# Synthesis, Characterization, Antimicrobial and Antioxidant Study of New Complexes Schiff Base Derived from 2, 5-diChloroaniline

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# ABSTRACT

Metal complexes of Mn(II), Co(II), Ni(II), Cu(II), Cd(II), Zn(II), Hg(II), Pd(II), and Pt(II) with Schiff base ligand (LH) derived from 2,5-dichloroaniline and 2-hydroxy-5-metheylbenzalaldehyde were synthesized and characterized using a variety of spectrophotometric techniques The findings of the spectroscopic analysis indicated that (LH) behaved as a binary coordinating agent to the metal ion by the N and O atoms, and the geometry shape of the complexes was octahedral, with the exception of the Pd and Pt complexes, which had a square planar geometry. Using the DPPH radical scavenging method, we investigated the antimicrobial activity of the compound against *Staphylococcus aureus* and *Escherichia coli*, as well as the antifungal activity of the compound against *Candida albicans*. In addition, the Schiff base ligand and its metal complexes were tested to determine their effectiveness as antioxidants. Antioxidant activities were demonstrated by the compounds, which worked to eliminate potentially damaging free radicals from the system.

Keywords: Transition metal, Antioxidant, DPPH radical, Antibacterial activity.

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# INTRODUCTION

The Schiff bases are a significant category of ligands that bind with metal ions by way of the azomethine nitrogen.<sup>1</sup> Schiff bases form during the condensation of amines and carbonyl compounds. A ligand is a "Schiff base" if it contains a carbon double bond and a nitrogen atom connected to an aryl or alkyl group, but not to a hydrogen atom. In other words, the ligand does not contain a hydrogen atom. The name of the ligand comes from this property. The carbonyl groups of aldehydes and ketones, respectively, are what give rise to the amines known as aldimines and ketoimines. These molecules acted as a binding site for metal ions via the nonbonding electrons of nitrogen, and aldimines and ketoimines are formed from the carbonyl groups of aldehydes and ketones, respectively.<sup>2,3</sup> The chemical formula for 2, 5-dichloroaniline, which represents the structure of this compound, is C<sub>6</sub>H<sub>3</sub>C<sub>12</sub>NH. This solid does not dissolve in water and lacks any discernible color. Dichloroaniline can take any one of its six possible forms, and this one is one of them. Due to the fact that it contains the primary amine group, it is necessary in the process of synthesizing the ligand for Schiff bases.<sup>4</sup> As a byproduct of the reaction between 2-hydroxy-1-naphthaldehyde and 2.5-dichloroaniline, a number of transition metal complexes are produced, some of which are Schiff base ligands. Infrared,

ultraviolet-visible, and elemental analyses were utilized, and all three were successful in identifying the substance.<sup>5</sup> In an aqueous medium with an alkaline pH, the polymer PDCBAP was manufactured by using air and NaOCl as the reactants.<sup>6</sup> In order to determine the structure of the Schiff base molecule N-enzylidene-2, 5-dichloroaniline, spectroscopic methods were synthesised and used.<sup>7</sup> Slow evaporation was used to produce the dimorphic forms of anthracene Schiff base crystals (N-(anthracen-9-ylmethylene)-2,5-dichloroaniline).<sup>8</sup> This research aims to synthesize, characterize, and conduct antibacterial and antioxidant tests on transition metal complexes containing Schiff bases derived from 2, 5-dichloroaniline and 2, 2-hydroxy-5-methylbenzaldehyde.

#### MATERIALS AND METHODS

#### **Materials and Instruments**

Stuart melting point kit is the tool used to measure melting points. The Euro CHN is used to measure all compounds (EA 3000). On a Shimadzu UV- 160A, ultraviolet-visible spectra are run.

The FTIR spectra are validated using an FTIR-8400S spectrophotometer on 4000–400 cm<sup>-1</sup> in KBr discs and an AA-620G Shimadzu spectrophotometer for the atomic absorption method.



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

The <sup>1</sup>H-NMR spectra were carried out at Mashhad University of medical sciences using a Bruker Ultra Shield 300 MHZ for <sup>1</sup>H-NMR with DMSO as the solvent, and the mass spectra were obtained by positive LC- MS using an Agilest Q-trap3200 mass spectrometer.

The magnetic sensitivity was also assessed using a Bruker BM6 instrument and the Faraday method.

# Preparation of Ligand (E)-2-(((2,5-dichlorophenyl)imino) methyl)-4-methylphenol (LH)

The Schiff base was created by mixing ethanol solutions of 2-hydroxy-5-metheylbenzaldehyde and 2, 5-chloroaniline (0.16 g, 0.001 mol each) (0.163 g, 0.001 mol). Following a period of five to six hours of refluxing, the resulting solution was brought down to room temperature, at which point the crystallized bright yellow substance formed. The substance was filtered, washed with cold ethanol, dried at room temperature, and re-crystallized with ethanol. The final results were a yield of 77% and a mass of 280.15. The MP values are 116, 59.53, 3.92, and 7.03°C.

# **Preparation of Complexes**

A solution of metal chloride that contained 0.126 g of Mn(II), 0.237 g of Co(II), 0.237 g of Ni(II), 0.17Cu(II), 0.17 g Zn(II), 0.201 g of Cd(II), 0.272 g of Hg(II), and 0.177 g of Pd(I) had a solution of ligand (0.560 g, 2 mmol) and KOH (2 mmol) added to (II). After being stirred for 160 minutes, the mixture is filtered through anhydrous CaCl<sub>2</sub> and then dried.

# **Biological Activity**

The compounds produced were tested using the disc diffusion method against one type of fungi, such as *Candida albicans*, and two types of bacteria, such as *Escherichia coli* and *Staphylococcus aureus*. The results of these tests were compared with each other. Producing the sample solution involves using DMSO as the solvent, and the concentration that is used is 0.001M. After bacteria and fungi have been allowed to grow for 24 hours at room temperature, the diameter of the inhibitory zone is measured to determine how much they have expanded.<sup>9-11</sup>

### **Antioxidant Study**

Radicals of 1,1-diphenyl-2-picrylhydrazyl being taken up by the body (DPPH). The evaluation of a substance's ability to scan DPPH radicals is the test typically used in research on antioxidant activity. It is a quick method for determining the radical-scavenging activity of various chemicals.<sup>12</sup> At three different concentrations of DPPH radicals (25, 50, and 100 g/mL), all of the substances and associations were evaluated to determine whether they could scavenge free radicals. After 30 minutes of incubation in the dark, absorbance (A) was measured using a UV-vis Shimadzu spectrophotometer against a blank at 517 nm. IC inhibition of DPPH color values were also determined at this time. There were identical sets of experiments conducted. The following formula was utilised to estimate the percentage of DPPH inhibition (Antioxidant Activity%):<sup>13-15</sup>

Antioxidant% = A blank - A sample / A blank 100.

# **RESULTS AND DISCUSSION**

The melting point and decomposition temperature of Schiff base ligand and metal complexes were calculated, and their molar absorptivity values ranged from 10.08 to 14.74 cm<sup>2</sup> mol<sup>1</sup> in DMSO.<sup>16</sup> Table 1 displays the microanalysis and atomic absorption M% values.

# <sup>1</sup>H-NMR and Mass Spectra of Ligand LH

Figure 1 shows the spectrum of the Schiff base ligand. The singlet signal at 2.48, 3.35 and 2.27 ppm can be attributed to the group of solvent and methyl protons. The numerous signals between  $\delta$  (7.26–7.73) ppm correspond to aromatic protons, whilst the singlet signals at  $\delta$  (8.97 and 6.87) ppm are attributable to the hydrogen (HC=N-) group and OH protons.<sup>17,18</sup> The spectrum LH, depicted in Figure 2 and Scheme 2, indicated the presence of peaks at m/z 279.2 (54%), 243.7 (100%), 134.2 (16%), 77.2 (18%) and 58.2 (8%), which were ascribed to the [M]+ molecular ion, [M- (Cl)]+, [M- C<sub>6</sub>H<sub>2</sub>Cl]+, [C<sub>6</sub>H<sub>5</sub>]+, and [C<sub>2</sub>H<sub>4</sub>NO]+, respectively.

# FTIR Spectra, UV-visible and Magnetic Sensitivity<sup>19</sup>

It was determined that a band at 1612 cm<sup>-1</sup> in the spectrum of the Schiff base corresponds to the (HC=N-) stretching vibration, and that another band at 3405 cm<sup>-1</sup> corresponds to the (O-H) stretching vibration (Table 2). Deprotonation occurs following coordination to the metal (II) ion, as evidenced by the removal of the wide band in the spectra of metal complexes. The migration of (C=N) stretching vibration bands in the spectra of metal complexes in the region revealed that complexation had taken place (1616-1639 cm<sup>-1</sup>). The emergence of additional bands in the spectra of metal complexes at (570-605) cm<sup>-1</sup> and (466-486) cm<sup>-1</sup> is also indicative of the coordination of the Schiff base ligand to the metal(II) ion. These new bands have been attributed to (M-O) and (M-N) stretching vibrations, respectively.<sup>20</sup> The magnetic sensitivity of the cobalt and copper complexes, which was measured at 2.66 and 1.74, respectively, suggested that they possessed paramagnetic properties.<sup>21</sup> Because of the \*transition, the UV-vis spectra of LH displayed peaks at 296 and 318, and at 398 nm, they displayed a peak that was caused by the n\* transition.<sup>22,23</sup> The complexes in Table 3 all have peaks in their electronic spectra between 270 and 280 nm. These peaks are caused by the ligand field. The spectra of the Mn(II) complex Exhibits absorption peaks at (345, 510, and 625) nm. These peaks are caused by the charge

Table 1: The Physical properties of ligand and their complexes								
Commente	M.wt (g/mole)	M.P (°C)	Color	Elemental analysis % Theoretical (practical)			Cond.	
Compounds				С	Н	Ν	М	$\Omega^{-l}$
$[Mn(L)_2H_2O)_2]$	649.25	<250 d	Pail - brown	51.80 51.76	3.73 3.69	4.31 5.11	8.46 8.37	14.74
$[\mathrm{Co}(\mathrm{L})_{2}\mathrm{H}_{2}\mathrm{O})_{2}]$	653.25	<250 d	Brown	51.48 51.35	3.70 3.65	4.29 4.88	9.02 9.00	12.11
[Ni (L) <sub>2</sub> H <sub>2</sub> O) <sub>2</sub> ]	653.01	<250 d	Pail-brown	51.50 51.48	3.70 3.66	4.29 4.98	8.99 8.77	10.26
$[\mathrm{Cu}(\mathrm{L})_{2}\mathrm{H}_{2}\mathrm{O})_{2}]$	657.86	<250 d	Orange	51.12 51.09	3.68 3.61	4.26 4.76	9.66 9.59	13.90
$[\mathrm{Cd}(\mathrm{L})_{2}\mathrm{H}_{2}\mathrm{O})_{2}]$	706.72	124	Pail- yellow	47.59 47.47	3.42 3.33	3.96 4.54	15.91 15.87	11.82
$[Zn (L)_2H_2O)_2]$	659.70	oily	yellow	50.98 50.85	3.67 3.62	4.25 4.89	9.91 9.95	10.19
$[\mathrm{Hg}(\mathrm{L})_{2}\mathrm{H}_{2}\mathrm{O})_{2}]$	794.90	126	pail yellow	42.31 42.27	3.04 3.25	3.52 4.31	25.23 25.11	10.08
$[Pd(L)_2]$	664.70	<250 d	yellow	50.59 5048	3.03 3.00	4.21 4.57	16.01 15.98	11.56
[Pt( L) <sub>2</sub> ]	753.36	148	yellow	44.64 44.57	2.68 2.79	3.72 4.02	25.89 25.82	11.03



Scheme 2: Fragmentation of LH



Figure 1: HNMR Spectrum of LH

Compound	v (O-H) phenol	$v(C=N)_{imine}$	v (M-N)	v (M-O) v (M-O) <sub>aq.</sub>	$(H_2O)+v(OH)aq.$
LH	3405	1612			
$[Mn(L)_2H_2O)_2]$		1618	590	470(447)	3078, 887
$[\mathrm{Co}(\mathrm{L})_{2}\mathrm{H}_{2}\mathrm{O})_{2}]$		1616	605	482(459)	3376, 813
$[\mathrm{Ni}(\mathrm{L})_{2}\mathrm{H}_{2}\mathrm{O})_{2}]$		1620	601	488(466)	3375,813
$[Cu(L)_2H_2O)_2]$		1639	597	493(451)	3433,810
$[Zn(L)_2H_2O)_2]$		1631	597	478(458)	3421,833
$[Cd(L)_2H_2O)_2]$		1616	597	478(466)	3443,833
$[Hg(L)_2H_2O)_2]$		1616	570	483(470)	3246,833
$[Pd(L)_2]$		1624	597	486	
[Pt(L) <sub>2</sub> ]		1612	597	470	

transmissions 6A1g(F)4T1g(G) and 6A1g(F)4T2g(D). The spectra of the cobalt complex exhibits absorption peaks at (345), 368, 508, and 733 nm. These peaks are caused by the charge transmissions 4T1g(F)4T1g(p) and 4TIg(F)4A2g(F). The charge transmissions 3A2g(F)3T1g(P), 3A2g(F)3T1g(F),

and 3A2g(F)3T2g(F) cause the Ni(II) complex to exhibit absorption peaks at 348, 410, 725, and 987 nm, respectively. In the spectrum of the copper complex, charge transfer and 2Eg 2T2g cause absorption peaks to appear at 345 and 891 nm, respectively. The CT causes peaks to appear at (348,345



Figure 2: The Mass Spectrum of LH



Figure 3: the values of Zone inhibition for Antimicrobial Activity

and 345) nm in the spectra of zinc, cadmium, and mercury complexes, which can be found in.  $^{\rm 24-26}$  The peaks that appear at (350,350) nm for Pd(II) and Pt(II) complexes correspond to C.T, 688 nm, and 883 nm, respectively. Based on all of these results, 1A1g 1B1g and 1A1g 1B2g, octahedral geometry is

		Table 3: Magnetic s	ensitivity and electron	ic spectral of compounds	
Compound	$\mu_{eff.}$ B.M.	$\lambda$ (nm)	$\acute{U}$ cm <sup>-1</sup>	E <sub>max</sub> molar <sup>-1</sup> cm <sup>1</sup>	Assignments
LH		264 311 348	37878 32154 28735	947 873 835	$\pi - \pi^*$ $\pi - \pi^*$ $n - \pi^*$
$[Mn(L)_2(H_2O)_2]$	5.35	265 305 345 510	37735 32786 28985 19607	1270 1730 1759 4	Intra ligand Intra ligand C.T ${}^{6}A_{1}g(_{E}) \rightarrow {}^{4}T_{1}g(_{C})$
[Co(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	3.76	625 277 345 368 508 733	16000 36101 28985 27173 19685 13642	3 2070 2324 1195 28 24	${}^{6}A_{1}g(_{F}) \rightarrow {}^{4}T_{2}g(_{D})$ Intra ligand C.T ${}^{4}T_{1}g(F) \rightarrow {}^{4}T_{1}g(P)$ ${}^{4}T_{2}g(F) \rightarrow {}^{4}A_{2}g(F)$
[Ni(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	2.83	265 305 336 348 410 725 987	37735 32786 29761 28735 24390 13793 10131	986 1147 1001 1098 24 10 12	Intra ligand Intra ligand Intra ligand C.T ${}^{3}A_{2}g(_{F}) \rightarrow {}^{3}T_{1}g(p)$ ${}^{3}A_{2}g(_{F}) \rightarrow {}^{3}T_{1}g(F)$ ${}^{3}A_{2}g(_{F}) \rightarrow {}^{3}T_{2}g(F)$
[Cu(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	1.75	266 305 345 891	37594 32786 28985 11223	1187 1787 563 10	Intra ligand Intra ligand C.T ${}^{2}Eg \rightarrow {}^{2}T_{2}g$
$[Zn(L)_2(H_2O)_2]$		264 311 348	37878 32154 28735	1045 1045 1024	Intra ligand Intra ligand C.T
$[Cd(L)_2(H_2O)_2]$		266 305 345	37594 32786 28985	1279 1742 1660	Intra ligand Intra ligand C.T
$[\mathrm{Hg}(\mathrm{L})_2(\mathrm{H}_2\mathrm{O})_2]$		266 305 345	37594 32786 28985	1240 1515 1377	Intra ligand Intra ligand C.T
[Pd(L) <sub>2</sub> ]	Dia.	266 305 350 688	37594 32786 28571 14534	1172 1615 1173 8	Intra ligand Intra ligand C.T ${}^{1}A_{1}g \rightarrow {}^{1}B_{2}g$
[Pt(L) <sub>2</sub> ]	Dia.	267 301 350 883	37453 33222 28571 11325	1397 1635 84 5	Intra ligand Intra ligand C.T ${}^{1}A_{1}g \rightarrow {}^{1}B_{2}g$
		005	11323	J	$n_1g \rightarrow D_2g$



Figure 4: DPPH scavenging activity of ligand and their some complexes

Compounds	Escherichia coli	Staphylococcus aureus	candida albicans
Ceftriaxone	35	30	0
Metronidazoleole	0	0	15
LH	21	18	12
$[Mn(L)_2(H_2O)_2]$	19	22	19
$[\mathrm{Co}(\mathrm{L})_2(\mathrm{H}_2\mathrm{O})_2]$	19	26	15
$[\mathrm{Ni}(\mathrm{L})_2(\mathrm{H}_2\mathrm{O})_2]$	25	21	17
$[Cu(L)_2(H_2O)_2]$	25	21	15
$[Zn(L)_2(H_2O)_2]$	21	32	15
$[Cd(L)_2(H_2O)_2]$	22	22	20
$[\mathrm{Hg}(\mathrm{L})_2(\mathrm{H}_2\mathrm{O})_2]$	31	31	30
$[Pd(L)_2]$	22	20	21
$Pt(L)_2$ ]	22	21	15

**Table 4:** The inhibition zone of prepared compounds



Figure 5: The octahedral geometry shape of complexes

 Table 5: IC<sub>50</sub> and DPPH scavenging activity of ligand and their some complexes

	1			
Sample	conc. µg			IC <sub>50</sub>
Tested with DPPH	25	50	100	
$[Pd(L)_2]$	1.107	1.081	1.041	405.1
$[Pt(L)_2]$	1.066	0.988	0.962	242.2
$[\mathrm{Co}(\mathrm{L})_2(\mathrm{H}_2\mathrm{O})_2]$	1.227	1.201	1.15	446.8
$[Ni(L)_2(H_2O)_2]$	1.338	1.261	1.26	625.5
$[Mn(L)_2(H_2O)_2]$	1.210	1.207	1.199	2658
$[Cu(L)_2(H_2O)_2]$	1.257	1.233	1.221	1143
$[Zn(L)_2(H_2O)_2]$	1.193	1.186	1.178	215.9
LH	1.242	1.234	1.122	320.8
Vit.C	0.163	0.095	0.065	14

advantageous for complexes of Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II), and Hg(II), whereas square-planar geometry is advantageous for complexes of Pd(II) and Pt(II).<sup>27</sup>

#### **Antibacterial Activities**

*In-vitro* evaluations of all compounds produced were conducted using microorganisms consisting of one strain of *Staphylococcus aureus, Escherichia coli* as a bacterium, and *Candida albicans* as a fungus (Table 4 and Figure 3). Because of "Tweedy's" chelation theory, the polarity of the metal ion will be reduced to a greater extent in the complexes. This will cause the ligand orbital to overlap, which will result in a partial sharing of the M +2 with donor groups. Additionally, the lipophilicity of the complexes is increased due to the delocalization of electrons with a charge of over the entire chelate ring as well as the large ring size of the ligands moiety.<sup>27,28</sup>

# Evaluation of Antioxidant Activity by DPPH Radical Scavenging Method:

The ability of newly synthesized Schiff bases ligands and some of their complexes to scavenge free radicals can be evaluated using the DPPH assays method; the results are presented in Table 5 and Figure 4, respectively. The DPPH radical is frequently used for quickly testing antioxidant activity because of its stability and its relative lack of complexity. The production of stable DPPH decreased the intensity of the DPPH band, which showed that ligand and metal (II) complexes as well as vitamin C, can neutralize free radicals.<sup>29</sup> The IC50 values of the test ligand and a few complexes were found to have a greater scavenging action.<sup>30</sup>

# CONCLUSION

The synthetic ligand and its complexes were used for diagnostic purposes by spectroscopic techniques. All complexes share octahedral geometry, whereas square planar geometry is characteristic of Pd and Pt complexes (Figure 5). The antibacterial activities of Schiff base and its metal complexes were investigated against a wide variety of bacterial and fungal strains. Experiments on antibacterial and antioxidant activity were carried out, and the results demonstrated that the complexes possessed superior antibacterial, antifungal, and DPPH-scavenging properties.

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