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RESEARCH ARTICLE

Synthesis, Diagnosis As Well as Biological Activity Studies of Some Metal Ion Complexes of Schiff Bases Derived from 4-Aminoantipyrine

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ABSTRACT

Polydintate Schiff base ligands L1 = orthohydroxy aniline, 4-aminoantipyrin-(N,N¯ benzilidene) and L2 = 5-Chloro-2-hydroxyaniline,4-aminoantipyrine-(N,N¯) benzilide obtained from condensation of benzil and 4-aminoantipyrine with 2-aminophenol or 2-amino 4-chlorophenol in (1:1:1) molar ratio for each component. Ligands employed to synthesis complexes in the general molecular formula [ML1Cl]Cl and [ML2]Cl2 respectively; M = Mn(II), Co(II), Ni(II) and Zn(II). To diagnose the ligands and the complexes the following techniques were adopted: UV-Visible FTIR and 1H-NMR spectroscopies, micro elemental analyses of metal ions and carbon, hydrogen and nitrogen, molar conductivity and magnetic susceptibility. Measurements obtained from the conductivity which was done with DMSO solution indicated that all the complexes are electrolytes with ratio (1:1) for L1 complexes and (1:2) for L2 complexes. The results of the measurements which used in this study showed coordination sites for L1 with central ion were through the phenolic oxygen atom, and two nitrogen atoms of the azomethin groups, L2 in addition to the previous donor atoms, the coordination is carried out by the oxygen atom of the terminal carbonyl group. All complexes are tetra-coordinate with (tetrahedral geometry). Evaluation of biological activity for the compounds tested against Gram (+) staphylococcus aurous and Gram (–) Klebsiella pneumonia.

Keywords: 4-aminoantipyrine, Bologicalactivity, Metal complex, Polydintate, Schiff base

Introduction

Schiff base ligands, since they were first prepared and studied in 1864[1] are still of great interest by many researcher.[2–4] This type of organic compound has wide applications in many different fields from medical,[5,6] pharmaceytical, industrial, agricultural and others.[7] Polydintate Schiff base is distinguished by its ability to easily attach with metal ions to form more stable coordination compounds, especially when nitrogen atom of C=N group and another donor atom share in coordination with the central atom,[8,9] The chemistry of this type of complex has attracted wide attention because of their biological activity, stability and potential application of many fields such as oxidation catalysis[10] electrochemistry etc.

Antipyrine Schiff base derivates have been extensively examined because they have shown wide range of applications in various fields like biological, analytical, therapeutical and are also used as precursors in the synthesis of bioactive compounds.[11] In recent years, many of researchers tended and interest to be published on transition metal complexes derived from antipyrine Schiff base.[12,13]

In the present study we report the synthesis of polydintate Schiff bases (L^1 and L^2) derived from 4-aminoantipyrine and benzil with 2-aminophenol or 2-amino 4-chlorophenol and describe their

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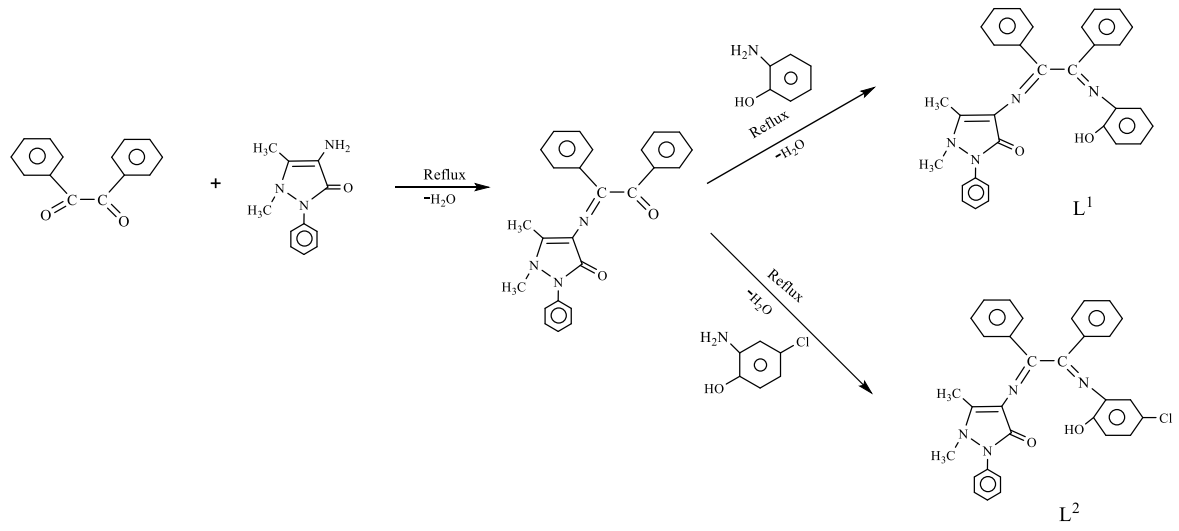


Fig. 1. Preparation equation of the ligands.

coordination behavior towards Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) as well as screening of their antibacterial activities against *Staphylococcus aureus* and *Klebsiella pneumonia* depending on the method of diffusion through the paper disk.

Materials and methods

All chemicals used in this work Viz; Benzil, 4-aminoantipyrine, 2-aminophenol, 2-amino 4-chlorophenol and metal (II) chlorid were chemically pure and equired from (fluka and sigma Aldrich), The elemental analysis (C.H.N.) was recorded on an Elementer Vario Micro Cube. The infrared spectra were recorded on shimadzu FTTR–8300 spectrophotometer using the KBr pellet technique. the UV–Vis spectra were obtained in DMSO on a Perkin Elmer Lambda 2-UV/Vis spectrophotometer the H-NMR spectra were recorded on Broker Advance DPX-250 UHz spectrometer with TUS as internal standard. In DMSO-d^6, ^{1}H-NMR spectra were recorded by using Bruker DPX-400 model instrument using DMSO as solvent and manual version 1.0. The magnetic susceptibility for the complexes is determined using Bruker BM6 instrument. The metal content was estimated by atomic absorption spectrometry using AA240 FS Varian instrument, A Jenway 4070 conductivity meters was used to measure the conductivity of all prepared complexes and were carried out in DMSO (10^{-3}M) solution at laboratory temperature.

Preparation of the ligands (L^1 and L^2)

Schiff base ligands were prepared depending on the reported methods[14,15] by reacting equimolar amounts of the starting materials. (2.01 gm, 0.01 mole) of benzil dissolved in (10 ml) methanol it was added slowly to 4-aminoantipyrene dissolved in methanol (2.03 gm, 0.01 mole) with the addition of drops of glacialacetic acid refluxed for 2 h. Then (1.09 gm, 0.01 mole) of 2-aminophenol or (1.43 gm, 0.01 mole) of 2-amino 4-chlorophenol each one dissolved in (10 ml) MeOH was added slowly to the reaction mixture, the resulting colored mixture was stirring with refluxed for 6 h, and leave to cool at laboratory temperature filtered well and washed several times with methanol and ether, and then dried for 24 h under vacuum, Fig. 1.

Preparation of complexes

Co (II), Ni(II), Cu(II) and Zn(II) complexes of L^1 and L^2 were prepared by the addition of 0.01 mole of the appropriate metal chloride hydrates dissolved in (25 ml) of ethanol to a hot mixture of ethanol solution (25 ml) containing 0.01 mole of the required Schiff base ligand. The mixture was then refluxed for 3h. The precipitated solids were filtered well and washed several times with ethanol and then dried under a vacuum.

Antibacterial activity

The antibacterial activity of the compounds was tested against Gram (+) bacteria *Staphylococcus aureus* and Gram (–) bacteria *Klebsiella-Pneumoniae* by using the agar diffusion method.

The test solution was prepared by dissolving 10 mg of the prepared materials in (1 ml) of DMSO. A (6 mm) diameter blank paper disease was cultivated

Table 1. Analytical data and physical properties of compounds.

NO	Compounds	Color	m.p. (°C)	Analysis Calc. (founds)% C	H	N	M% Calc. (found)	Δ_m ohm^{-1}. cm^2.mol^{-1}
L^1	$C_{31}H_{26}N_4O_2$	Dark yellow	130	76.54 (76.24)	5.34 (5.25)	11.52 (10.76)	–	–
1	$[ML^1Cl]Cl$	Light gray	258	68.77 (67.98)	4.80 (4.32)	10.35 (10.13)	10.15 (9.93)	36.1
2	$[CoL^1Cl]Cl$	Brown	245	68.26 (67.80)	4.77 (4.52)	10.27 (9.82)	10.81 (10.53)	40.6
3	$[NiL^1Cl]Cl$	Light brown	147	68.29 (67.55)	4.77 (4.23)	10.28 (9.97)	10.77 (10.23)	34.4
4	$[CuL^1Cl]Cl$	Dark brown	264	67.69 (67.12)	4.73 (3.91)	10.19 (6.38)	11.56 (10.95)	39.9
5	$[ZnL^1Cl]Cl$	Yellow	210	67.46 (67.05)	4.71 (4.62)	10.15 (9.82)	11.85 (11.27)	38.1
L^2	$C_{31}H_{25}N_4O_2Cl$	Dark orange	174	71.46 (71.82)	4.80 (5.00)	10.75 (10.26)		
6	$[ML^2]Cl_2$	Light orang	112	64.64 (64.25)	4.34 (4.15)	9.73 (9.21)	9.54 (9.27)	69.2
7	$[CoL^2]Cl_2$	Light brown	110	64.20 (63.88)	4.31 (4.17)	9.66 (9.19)	10.17 (9.82)	73.7
8	$[NiL^2]Cl_2$	Light brown	135	64.22 (46.19)	4.31 (5.14)	9.66 (6.55)	10.13 (9.69)	70.11
9	$[CuL^2]Cl_2$	Dark brown	104	63.69 (63.20)	4.28 (3.97)	9.58 (9.13)	10.87 (10.56)	82.00
10	$[ZnL^2]Cl_2$	Gray	216	63.49 (63.23)	4.26 (3.94)	9.55 (9.07)	11.16 (10.85)	68.11

of 37C° for 24 h. The diameter of the inhibition zone in (mm) was determined and the resulting activity was estimated. A Standard disk of Ciprofloxacin used as a positive control. [16,17]

Results and discussion

The synthesized Schiff base ligands (L^1 and L^2) scheme 1 forms 10 stable complexes with Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) in (1:1) molar ratio (M:L). All the complexes were soluble in most organic solvents and stable in dry air and their melting points ranged from 104 to 258°C. The results obtained from the analytical as well as the physical traits of each compound are recorded in Table 1. The obtained analysis results for the prepared complexes fit in with the suggested formula $[ML^1Cl]Cl$ and $[ML^2]Cl_2$. The conductivity results in DMSO showed that all complexes are electrolytic with (1:1) and (1:2) ratio for L^1 complexes and L^2 complexes respectively.

The suggested structures of the prepared complexes are shown in Fig. 2.

^{1}H-NMR spectra

The ^{1}H-NMR spectra of L^1 and L^2 were registered in DMSO–d^6 solvent and the ^{1}H-NMR assignments for the compounds are presented in Table 2 and Fig. 3. [2,3]

IR-spectra

Important peaks for all compounds have been diagnosed by IR spectra. The band at 1591 and 1589 cm^{-1} in IR spectra of L^1 and L^2 supported the formation of ν(C=N) group, [18,19] this band shifted to higher frequency in the spectrum complexes, [20,21] which indicates the participation of the nitrogen atom of the azomethine group in bonding.

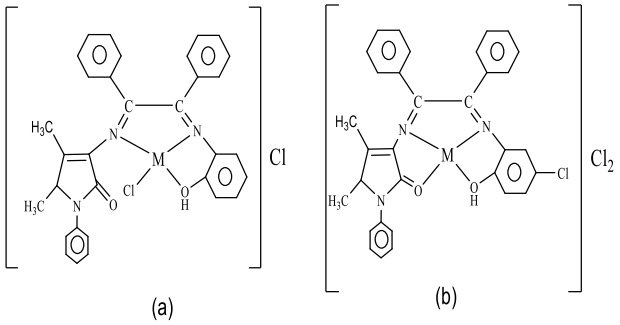


Fig. 2. Suggested structure of (a): $[ML^1Cl]Cl$ complexes (b): $[ML^2]Cl_2$ complexes. M = Mn(II), Co(II), Ni(II), Cu(II), Zn(II).

Table 2. The ^{1}H-NMR data of the ligands.

ς(ppm)	Assignment
L^1	
6.53–7.71	(m,19H, ArH)
3.14	(S,3H, C-CH_3)
2.47	(S,3H, N-CH_3)
8.91	(bs, 1H, OH)
L^2	
7.15–7.71	(m,19H, ArH)
3.14	(S,3H, C-CH_3)
2.47	(S,3H, N-CH_3)
8.91	(bs, 1H, OH)

S = singlets, M = multiplets, Bs = broad signal.

It is clear from the IR spectrum of the free ligand that the bands at 3375 and 3379 cm^{-1} due to phenolic ν (OH) group of L^1 and L^2 respectively this band shift to higher frequencies in the spectum of the complexes this indicates the coordination of phenolic oxygen to central ion without deprotonating, [22–25] another important band appeared at 1664 and 1662 cm^{-1} due to ν (C=O) of cyclic keton. In L^1 complexes(1–5) this band remains almost unchanged on complexation, which indicates that the carbonyl oxygen atom of

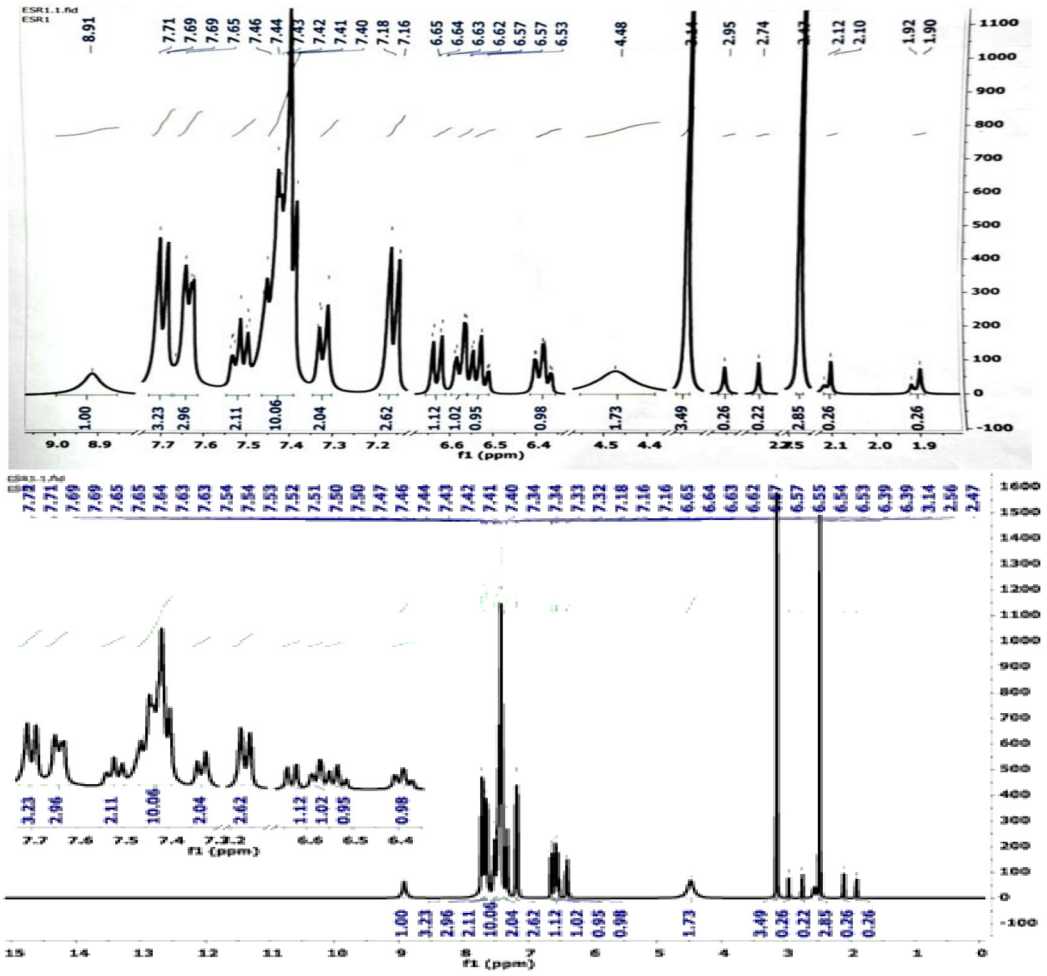


Fig. 3. ^{1}H-NMR spectra of L^1.

Table 3. IR bands of L^1, L^2 and their complexes.

NO.	Compounds	ν (C=N)	ν (O-H)	ν (C=O)	ν(M-O)	ν(M-N)
L^1		1591	3375	1664		
1	$[MnL^1Cl]Cl$	1600	3396	1664	586	466
2	$[CoL^1Cl]Cl$	1598	3383	1666	505	440
3	$[NiL^1Cl]Cl$	1599	3392	1662	584	430
4	$[CuL^1Cl]Cl$	1599	3390	1664	507	461
5	$[ZnL^1Cl]Cl$	1602	3387	1662	510	445
L^2		1589	3379	1662		
6	$[MnL^2]Cl_2$	1601	3433	1676	503	440
7	$[CoL^2]Cl_2$	1600	3391	1675	549	441
8	$[NiL^2]Cl_2$	1599	3394	1672	586	440
9	$[CuL^2]Cl_2$	1605	3406	1675	580	420
10	$[ZnL^2]Cl_2$	1601	3393	1674	560	435

cyclic ketone is not involved in coordination in these complexes.

While in L^2 complexes (6–10) this band shifted to higher frequencies indicating that the carbonyl oxygen atom of cyclic keton considered one of the coordinating sites,[23] on the other hand on the spectrum of all complexes a new band was appeared at 503–586 cm^{-1} and 420–466 cm^{-1} due toν(M-O) and ν(M-N)stretching vibration.[25] ν (M-Cl) band is not registered because this band is below the spectrophotometer limits. This discussion suggested that L^1 coordinates to metal in tridentate fashion NNO while L^2 is tetradentate fashion NNOO. The results are mentioned in the following Table 3 and shown in Fig. 4.

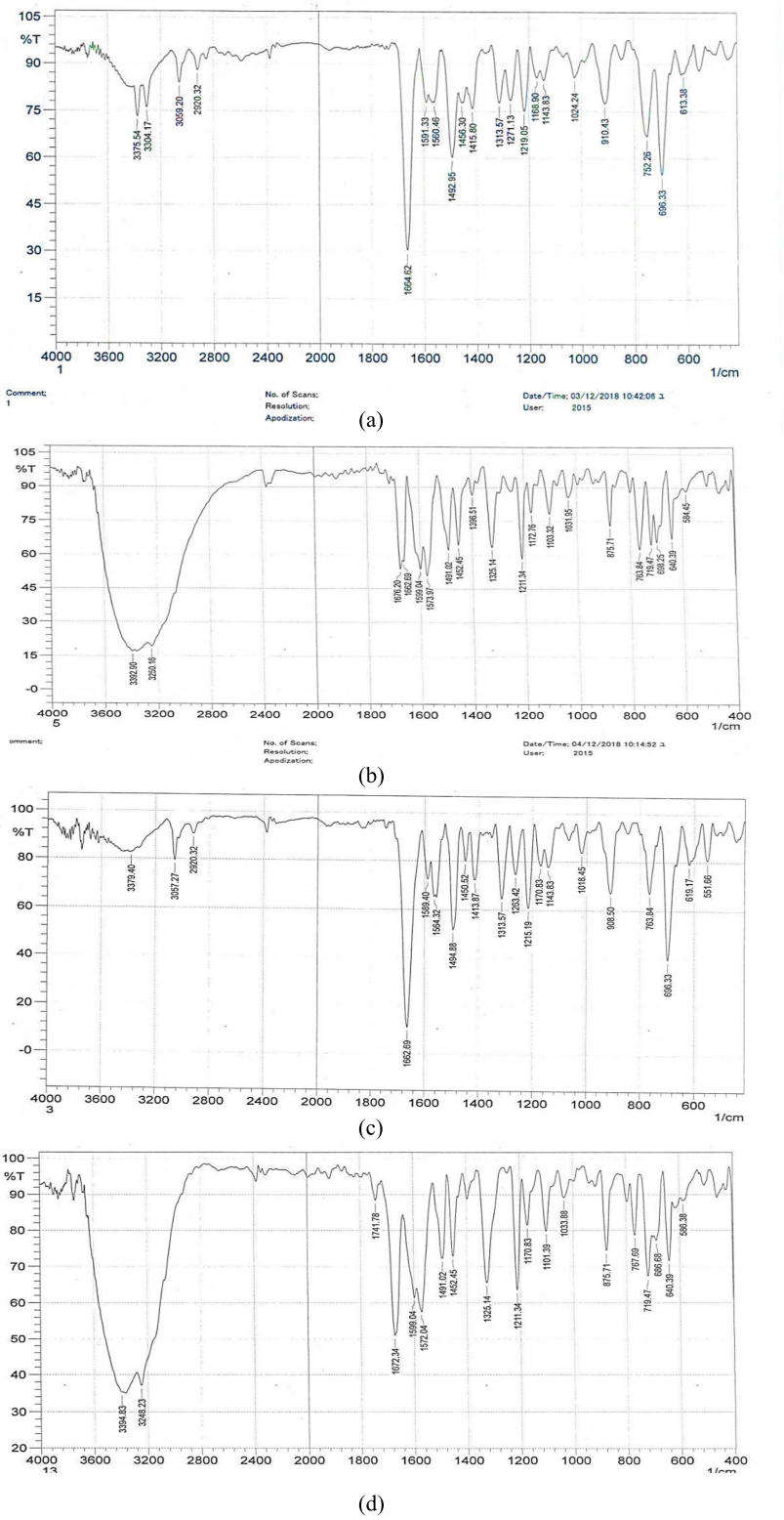


Fig. 4. The IR spectrum of (a) L^1, (b) $[NiL^1Cl]Cl$, (c) L^2, (d) $[NiL^2]Cl_2$.

Table 4. Results of magnetic moment and UV-vis.

No	Compounds	μ_{eff} B.M.	Band maxima λ cm^{-1}	Structure	Assigned transition
L^1	$C_{31}H_{26}N_4O_2$		39062, 31250		
1	$[ML^1Cl]Cl$	5.7	28248	Td	C.T
2	$[CoL^1ClCl$	4.15	14857, 16826, 23255	Td	$^4A_{2(F)}\rightarrow{}^4T_{1(P)}$, C.T
3	$[NiL^1Cl]Cl$	3.91	14772, 26231	Td	$^3T_{1(F)}\rightarrow{}^3T_{1(P)}$, C.T
4	$[CuL^1Cl]Cl$	2.44	13973, 27781	Td	$^2T_2\rightarrow{}^2E$, C.T
5	$[ZnL^1Cl]Cl$	Dia	28255	Td	C.T
L^2	$C_{31}H_{25}N_4O_2Cl$		45871, 38759		
6	$[ML^2]Cl_2$	6.10	29673	Td	C.T
7	$[CoL^2]Cl_2$	4.33	15083, 16477, 20876	Td	$^4A_{2(F)}\rightarrow{}^4T_{1(P)}$, C.T
8	$[NiL^2]Cl_2$	3.77	14084,23277	Td	$^3T_{1(F)}\rightarrow{}^3T_{1(P)}$, C.T
9	$[CuL^2]Cl_2$	2.59	14992, 24277	Td	$^2T_2\rightarrow{}^2E$, C.T
10	$[ZnL^2]Cl_2$	Dia	28385	Td	C.T

Table 5. The bacterial examination results of L^1, L^2 and their complexes.

No.	Compound	Staphylococcus *aureus* (mm)	Klebsiella pneumonia (mm)
L^1	$C_{31}H_{26}N_4O_2$	20	2
1	$[ML^1Cl]Cl$	18	4
2	$[CoL^1Cl]Cl$	13	8
3	$[NiL^1Cl]Cl$	2	4
4	$[CuL^1Cl]Cl$	17	2
L^2	$C_{31}H_{25}N_4O_2Cl$	2	2
6	$[ML^2]Cl_2$	2	2
7	$[CoL^2]Cl_2$	13	9
8	$[NiL^2]Cl_2$	11	11
9	$[CuL^2]Cl_2$	10	13
Control	Ciprofloxacin	28	28

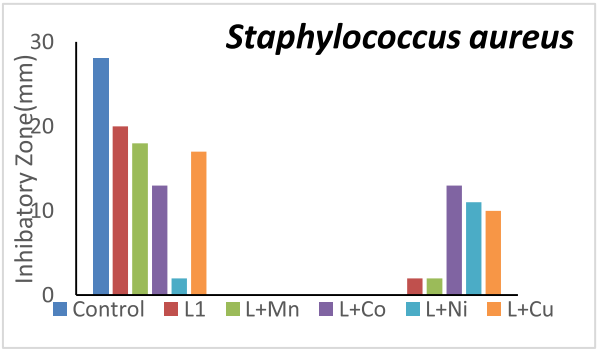
Ultraviolet visible (UV) and magnetic properties

The electronic spectra and magnetic properties are very useful in elucidating structure, the results of both measurements are specified in Table 4. The electronic spectra of the ligands in DMSO show the aromatic intense bands at 39062 cm^{-1} and 45871 cm^{-1} assigned to ($\pi\rightarrow\pi^*$) due to transitions relating molecular orbitals situated on the benzene ring of the ligand group and at 31250 and 38759 cm^{-1} attributed to the (n→π^*) transition due to azomethine groups, [26,27] which shifted in the spectrum of the complexes which confirms the coordination of ligands with the central ion. [28–30] In the spectrum of all complexes new bands appeared at the region (20876–29673) cm^{-1} may be attributed to LMCT transition. [31,32]

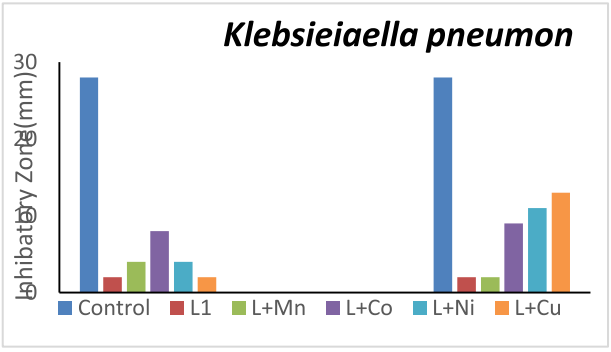
At room temperature Bohr magneto values of Mn(II) complexes (1 and 6) were (5.71 and 6.10) B.M. supports the tetrahedral arrangement resulting from the presence of the five unpaired electrons which confirms high spin state. The electronic spectra of Mn (II) complexes did not give clear absorption which can indicate to d-d transition. The d-d transitions in the Mn(II) tetrahedral environment is spin-forbidden but no longer parity forbidden, these transitions are ~100times stronger, therefore it is possible to appoint the structure of Mn(II) complexes depending on the results of other measurements namely. [33–35]

The Bohr magneto values of Co (II) complexes (2 and 7) at (4.15 and 4.33) B.M. which refers to the tetrahedral structure of these complexes, their electronic spectra exhibit one transition ν_3 $^4A_{2(F)}\rightarrow{}^4T_{1(P)}$ consisting of two humps located at 14857 and 15083 cm^{-1} and 16826 and 16477 cm^{-1} respectively, the fission of this band which caused by Jahn-Teller type of the tetrahedral structure in the excited state, [36–38] the other two bands ν_1 and ν_2 are below the spectrophotometer limits.

The Bohr magneto values of Ni(II) complexes (3 and 8) were found to be (3.91 and 3.77) B.M. and its electronic spectrum also recorded a clear absorption band ν_3 at (14772 and 14084) cm^{-1} respectively due to $^3T_{1(F)}\rightarrow{}^3T_{1(P)}$ transition in tetrahedral geometry, [39–41] the other two bands ν_1 and ν_2 are located in the lower part of the electronic spectrum which is below the scale limits, the obtained magnetic moment values of Cu (II) complexes (4 and 9) found to be (2.44 and 2.59) B.M. this confirms the existence of unpaired electron in the electronic spectrum of these complexes a wide band was seen at 13973 and



(a) Staphylococcus aureus



(b) Klebsieiaella pneumon

Fig. 5. Antibacterial activity of compounds agents (a) *Staphylococcus aureus* (b) *Klebsieiaella pneumon.*

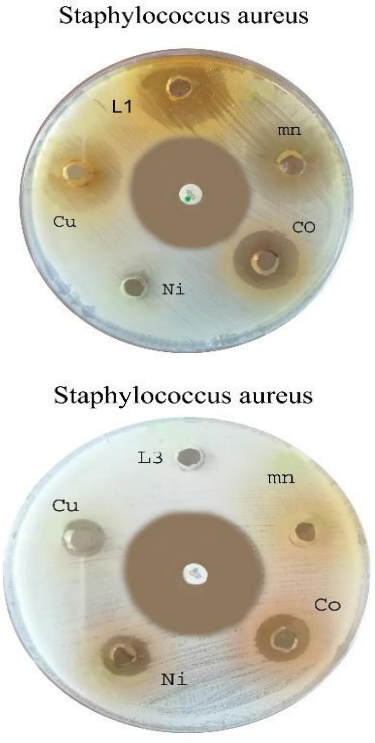


Fig. 6. Antibacterial activity of the compounds.

14992 cm^{-1} respectively attributed to $^2T_2 \rightarrow {}^2E$ transition which suggests the tetrahedral structure of these complexes. [42]

The electronic spectra of Zn(II) complexes (5 and 10) show only a band at 28255 and 28385 cm^{-1} respectively which represents the charge transfer spectra.

The Zn(II) complexes are expected to be diamagnetic and their geometry is most probably similar to the Co(II), Ni(II) and Cu(II) complexes based on the rest of results for the other measurements in particular the results of metal content and IR spectra. [43,44]

Antibacterial activity

The evaluation of biological activity for the ligands and complexes with 1×10^{-3} M concentrations was studied against selected types of G(+) bacteria *Staphylococcus aureus* and G(–) bacteria *Klebsiella pneumonia*. The obtained results have been tabulated and shown in Table 5, Figs. 5 and 6 and compared with a standard drug (Ciprofloxacin). The evaluation showed that the ligands and their complexes have an activity described as less than effective of ciprofloxacin against the bacteria used in this research.

Conclusion

New complexes of Schiff base ligands were prepared and diagnosed by various physical and chemical methods. The data revealed that (L^1) ton in [ML'C1] Cl acts as a polydentate ligand and coordinated with the central atoms through the oxygen atom of Phenol group and nitrogen atoms of the azomethin groups, (L^2) in [M L^2] Cl_2 coordinated through oxygen atom of terminal carbonyl group in addition to the atoms listed above and all complexes classified as mononuclear with tetrahedral geometry. The ligands and their complexes were examined against both Gram (+) and Gram (–) bacteria, the test results were compared with the standard drugs and showed that the ligands and complexes have an activity described as less than the drug.

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Author's declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for republication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- No potentially identified images or data are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Mosul.

Author's contributions statements

A.M.M. Carried out the experiment and analysed all parameters, I.A.S. Collected the samples and taking measurements, K.H.N. Conceived the idea, supervised the project and wrote the manuscript.

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تحضير، تشخيص فضلاً عن دراسة الفعالية البايولوجية لمعقدات بعض من ايونات الفلزات مع قواعد شيف المشتقة من 4 - امينو انتي بايرين

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الخلاصة

قواعد شيف متعددة السن ذوات الصيغة L^1= اورثو هايدروكسي انيلين -٤ - امينو انتبيرين (N′,N بنزيليدين) تم تحضيرها من تكثيف البنزايل و٤-امينو انتيبرين مع ٢-امينو فينول -١،٢ امينو ٤ كلورو فينول بنسبة مولية (١:١:١) لكل مكون. واستخدمت هذه الليكاندات لتحضير المعقدات ذوات الصيغة $[ML^1\ Cl]Cl$ و $[ML^2]Cl_2$ حيث ان M = Mn(II), Co(II), Ni(II), Zn(II). تم استخدام التقنيات التالية لتشخيص الليكاندات والمعقدات: مطيافية الاشعة فوق البنفسجية والاشعة تحت الحمراء - المرئية وطيف الرنين النووي المغناطيسي للبروتون ^1HNMR والتحليل الدقيق لايونات العناصر والكاربون والهيدروجين والنتروجين بالاضافة الى التوصيلية الكهربائية المولارية وقياسات الحساسية المغناطيسية. دلت القياسات التي تم الحصول عليها من التوصيلية الكهربائية المولارية والتي تم اجراؤها بمحلول DMSO ان جميع معقدات (L^1) كانت الكتروليتية بنسبة (١:١) بينما معقدات (L^2) كانت الكتروليتية بنسبة (1:2). كما اظهرت نتائج القياسات المستخدمة في هذه الدراسة ان مواقع التناسق لليكاند (L^1) مع الأيون المركزي كانت من خلال ذرة الاوكسجين الفينولية وذرتين نيتروجين لمجموعتي الازوميثين اما بالنسبة لـ (L^2) فبالاضافة للذرات المانحة المذكورة سابقا يتم التناسق مع ذرة الاوكسجين لمجموعة الكاربونيل الطرفية. جميع المعقدات اظهرت التناسق الرباعي بشكل هندسي رباعي السطوح ايضا تمت دراسة الفعالية البايولوجية للمركبات المحضرة ضد البكتريا موجبة الكرام (+)*Staphylococcus aurous* وسالبة الكرام (-) *Klebsiella pneumonia*.

الكلمات المفتاحية: 4-aminoantipyrine، النشاط البيولوجي، المركب المعدني، Polydintate، قاعدة شيف.