in Table 3, exhibit μeff (3.52, 3.37, 2.86, 1.74) B.M, respectively ,which can be a normal values for high spin octahedral complexes.[24] The UV–visible spectra

Table 1: Physical properties of ligand and their complexes

Compounds	*M.wt g/mole*	*M.P°C, (dec.)*	*Color*	*Elemental analysis% theoretical (practical)*				*Cond. Ω^{-1}*
				C	*H*	*N*	*M*	
$[Mn(L)_2H_2O)_2]$	747.66	180	Pale-brown	64.26 64.18	4.31 4.30	11.24 12.30	7.35 7.33	10.5
$[Co(L)_2H_2O)_2]$	751.65	(210)	Brown	63.92 63.89	4.29 4.26	11.18 12.20	7.84 7.82	6.8
$[Ni(L)_2H_2O)_2]$	751.41	(215)	green	63.94 63.91	4.29 4.21	11.18 12.00	7.81 7.79	11.9
$[Cu(L)_2H_2O)_2]$	756.26	(210)	Orange	63.53 63.48	4.26 4.22	11.11 12.05	8.40 8.38	5.7
$[Zn(L)_2H_2O)_2]$	758.11	(224)	Pail-yellow	63.37 63.31	4.25 4.23	11.09 12.06	8.63 8.62	7.9
$[Cd(L)_2H_2O)_2]$	805.13	(200)	yellow	59.67 59.63	4.01 3.90	10.44 11.23	13.96 13.89	10.2
$[Hg(L)_2H_2O)_2]$	893.31	(190)	yellow	53.78 53.66	3.61 3.53	9.40 10.87	22.45 22.01	11.6
$[Pd(L)_2]$	763.11	(200)	Deep brown	62.96 62.93	3.70 3.72	11.01 11.98	13.95 13.95	5.9
$[Pt(L)_2]$	851.77	(200)	brown	56.40 56.38	3.31 3.29	9.87 10.36	22.9022.87	11.6

Table 2: FTIR spectra of the schiff base and complexes

Compound	*υ (O-H)henol*	*υ (C=N) imine*	*υ (C=N) pyrmi.*	*υ (M-N)*	*υ (M-O)*	*Other band (H_2O)+υ(OH)aq.*
LH	3209	1654	1593	-	-	-
$[Mn(L)_2(H_2O)_2]$	-	1620	1597	516	451	3348 756
$[Co(L)_2(H_2O)_2]$	-	1624	1597	532	466	3410 756
$[Ni(L)_2(H_2O)_2]$	-	1627	1597	513	412	3414 752
$[Cu(L)_2(H_2O)_2]$	-	1616	1585	501	466	3402 748
$[Zn(L)_2(H_2O)_2]$	-	1627	1593	513	470	3429 752
$[Cd(L)_2(H_2O)_2]$	-	1620	1593	543	462	3387 752
$[Hg(L)_2(H_2O)_2]$	-	1620	1589	574	420	3441 763
$[Pd(L)_2]$	-	1651	1620	540	470	--
$[Pt(L)_2]$	-	1627	1593	509	459	--

Table 3: The electronic spectral data of the metal complexes with ligand

Compound	*Wave number*		ε_{max} *molar*$^{-1}$ *cm*$^{-1}$	*Assignment*	*Magnetic moment*	*Suggested structure*
	nm	*cm*$^{-1}$				
$[Mn(L)_2(H_2O)_2]$	270	37037	1402	Intra ligand	3.52	Octahedral
	322	31055	789	$^6A_1g_{(F)} \rightarrow {}^6Eg_{(D)}$		
	354	28248	760	$^6A_1g_{(F)} \rightarrow {}^4T_2g_{(D)}$		
	369	27100	402	$^6A_1g_{(F)} \rightarrow {}^4A_1g, {}^4Eg_{(G}$		
	423	23640	287	$^6A_1g_{(F)} \rightarrow {}^4T_2g_{(G)}$		
	523	19120	180	$^6A_1g_{(F)} \rightarrow {}^4T_1g_{(G)}$		
$[Co\ (L)_2(H_2O)_2]$	271	3900	1525	Intra ligand	3.37	Octahedral
	395	25316	671	C.T		
	615	16260	229	$^4T_1g^{(F)} \rightarrow {}^4T_1g^{(p)}$		
	675	14814	298	$^4T_1g^{(F)} \rightarrow {}^4A_2g^{(F)}$		
$[Ni\ (L)_2(H_2O)_2]$	271	36900	1759	Intra ligand	2.86	Octahedral
	395	25316	655	C.T		
	423	23640	490	$^3A_2g_{(F)} \rightarrow {}^3T_1g\ (_p)$		
	515	19417	129	$^3A_2g_{(F)} \rightarrow {}^3T_1g\ (_F)$		
	880	11363	18	$^3A_2g_{(F)} \rightarrow {}^3T_2g\ (_F)$		
$[Cu\ (L)_2(H_2O)_2]$	290	34482	2119	Intra ligand	1.74	Octahedral
	305	32786	419	Intra ligand		
	544	18382	21	$^2B_1g_{(F)} \rightarrow {}^2B_2g_{(F)}$		
	858	11655	44	$^2B_1g_{(F)} \rightarrow {}^2A_1g_{(F)}$ or $^2Eg \rightarrow {}^2T_2g$		
$[Zn(L)_2(H_2O)_2]$	271	36900	981	Intra ligand	--	Octahedral
	395	25316	431	C.T		
	544	18382	55	C.T		
$[Cd\ (L)_2(H_2O)_2]$	272	36764	1750	Intra ligand	--	Octahedral
	340	29411	880	Intra ligand		
	395	25316	445	C.T		
$[Hg\ (L)_2(H_2O)_2]$	271	36900	1695	Intra ligand	--	Octahedral
	376	26595	444	C.T		
$[Pd\ (L)_2]$	270	37037	1498	Intra ligand	Dia.	Square planner
	355	28169	1489	C.T		
	383	26109	706	$^1A_1g \rightarrow {}^1B_1g$		
	764	13089	4	$^1A_1g \rightarrow {}^1B_2g$		
$[Pt\ (L)_2]$	277	36101	2103	Intra ligand	Dia.	Square planner
	397	25188	1098	C.T		
	485	20618	627	$^1A_1g \rightarrow {}^1B_1g$		
	520	19230	588	$^1A_1g \rightarrow {}^1B_2g$		

of ligand and their complexes Table 3. in DMSO as a solvent showed absorption bands between (272, 386 and 471) nm are assigned to π–π*, n–π* for (–C=N, –C=O) transitions and L-LCT for the long pair electrons of nitrogen and oxygen atoms respectively. the UV–visible spectrum of the complexes Table 3, show five absorption bands for Mn(II) complex at 322,354, 369, 423 and 523 nm which are attributed to $^6A_1g_{(F)} \rightarrow {}^6Eg_{(D)}$, $^6A_1g_{(F)} \rightarrow {}^4T_2g_{(D)}$, $^6A_1g_{(F)} \rightarrow {}^4A_1g, {}^4Eg_{(G)}$, $^6A_1g_{(F)} \rightarrow {}^4T_2g_{(G)}$ and $^6A_1g_{(F)} \rightarrow {}^4T_1g_{(G)}$ transitions, respectively, Co(II) complex display peaks at (615 and 675) nm are attributed to $^4T_1g_{(F)} \rightarrow {}^4T_1g_{(p)}$ and $^4T_1g_{(F)} \rightarrow {}^4A_2g_{(F)}$. Ni (II) complex show three peaks at (423, 515 and 880) nm due to $^3A_2g_{(F)} \rightarrow$

Table 4: The antimicrobial activity data for the LH and its complexes

Compounds	*E. coli*	*Staphylococcus aureus*	*Klebsiella pneumoniae*	*Bacillus subtitus*	*Canidia albicans*
Amoxicillin	28	25	8	13	0
Metronidazole	0	0	0	0	18
Am	13	13	25	24	18
AL	16	14	24	26	5
LH	17	17	26	28	18
$[Mn(L)_2(H_2O)_2]$	24	24	27	13	6
$[Co\ (L)_2(H_2O)_2]$	17	21	32	21	15
$[Ni\ (L)_2(H_2O)_2]$	16	25	29	22	6
$[Cu\ (L)_2(H_2O)_2]$	24	20	34	15	5
$[Zn(L)_2(H_2O)_2]$	26	27	30	20	17
$[Cd\ (L)_2(H_2O)_2]$	28	16	32	13	17
$[Hg\ (L)_2(H_2O)_2]$	25	21	29	14	30
$[Pd\ (L)_2]$	23	19	24	28	20
$[Pt\ (L)_2]$	29	17	23	21	16

Where: Al = 2-Hydroxynaphthaldehyde; Am = 3-amino-1-phenyl-2-pyrazoline-5-one.

Table 5: The antioxidant activity data for the LH and its complexes

Compounds	*Conc. (µg/ mL)*	*Scavenging %*	*Inhibition%*	IC_{50}
Vit C.	25	50.7	--	36.3
	75	74.8	--	
LH	25	2.9	2.9	589.6
	75	6.1	6.1	
Mn	25	2.9	2.9	807.8
	75	4.2	4.2	
Co	25	4.8	4.8	526.3
	75	6.3	6.3	
Ni	25	2.5	2.5	600.2
	75	6.1	6.1	
Cu	25	3.6	3.6	668.4
	75	5.0	5.0	
Zn	25	6.3	6.3	309.6
	75	11.4	11.4	
Cd	25	3.8	3.8	560.5
	75	6.2	6.2	
Hg	25	13.5	13.5	---
	75	1.7	1.7	
Pd	25	2.9	2.9	673.9
	75	5.2	5.2	
Pt	25	8.9	8.9	140.6
	75	26.7	26.7	

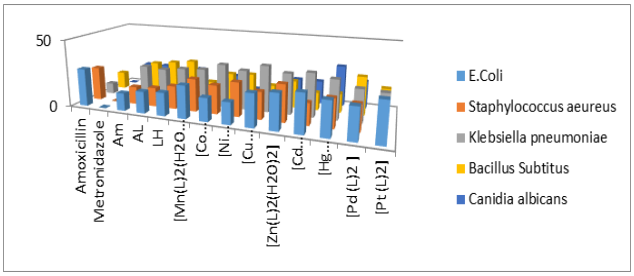

Figure 4: The zone of inhibition values for antimicrobial activity.

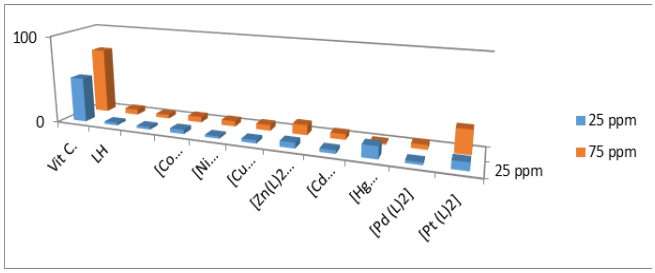

Figure 5: The inhibition values for antioxidant activity.

$^3T_1g_{(p)}$, $^3A_2g_{(F)} \rightarrow {}^3T_1g_{(F)}$ and $^3A_2g_{(F)} \rightarrow {}^3T_2g_{(F)}$ transitions respectively.[25] Cu(II) complex display two peaks at 544nm and 858nm may be due to $^2B_1g_{(F)} \rightarrow {}^2B_2g_{(F)}$ and $^2B_1g_{(F)} \rightarrow {}^2A_1g_{(F)}$ or $^2Eg \rightarrow {}^2T_2g$. Zn(II) , Cd(II) and Hg(II) complexes shows many peaks at (395,544), 395nm and 376nm were due to C.T transitions respectively while the peaks of Pd(II) and Pt(II) complexes at (383, 485) nm and (764, 520) nm may be attributed to $^1A_1g \rightarrow {}^1B_1g$ and $^1A_1g \rightarrow {}^1B_2g$, from these results, we conclude that octahedral geometry around Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) complexes while the Pd(II) and Pt(II) complexes square-planar geometry.[26,27]

Antimicrobial Activity

The Antibacterial and antifungal activity of Am(3-amino-1-phenyl-2-pyrazoline-5-one), Al (2-Hydroxynaphthaldehyde), Schiff base LH and its metal(II) complexes Figure 4 and Table 4, against bacteria such as *S. aureus*, *B. subtitus as a gram+, K. pneumonia* and *E. coli*, and fungi such as *C. albicans* was

tested in order to evaluate potential antimicrobial agents. According to the data can be observed that the [HL] and its complexes did show good biological activity, this means that the activity of the compounds synthesized against different microorganisms is enhanced with chelation with different biological active metals.[28]

Antioxidant Activity

Using the DPPH assays method, the ability of the newly synthesized schiff bases ligands and their complexes to scavenge free radicals may be assessed, and the findings are shown in Table 5 and Figure 5. Because of its stability and simplicity, the DPPH radical is frequently used to evaluate antioxidant activity quickly. The creation of stable DPPH decreased the intensity of the DPPH band, demonstrating the ability of metal complexes and ascorbic acid (Vitamin C used as a standard) to scavenge free radicals.[29] Increased antioxidant activity is indicated by a decrease in absorbance and a decrease in the IC_{50} value.[30] The IC_{50} values of the test compounds were observed has a stronger scavenging activity.

CONCLUSION

The prepared compounds were confirmed by using spectroscopic techniques. The antibacterial properties of Schiff base and its metal complexes were investigated against a varied range of gram-positive and gram-negative bacterial strains and fungi. Also, the antifungal and antioxidant assays were studied. The complexes shown better biological activities for antibacterial and antifungal, whereas Pt (II), Zn(II) and Hg(II) complexes shown good DPPH scavenging activity.

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