

Synthesis, Characterization, Anti-bacterial Activity Study of Vanillin Schiff Base Complexes

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Abstract

The Fe(II), Co(II), Ni(II) Cu(II), Zn(II) and Hg(II) complexes were prepared by reacting their salts with the derived Schiff base ligands from condensing vanillin and o-phenylenediamine with a ratio (1:1), (2:1) respectively. Ligands and their complexes were distinguished using analytical, physical and spectroscopic methods. The elemental analysis values show that stoichiometry to be 1:1 [M: L]. Molar conductivity measurements detect that the complexes are non-electrolytic. From the information obtained through electronic measurements, magnetic sensitivity. The square planar geometry around of Ni (II) and Cu (II), and tetrahedral geometry around of the other metals. The prepared complexes showed different activity towards inhibiting bacterial growth for E. coli k.pneumonia.

1. Introduction:

The Schiff bases are compounds containing the (C=N) group and are prepared from the reaction of the primary amines with the compounds containing the carbon group[1]. Because of the presence of the group (C=N) considered as an important class of ligands which have been extensively studied in coordination chemistry and has many industrial and biological applications.[2] Schiff bases have many uses for their ability to act as catalysts in the hydrogenation of olefins, as well as for antibacterial[3], antifungal[4],[5] and anti-tumor medications[6]. Some of Schiff bases use as anticorrosion, It has distinct properties because it contains the nitrogen atom, which gives back to electrons, which provides electrons to the metal surface and prevents corrosion[7]. In this paper, we prepared the Schiff bases complexes were derived from vanillin and o-phenylenediamine.

2. Experimental:

2.1 Materials and Methods:

The chemicals used in this study the highest purity analytical reagent grade. (Fluka A.G., Merck, BDH) and used without purification. Infrared spectra measured by Nicolet FT-IR In-

frared Spectrophotometer 200-4000 cm^{-1} range. NMR spectroscopy recorded by Bruker Bio Spin GmbH 400 MHz instrument. The conductivity meter Jenway PCM3 using to measure conductivity and accomplished on 10^{-3} M solution of the complexes in DMF. The electronic spectra measured in DMF as solvent at 25^o C by a PgT92+ UV-visible spectrophotometer. Determination of metal percentage were done employing spectrophotometric measurements using VarianAA- 240FS atomic absorption Spectrophotometer, Elemental analyses (C.H.N) were carried out using micro analytical techniques on Costech Instruments Elemental Combustion. Magnetic sensitivity measurements were recorded at 25 C^o by Gouy's method using the Sherwood Scientific instrument.

2.2 Syntheses of the ligand ((Z)-4-(((2-aminophenyl)imino) methyl)-2-methoxyphenol) (L₁):

Vanillin (1.37 g, 0.009 mmol) was dissolved in methanol (15ML), and methanolic solution of o-Phenylenediamine (1g, 0.009 mmol) was added in the ratio (1:1). Some of glacial acetic acid drops was added. The reaction was stirred for (2 h), the volume reduced by rotary evaporator, and it was recrystallized from cold methanol (75%). Scheme 1.

Table 1. Some physical and analytical properties of the prepared complexes.

Comp No.	Chemical formula		m.p $^{\circ}C$	Λ $ohm - 1.cm^2.mol^{-1}$	$M\%$	Elemental $C\%$	Analysis $H\%$	$N\%$
1.	[Fe(L1)SO ₄]	Brown	Dec.	22.5	14.16 (14.16)	42.66 (42.66)	3.55 (3.53)	7.11 (7.01)
2.	[Co(L1)Cl ₂]	Blue	148-150	9.5	15.83 (15.81)	45.28 (45.26)	3.76 (3.66)	7.54 (7.50)
3.	[Ni(L1)Cl ₂]	Dark green	147-150	8.6	15.76 (15.74)	45.20 (45.18)	3.76 (3.74)	7.53 (7.50)
4.	[Cu(L1)Cl ₂]	Dark brown	132-135	15.3	16.87	44.64	3.72	7.44
5.	[Zn(L1)SO ₄]	White	103-106	20.1	16.21 (16.20)	41.64 (41.44)	3.47 (3.47)	6.94 (6.91)
6.	[Cd(L1)Cl ₂]	Light yellow	150-152	10.2	26.43 (26.39)	39.50 (39.41)	3.29 (3.29)	6.58 (6.58)
7.	[Hg(L1)Cl ₂]	Creamy	143-147	18.7	39.07 (39.07)	32.72 (32.72)	2.72 (2.72)	5.45 (5.45)
8.	[Fe(L2)SO ₄]	Brown	142-145	16.2	13.88 (13.88)	50.01 (50.01)	3.78 (3.78)	5.30 (5.30)
9.	[Co(L2)Cl ₂]	Blue	158-160	10.5	11.64 (11.50)	52.20 (52.23)	3.95 (3.93)	5.53 (5.50)
10.	[Ni(L2)Cl ₂]	Dark green	156-159	11.2	11.59 (11.58)	52.21 (52.21)	3.95 (3.86)	5.55 (5.54)
11.	[Cu(L2)Cl ₂]	Dark blue	158-160	11.1	12.34 (10.50)	51.71 (51.68)	3.91 (3.92)	5.48 (5.46)
12.	[Zn(L2)SO ₄]	White	146-149	10.3	12.16 (12.15)	49.13 (48.03)	3.72 (3.73)	5.21 (5.01)
13.	[Cd(L2)Cl ₂]	Light yellow	164-166	6.8	20.10 20.13	47.21 47.25	3.57 3.55	5.00 5.05
14.	[Hg(L2)Cl ₂]	White	130-133	18.2	30.98	40.78	3.08	4.32

2.3 Syntheses of the ligand (4,4'-((1Z,1'E)-(1,2-phenylenebis (azanylylidene)) bis (methanylylidene)) bis (2-methoxyphenol))(L₂):

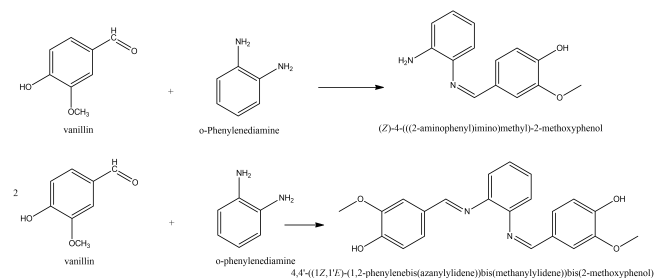
The ligand was prepared similarly as in the procedures 2.2 except change the ratio of (2:1) o-phenylenediamine with vanillin in absolute ethanol respectively. (78%). Scheme 1.

2.4 Synthesis of the complexes [ML₁Cl₂]M=Fe(II) Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II):

Metal salts of CoCl₂.6H₂O (2.30g, 0.001 mol), NiCl₂.6H₂O (2.36 g, 0.001 mol), CuSO₄ (0.167 g, 0.001 mol), ZnCl₂ (0.136 g, 0.001 mol) and CdCl₂ (0.183, 0.001mol) dissolved in ethanol are added to the solution of (L₁) (0.242 g, 0.001 mol) drop wise the reaction mixture was re fluxed and stirred for 4 hours. The precipitate formed was filtered, washed with 10 mL of cold ethanol and dried under reduced pressure.

2.5 Synthesis of the complexes [ML₂Cl₂]M=Fe(II) Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II):

The preparation was done in the same way as before, only the ligand was changed.



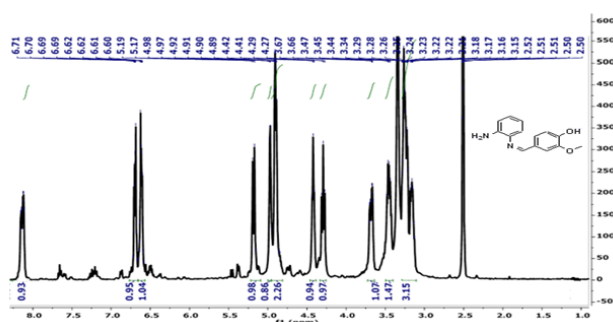
Scheme 1. preparation of ligands

Table 2. IR Spectral Data of the ligand and the complexes.

Compound	$\nu(\text{C}=\text{N})$	$\nu(\text{M}-\text{N})$	$\nu(\text{SO}_4)$	$\nu(\text{M}-\text{Cl})$	$\nu(\text{O}-\text{H})$
L1(1:1)	1607 _m	–	–	–	3363
[Fe(L1)SO ₄]	1624b _m	531 _m	1134 _s , 1096 _s , 1017 _m , 836 _m	–	3390
[Co(L1)Cl ₂]	1605 _m	455 _m	–	295 _w	3339
[Ni(L1)Cl ₂]	1605 _m	457 _m	–	296 _w	3340
[Cu(L1)Cl ₂]	1606 _m	517 _m	–	213 _{sh}	3358
[Zn(L1)SO ₄]	1604 _m	568 _m	1134 _s , 1103 _s , 1086 _s , 875 _m	–	3349
[Cd(L1)Cl ₂]	1605 _m	456 _m	–	295 _w	3340
[Hg(L1)Cl ₂]	1605 _m	456 _m	–	301 _w	3338
L2(2:1)	1606 _s	–	–	–	3376
[Fe(L2)SO ₄]	1623 _m	455 _w	1103 _s , 1078 _s , 1016 _s , 992 _m – 3379	–	–
[Co(L2)Cl ₂]	1605 _m	456 _w	–	221 _w	3379
[Ni(L2)Cl ₂]	1605 _m	455 _w	–	278 _w	3299
[Cu(L2)SO ₄]	1605 _m	452 _w	1143 _s , 1107 _s , 1048 _m , 836 _m	–	3299
[Zn(L2)SO ₄]	1607 _m	458 _m	1139 _s , 1113 _s , 1096 _s , 885 _m	–	3349
[Cd(L2)Cl ₂]	1605 _m	455 _w	–	279 _w	3298
[Hg(L2)Cl ₂]	1605 _m	456 _w	–	279 _w	3298

Table 3. ¹HNMR Data of the ligands and the Zn (II) complexes

Compound	$\delta(\text{H}-\text{C}=\text{N})\text{ppm}$	$\delta(\text{Ar}-\text{OH})\text{ppm}$	$\delta(\text{OCH}_3)\text{ppm}$	$\delta(\text{Ar}-\text{NH}_2)\text{ppm}$
L1	8.15	3.74	3.26	4.90
L2	(6.6, 6.7)	3.45	(3.16-3.26)	3.47
[Zn(L1)SO ₄]	8.97	6.21	3.3	5.6

**Figure 1.** ¹HNMR spectrum of the ligand (L1)

3. Results and Discussion:

The data of physical and analytical properties of the Schiff base ligands and their complexes are given in Table 1. The complexes dissolve in DMF and DMSO. Molar conductivity was measured in DMF solution. The low molar conductance

values of 10^{-3} solutions in DMF of complexes indicate that non-electrolytic [8]. As shown in Table 1.

3.1 Infra-Red Spectroscopy:

Common Infrared Frequencies for Schiff Base and their complexes are described in Table 2. In Schiff base ligands spectrum the absorption band at 1650 -1850 Cm^{-1} due to (C=O) group is absence and observed a strong band at 1677 cm^{-1} can be traced back to (C=N) azomethine group[9] [10] Indicated that the ligands were formed [11]. This band shifted to lower and higher frequency in the spectra of its metal complexes [12] [13]. The ligands and complexes spectra showed band at (3298- 3390) Cm^{-1} arising O-H phenolic. This suggests that the oxygen atom of the hydroxyl group does not participate in coordination[14]. Table 2.

3.2 ¹H NMR Spectroscopy:

The Figures 1, 2 and 3 represent the ¹HNMR spectra of ligands and Zn (II) complex, Were measured in $\text{DMSO}-d^6$ sol-

Table 4. the magnetic measurement and electronic spectra values.

NO.	Chemical formula	Band position	Assignments	Geometry	μ_{eff} B.M
1.	L1	262 302	$n \rightarrow \pi^*$ $\pi \rightarrow \pi$		
2.	L2	262 302	$n \rightarrow \pi^*$ $\pi \rightarrow \pi$		
3.	<i>Fe(L1)SO₄</i>	257 298 868	$n \rightarrow \pi^*$ $\pi \rightarrow \pi$ ${}^5E \rightarrow {}^5T_2$	Tetrahedral	5.1
4.	<i>Co(L1)Cl₂</i>	260 300 – – 645	$n \rightarrow \pi^*$ $\pi \rightarrow \pi$ ${}^4A_2(F) \rightarrow {}^4T_2(F)$ ${}^4A_2(F) \rightarrow {}^4T_1(F)$ ${}^4A_2(F) \rightarrow {}^4T_1(P)$	Tetrahedral	4.4
5.	<i>Ni(L1)Cl₂</i>	242 280 540 351	$n \rightarrow \pi^*$ $\pi \rightarrow \pi$ ${}^1A_{1g} \rightarrow {}^1A_{2g}$ ${}^1A_{1g} \rightarrow {}^1B_{1g}$	Square planar	dia
6.	<i>Cu(L1)SO₄</i>	260 290 587	$n \rightarrow \pi^*$ $\pi \rightarrow \pi$ Combination of transition ${}^2B_{1g} \rightarrow {}^2A_{1g}$ ${}^2B_{1g} \rightarrow {}^2E_g$	Square planar	1.5
7.	<i>Fe(L2)SO₄</i>	258 284 860	$n \rightarrow \pi^*$ $\pi \rightarrow \pi$ ${}^5E \rightarrow {}^5T_2$	Tetrahedral	5.2
8.	<i>Co(L2)Cl₂</i>	258 298 – – 602	$n \rightarrow \pi^*$ $\pi \rightarrow \pi$ ${}^4A_2(F) \rightarrow {}^4T_2(F)$ ${}^4A_2(F) \rightarrow {}^4T_1(F)$ ${}^4A_2(F) \rightarrow {}^4T_1(P)$	Tetrahedral	4.2
9.	<i>Ni(L2)Cl₂</i>	260 300 627 394	$n \rightarrow \pi^*$ $\pi \rightarrow \pi$ ${}^1A_{1g} \rightarrow {}^1A_{2g}$ ${}^1A_{1g} \rightarrow {}^1B_{1g}$	Square planar	dia
10.	<i>Cu(L1)SO₄</i>	282 300 598	$n \rightarrow \pi^*$ $\pi \rightarrow \pi$ Combination of transition ${}^2B_{1g} \rightarrow {}^2A_{1g}$ ${}^2B_{1g} \rightarrow {}^2E_g$	Square planar	1.5

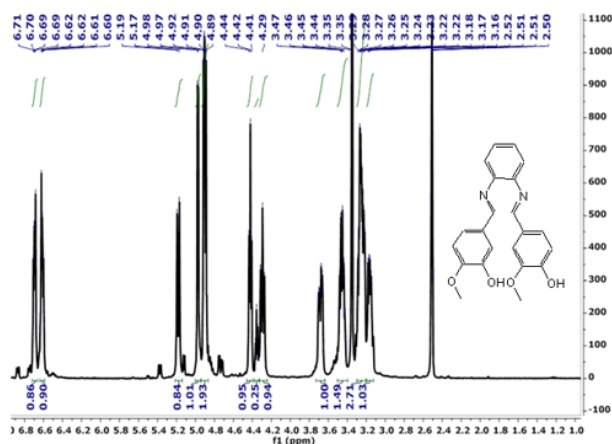


Figure 2. ^1H NMR spectrum of the ligand (L2)

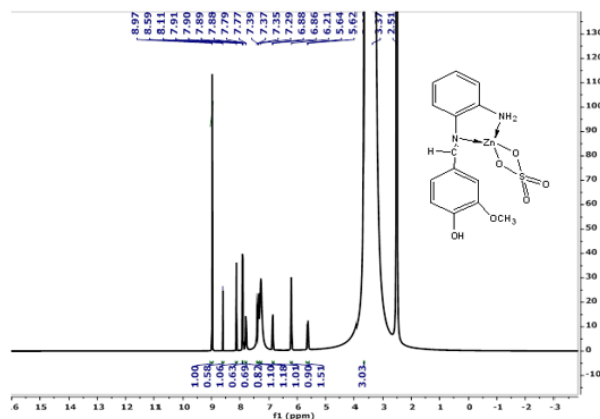


Figure 3. ^1H NMR spectrum of the Zn (II) complex

vent. The values of chemical shift of the ligands and metal complexes were showed in Table 3. The spectra displayed a multiple signal at $\delta(3.26-3.45)$ ppm is attributed to (m, OCH_3) and 3.47δ (s, $\text{Ar}-\text{OH}$) [15]. Two protons of ($\text{Ar}-\text{NH}_2$) amine group appears as a signal at ($\delta=4.90$ ppm), the azomethine proton ($\text{H}-\text{C}=\text{N}$) appears as a signal at ($\delta=8.15$ ppm) [16]. ^1H NMR spectrum of the Zn(II) compound, show signal due to protons bound to the azomethine group appeared at 8.97 ppm. This field shift indicates the deprotection of the azomethine proton upon coordination through the nitrogen atom of the azomethine group [17].

3.3 Magnetic measurement:

The magnetic moment for the Fe (II) and Co(II) complexes are $5.1 \mu\text{B}$, $1.4 \mu\text{B}$ respectively which were consistent with the tetrahedral geometry [18] [19]. The Ni(II) complex has a magnetic moment value of (0), due to the Ni (II) complex exhibit square geometry around Ni (II) [20] and 1.5 of Cu (II) complex [21] indicating the existence of unpaired electron

Table 5. The percentage of bacteria growth inhibition for complexes.

Complex No.	Chemical formula	Inhibition%	
		E .coli	K .pneumonia
1.	$[\text{Hg}(\text{L1})\text{Cl}_2]$	44.4	47.3
2.	$[\text{Zn}(\text{L1})\text{SO}_4]$	0	28.5
3.	$[\text{Zn}(\text{L2})\text{SO}_4]$	37.5	0
4.	$[\text{Hg}(\text{L2})\text{Cl}_2]$	44.4	50

,square planar geometry around Cu(II) metal [22]. Table 3.

3.4 Electronic spectra:

The spectra of the UV-Vis complexes were measured at room temperature in the region 190–900 nm. The electronic spectra of the prepared complexes displayed bands at (242–282), (280–302) nm referring to intra-ligand ($n-\pi^*$), ($\pi^*-\pi^*$) respectively [11]. Fe(II) complexes showed three band at (257–258, 284–298, 860–868) nm which due to ($n\rightarrow\pi^*$, $\pi^*\rightarrow\pi^*$, $^5E\rightarrow^2T_2$) respectively. The Co (II) complexes exhibited an absorption band at (602,645) nm region, which assigned to ($^4A_2(F)\rightarrow^4T_1(P)$) transition in tetrahedral configuration [23][24]. The absence of ($^4A_2(F)\rightarrow^2T_2(F)$) and ($^4A_2(F)\rightarrow^4T_1(F)$) transitions, are due to their low energy which is out of the range of the instrument. The electronic spectra of the prepared four coordinate nickel (II) complexes displayed bands at (540,627) nm, (351,394) nm referring to ($^1A_1g\rightarrow^1A_2g$), ($^1A_1g\rightarrow^1B_1g$), respectively and other bands referred to a ($n-\pi^*$), ($\pi-\pi^*$) which appeared at (242,260) nm, (280,300) nm region which, in turn the Square planar geometry [25]. Spectrum of green Cu (II) complex showed abroad bands at (587,598) nm which may be assigned to $^2E_g\rightarrow^2T_2g$ transition [26]. Table 4.

3.5 Anti-bacterial activity of some of the prepared complexes:

The efficacy of the prepared complexes was evaluated by two types of bacteria E .coli K .pneumonia. The prepared complexes showed different effects on bacteria. From previous studies that prove presence of a very large effect of the complexes of Schiff bases in the process of inhibiting bacterial growth [27][28]. The inhibitory ability of the complexes was tested on two types of bacteria, namely E. coli K. pneumonia at a concentration of 500 PPM. As the complex $[\text{Hg}(\text{L1})\text{Cl}_2]$ showed an inhibition rate of 44.4% for E. coli bacteria, while it showed an inhibition rate of 47.3% for K. pneumonia bacteria. As for the complex ratio $[\text{Zn}(\text{L1})\text{SO}_4]$, the inhibition rate was 0% for E. coli bacteria, while it showed an inhibition rate equal to 28.5% for K. pneumonia bacteria. As for the complex $[\text{Zn}(\text{L2})\text{SO}_4]$, the percentage of inhibition was shown 37.5 for E. coli bacteria, while the rate of inhibition of 0% for K. pneumonia was shown. As for the complex $[\text{Hg}(\text{L2})\text{Cl}_2]$, the rate of inhibition was 44.4% for E. coli while showing 50% inhibition of K. pneumonia Table 5.

4. Conclusions:

From the conductivity, magnetic and spectroscopy measurement refer to the L1 ligands was Coordinated as bidentate chelate ligands through the nitrogen atoms of the azomethine and amine groups with the metal ions, or through the two nitrogen atoms of the two azomethine groups in the L2 ligand with the metal ions. The Ni (II), Cu (II) complexes display square planar geometry around the metals while as a tetrahedral for others. Evaluation of the inhibitory effect of the prepared complexes on the growth of bacteria and the results displayed that the $[Hg(L_2)Cl_2]$ showed the highest activity against the pathogenic bacteria species.

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None

Declarations:

Conflict of interest The authors declare that they have no conflict of interest

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تحضير وتشخيص معقدات قواعد شف للفانيلين ودراسة تثبيط نمو البكتريا

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الخلاصة

في هذا البحث نستخدم طريقة الطول الأقصى للأثر حضرت معقدات لل $Co(II)$ ، $Ni(II)$ ، $Cu(II)$ ، $Zn(II)$ ، $Hg(II)$ ، من تفاعل املاح تلك الفلزات مع ليكاندات قواعد شف مشتقة من تكاثف الفانيلين مع اورثوفلين ثنائي امين بنسبة (1:1):(2:1) على التوالي شخصت الليكاندات والمعقدات بواسطة الطرق التحليلية . الفيزيائية والطيفية . بينت قياسات التحليل الدقيق للعناصر ان نسبة الفلز الى الليكاند هو 1:1 ظهرت قياسات التوصيلية الكهربائية المولارية ان المعقدات تكون غير الكتروليتية كما ان القياسات الطيفية والمغناطيسية ان معقدات النيكل الثنائي والنحاس الثنائي اتخذت شكل المربع المستوي وشكل رباعي السطوح لبقية المعقدات تم اختبار القدرة التثبيطية للنمو البكتيري للمجمعات المحضرة ضد نوعين من *E.colik.pneomonina* ووجد أن المعقدات المحضرة اختلفت في التأثير ، بعضها أظهر تأثير مناسب في تثبيط نمو مستعمرات النوعين.

الكلمات الدالة: قواعد شيف ، فانيلين ، معقدات ، اورثوفلين ثنائي امين ، تثبيط نمو البكتريا.