Sys Rev Pharm 2021;12(1):107-115
A multifaceted review journal in the field of pharmacy

CUPPER (II) AND MERCURY (II) COMPLEXES WITH SCHIFF BASE LIGANDS FROM BENZIDINE WITH ISATIN AND BENZOINE: SYNTHESIS, SPECTRAL CHARACTERIZATION, THERMAL STUDIES AND BIOLOGICAL ACTIVITIES

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ABSTRACT

The antimicrobial study, characterization and synthesis of cupper (II) and mercury (II) complexes from the Schiff base ligands (L_1) as well as (L_2) were indicated in the presented work. Also, the ligand (L_1),[2,2-(biphenyl-4,4diylbis(azan-1-yl-1-ylidene))bis(1,2-diphenylethanol)]is acquired via the condensation benzidine and benzoine, whereas the ligand (L_2), [3,3-(biphenyl-4,4-diylbis(azan-1yl-1-ylidene))diindolin-2-one] was acquired via the condensation of the benzidine and isatin. Complexes and ligands have been specified via Infrared spectra, vibration electronic, molar conductance, NMR spectrum as well as other studies. In addition, such complexes molar conductance is conveying their ionic character, while the spectral data is showing that the composition related to metal complexes as $[Cu_2(L_1)(L_2)]Cl_2$ and $[Hg_2(L_1)(L_2)(H_2O)_4]Cl_2$, ($L_1$ and L_2 were schiff base ligands).Antifungal and antibacterial activities of such complexes of cupper (II) and mercury (II)with Schiff bases were done via disc diffusion approach. Lastly, the results of biological activities and characterization are provided in the presented work.

Keywords: characterization, benzidine, biological activity, copper, structure

1. INTRODUCTION

The Schiff base with powerful doner atoms like carboxylate oxygen and imine nitrogen were essential in structural richness, catalysis and a lot of biological applications [1]. Due to their excellent solubility in common solvents and elevated stability of coordination compounds, the Schiff bases were utilized as ligands. Often, the π-system in Schiff base imposing geometrical constrictions as well as affecting the electronic structure. Also, the thermo-chemical characteristics of Schiff bases were the focus of many researchers because of their capability in coordinating metal ions, act as tetradentate or bidentate ligands in the chelates of the metal, including N_2O_2 or NO Schiff base donor atom sets, such derivatives of the Schiff base metals are of high importance as a result of their part as model complexes to the biological systems, adding to knowledge which is related to their behavior and structure [2,3]. Isatin can be defined as one of the synthetically versatile substrates, in which it might be utilized for synthesizing a lot of heterocyclic compounds, like quinolones and indoles, also it is considered as one of the raw materials for the drug synthesis. Because of its cis α-dicarbonyl moiety, Isatinis one of the essential substrates to synthesize metal complexes. Deprotonated or alone, isatin might be located in the mammalian tissues [4], stemming from the interests in pharmacological and biological characteristics of isatin derivatives [5-8]. Benzidineis utilized as amination reactant for Schiff base's synthetic. It is anticipated that interactions of the compounds of the carbonyl with the benzidine might continue with participation of the two amino groups, leading to cross-linking related to the 2 molecules of substrate [9,10]. Copper is one of the critical follow components existing in the human body, plants and creatures. Not with standing, high measures of this component can bring about genuine medical issues, including disturbance of nose and throat, queasiness, heaving, and looseness of the bowels and additionally harm to the liver and kidney [11]. In the perspective of the natural and ecological significance an impressive consideration has been centered around recognition of Cu (II) ion [12]. Previously, resulted are reported for structural study regarding a few of the Schiff bases taken from diamines, β-diketones and their complexes [13]. In the presented study, new Schiff base is synthesized from reacted isatin with benzidine and benzoine with benzidine and its complexes with Cu (II) and Hg (II) ions.

2. Experimental

2.1 Reagents and Materials:

All the commercially available reagents have been utilized with no more purification and bought from Merck Chemical Company in high-purity.

2.2 Instrumentation:

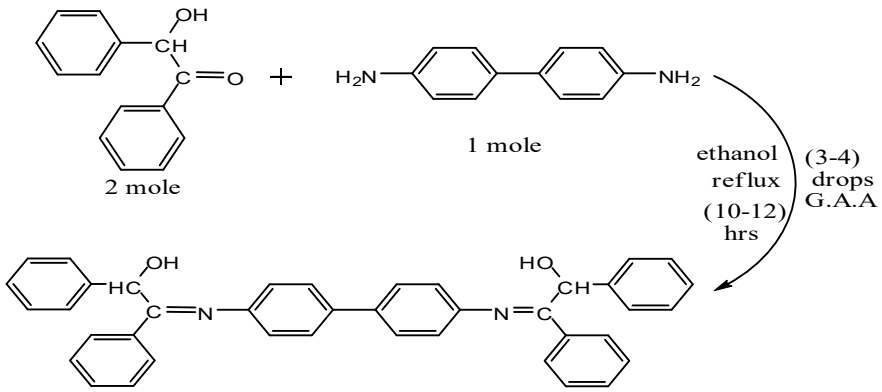
Infrared spectra for the prepared compounds have been recorded as KBr discs utilizing FTIR tests Shimadzu (FT-IR) 8300 series spectrophotometer in range between (4000 and 400cm^{-1}), while the electronic spectra have been evaluated in region between 200 and 1100 nm for 10^{-1} M solution in the DMSO at a temperature of 25 Celsius utilizing Shimazdzu 160-A Spectrophotometer with 1.000 ≠0.001 cm matched quart Z cell. Elemental micro analysis has been performed on a (C.H.N) analyzer from (Elemental micro analyses, Euro

Vector, model EA3000 origin). ^{1}H-NMR spectra related to the prepared ligands are recorded in DMSO-d_6 using model: NMR Ready pro60 MHz, Origin: Canada and reported in ppm(s). Also, the electrical conductivity measurements regarding complexes are recorded at a temperature of 25 Celsius for 10^{-3} M solution related to sample in DMSO utilizing Eutech-150 conductivity meter. In addition, the magnetic susceptibility measurement is acquired via balance Magnetic model of susceptibility MSBMKI, while the metal contents related to all complexes are specified via atomic absorption approach via utilizing Shimadzu (AA680G). Furthermore, the TG is acquired utilizing apparatus with the type STAPT1000 NSEIS as temperature in range between 30 and 600 CO, measurement acquired in central laboratory. Melting points are acquired utilizing (Stuart melting point Apparatus) type Dig melt (MSRS).

2.3 Components Synthesis

2.3.1 Synthesis of the ligand (2,2-(biphenyl-4,4diylbis(azan-1-yl-1-ylidene)) bis(1,2-diphenylethanol) (L_1)

Benzoine (4.24 gm,0.02 mole) was dissolved in ethanol (25ml) and stirred at room temperature to this stirring solution of benzidine (1.84gm, 0.01mole) in ethanol (25ml) was added. The Schiff base has been prepared from reaction (4.24gm, 0.02mole) benzoine, with benzidine (0.184gm, 0.01mole) in ethanol absolute of (25ml) and 3 to 4 drops of the glacial acetic acid, such mix is refluxed on a water bath in (10 to 12) hours at a temperature of 50 Celsius, while the yellow-colored solid product separated has been filtered as well as washed with anhydrous ethanol [14]. Yield =87%, M. P=110ºC. Scheme (1) is showing the synthesis route of ligand (L_1).

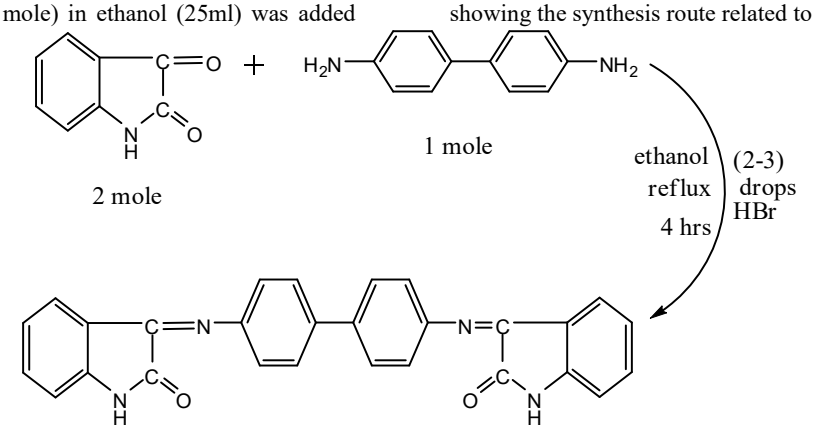


2,2'-(biphenyl-4,4'-diylbis(azan-1-yl-1-ylidene))bis(1,2-diphenylethanol)

Scheme 1: Structure of Ligand (2,2-(biphenyl-4,4-diylbis(azan-1yl-1-ylidene)) bis(1,2-diphenylethanol) (L_1)

2.3.2 Synthesis of ligand (3,3-(biphenyl-4,4-diylbis(azan-1-yl-1-ylidene)) diindolin-2-one) (L_2)

Isatin(2.94 gm,0.02 mole) was dissolved in ethanol (25ml) and stirred at room temperature to this stirring solution of benzidine (1.84gm, 0.01mole) in ethanol (25ml) was added and (2-3)drops of 48% HBr. Reaction mixture has been refluxed for approximately (4) hrs. The orange-colored solid product separated is filtered and washed with anhydrous ethanol [15]. Yield =85%, M. P=182ºC. Scheme (2) is showing the synthesis route related to ligand (L_2).



3,3'-(biphenyl-4,4'-diylbis(azan-1-yl-1-ylidene))diindolin-2-one

Scheme 2: Structure of Ligand (3,3-(biphenyl-4,4-diylbis(azan-1-yl-1ylidene)) di-indolin-2-one) (L_2)

2.3.3 Synthesis of Cu (II) complex (L_1L_2).

Metal cupper salt (0.341gm,0.002 mole) is dissolved in the hot ethanol (25ml) as well as added slowly to ligand L_1 (0,572gm,0.001mole) and ligand L_2 (0.0442gm,0.002mole) in hot ethanol and added (0.08gm,0.002mole) of NaOH. Reaction mixture was refluxed (4hrs) at 60ºC. Furthermore, the precipitate is created, filtered, also re-crystallized from the methanol.

2.3.3 Synthesis of Hg (II) complex (L_1L_2).

Metal mercury salt (0.542 gm,0.002 mole) is dissolved in hot ethanol (25ml) as well as added slowly to ligand L_1 (0,572gm,0.001mole) and ligand L_2 (0.0442gm,0.002mole) in hot ethanol and added (0.08gm,0.002mole) of NaOH. Reaction mixture was refluxed (4hrs) at 60 ºC. Furthermore, the precipitate is created, filtered, also re-crystallized from the methanol.

The elemental analyses and physical properties of the ligands

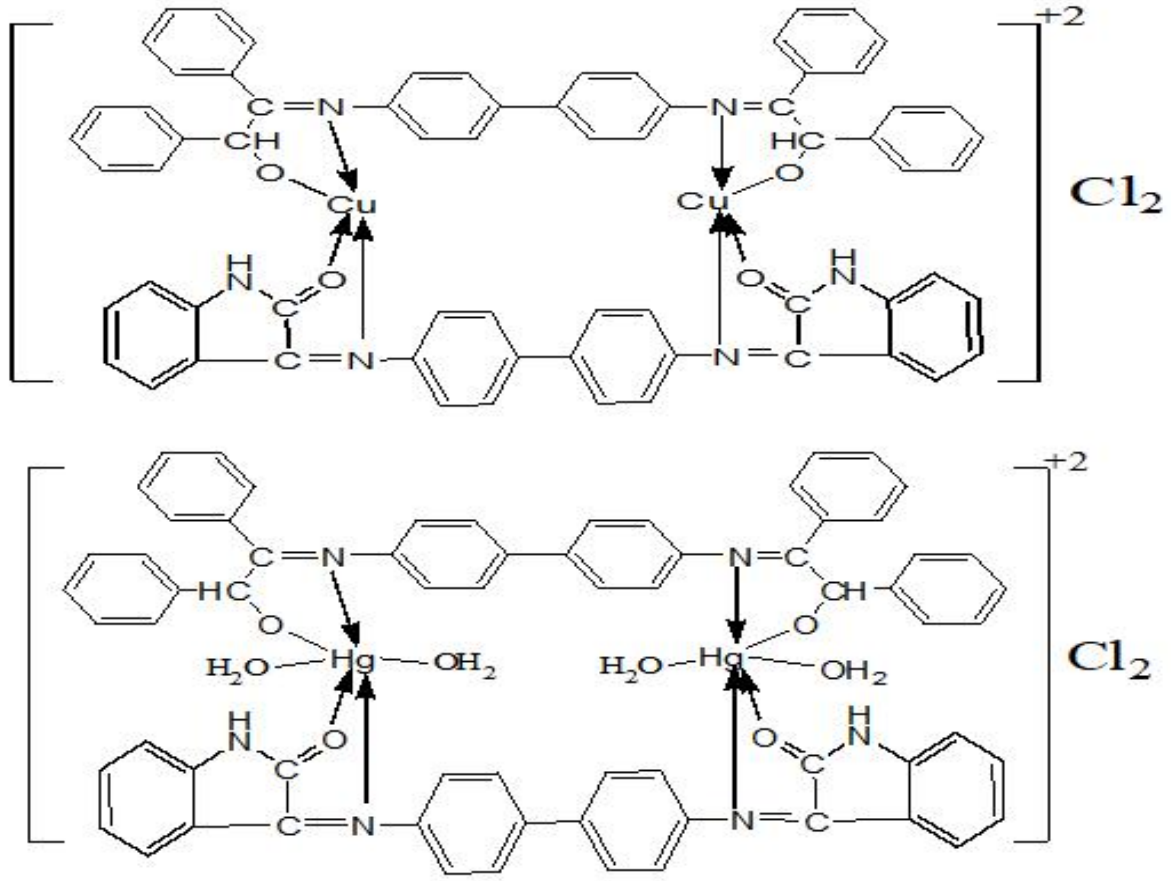
along with their complexes are given in table1 below:

Table 1. Elemental analyses and physical characteristics of ligands (L_1, L_2) as well as their complexes

Elemental Analyses Found (Calc.) %(calculated)					Color	Yield %	M.P Dec.	Empirical Formula (formula wt.)	Compound
Cl	M	N	H	C					
-	-	4.19 (4.89)	4.53 (5.63)	83.34 (83.89)	yellow	87	110	C40H32N2O2 572.69	L1
-	-	12.34 (12.66)	4.25 (4.10)	76.19 (76.01)	orange	85	182	C28H18N4O2 442.47	L2
5.46 (5.85)	10.23 (10.49)	6.75 (6.94)	3.63 (3.99)	67.84 (67.43)	Drak green	62	254 dec.	C68H48Cu2Cl2 N6O4 1211.14	[Cu2(L1) (L2)] Cl2
4.33 (4.55)	25.41 (25.76)	5.65 (5.40)	4.63 (3.62)	52.61 (52.45)	Masturd	68	269 dec.	C68H56Hg2Cl2 N6O81557.29	[Hg2(L1) (L2) (H2O)4] Cl2

M.P=melting point; Dec.= decomposition; Calc.=calculated

Figure1: Suggested Hg (II) and Cu (II) complexes structure



3. Results and Discussion

The analytical data and the physical characteristics which are related to Schiff bases as well as their complexes are provided in table (1), while Schiff base ligands as well as their complexes have been stable at 25 Celsius. Furthermore, complexes have been soluble in most common organic solvents and insoluble in ether.

3.1 IR spectra

IR spectrum regarding free ligand L_1(Figure 2) is showing a band at 3352 cm^{-1}which is due to (OH) group and showing strong band identified at 1612cm^{-1} that was allocated to (C=N_1), such band was shifted to low wave number side(ΔV= 24cm^{-1})in a case of Cu(II)complex as well as(ΔV= 23cm^{-1}) in a case of Hg(II) complex (Figure 4), indicating the participation regarding azomethine group in the coordination to metal ions via lone electrons'pair on nitrogen[16]. Also involving the (OH) group in the bonding with the metal ions might be identified via (C-O) band that was shifted to the high wave number side(ΔV=22 cm^{-1}) in the case of Cu(II) complex as well as(ΔV= 26cm^{-1}) in a case of Hg(II) complex, such band was disappeared in all complexes specifying the coordination of oxygen atom through deprotonation [17], such fact was provided via the shift of (C–O) vibrations in ligand, between 1261cm^{-1} and 1265 cm^{-1} to high frequencies in complexes Cu(II) complex as well as Hg(II) complex.

IR spectrum related to L_2 (Figure 3) showing new band at 1643cm^{-1}which correspond to (C=N_2), such band was shifted to low wave number side ΔV= 21cm^{-1} in a case of Cu(II) complex, also ΔV= 16 cm^{-1}in the case of Hg(II) complex, indicating the participation regarding groups of the azomethine in the coordination to metal ions via lone pair of electrons on nitrogen[16].Moreover, the strong band identified in ligand L_2 at 1708 cm^{-1}was allocated to (C=O) amide carbonyl group. With regard to complexes, such band was shifted to high wave number 1720 cm^{-1} and 1732 cm^{-1}in complexes Cu(II) and Hg(II), specifying the coordination regarding carbonyl oxygen to metal ion [18], while Hg(II) complex showed new broad peaks in region (3445, 3414 cm^{-1}) allocated to the coordinated water, that might be due to the combination (O-H) related to coordinated water [19].Therefore, FTIR data suggesting that Schiff bases were bound to metal ions via imino nitrogen atoms and oxygen atom. Table (2) shows the IR spectral data.

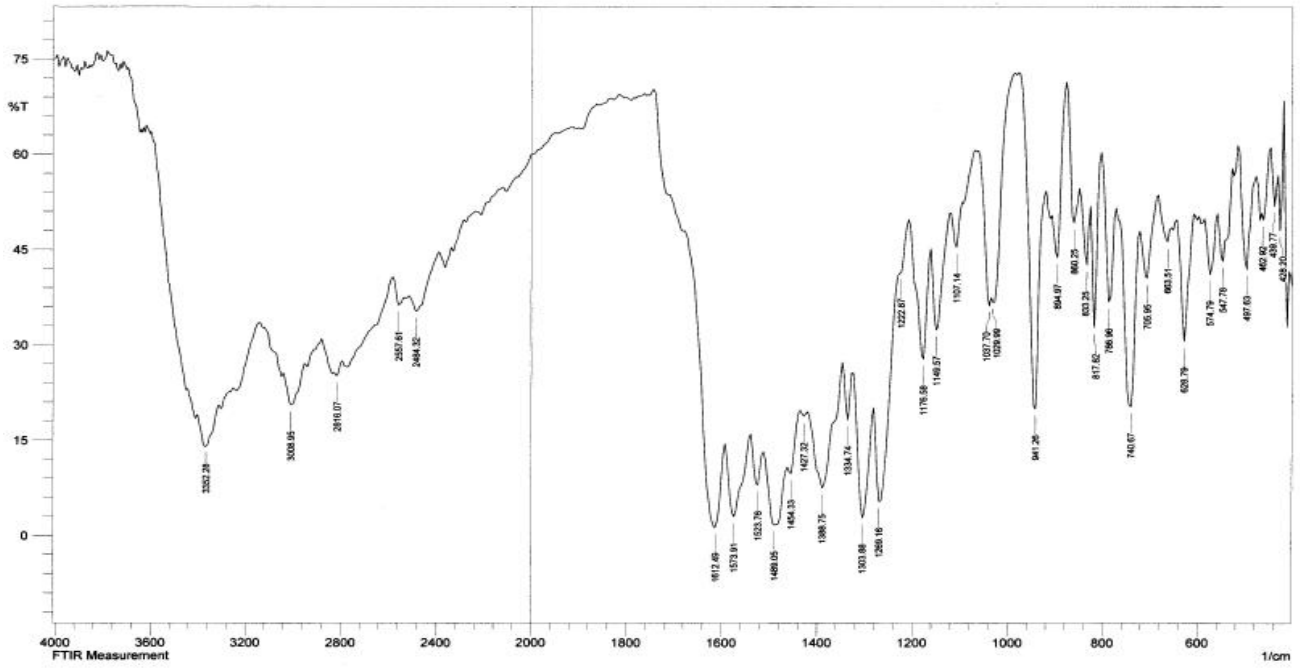


Figure 2. The FTIR spectrum of ligand (L_1)

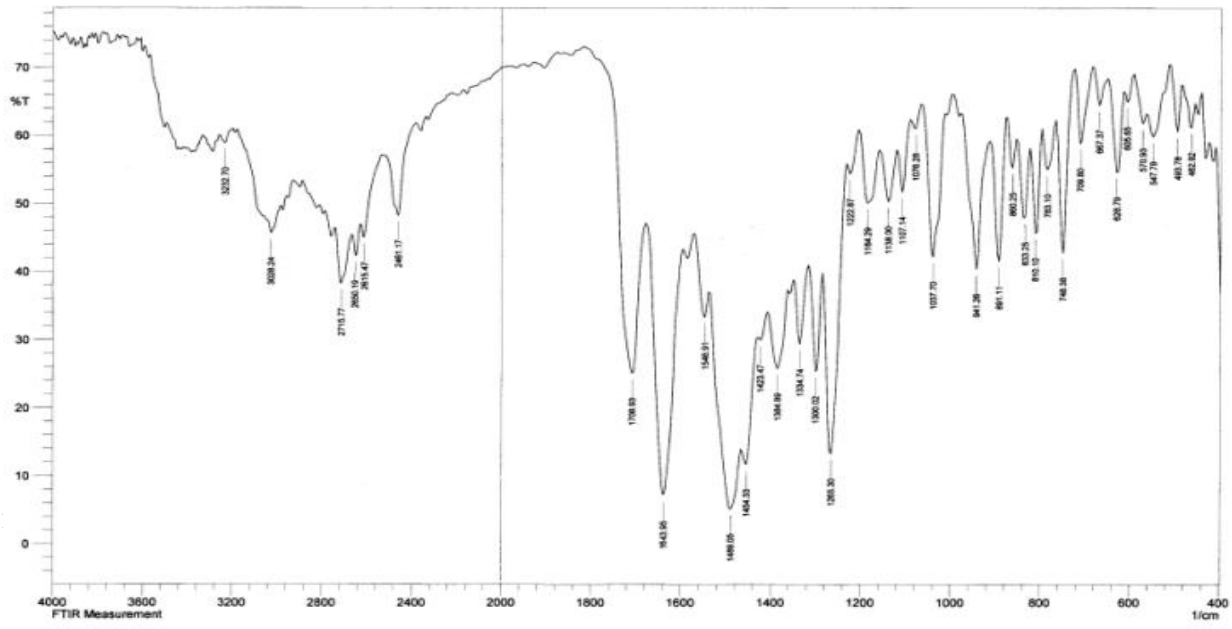


Figure 3. The FTIR spectrum of ligand (L_2)

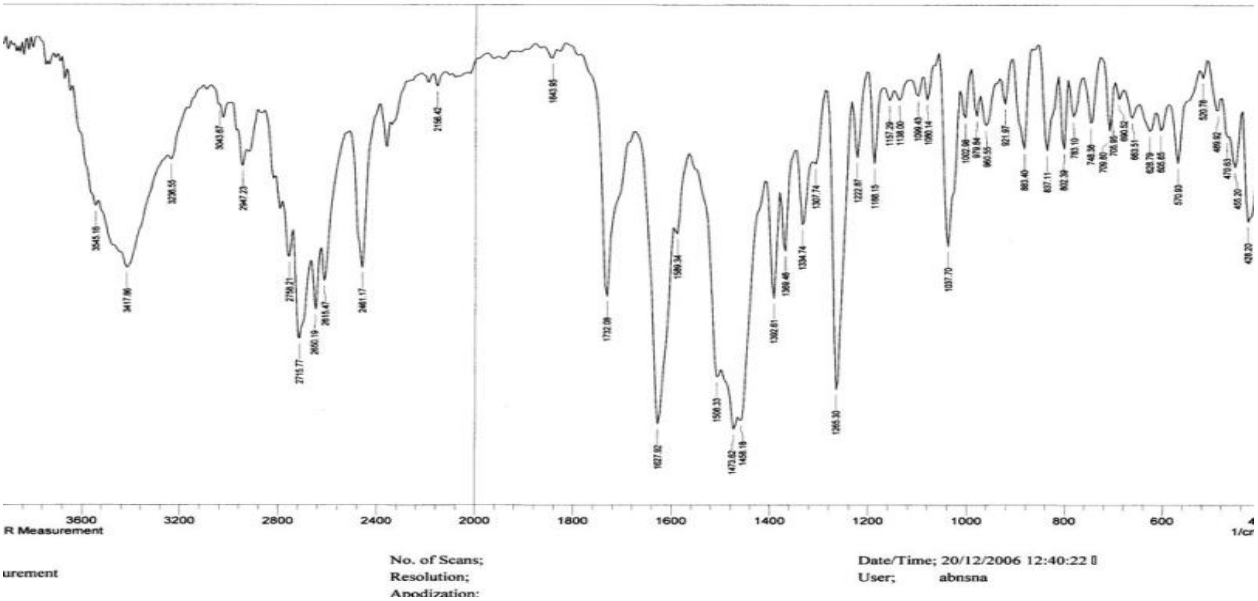


Figure 4. The FTIR spectrum of $[Hg_2 (L_1) (L_2) (H_2O)_4] Cl_2$complex

Table (2). Infra-red spectral data (wave number υ⁻) $cm^{-1}$of Schiff bases and their complexes

ν (M-O)	ν (M-N)	δ (OH) H2O	ν(C-H)arom.	ν(C-O)	ν(C=N)imine	ν(C=O)amide	ν(N-H)	υ(OH) H2O	υ(OH)	Compounds
-	-	-	3008	1239	1612	-	-	-	3352	(L1)
-	-	-	3028	-	1643	1708	3232	-	-	(L2)
445 432	512 459	-	3032	1261	1588 1622	1720	3240	-	-	[Cu2(L1) (L2)] Cl2
470 455 428	520 489	921	3043	1265	1589 1627	1732	3236	3445 3414	-	[Hg2(L1) (L2) (H2O)4] Cl2

3.2 ^{1}H-NMR spectrum of the ligands(L_1) and (L_2)

^{1}H-NMR (DMSO-d_6) of (L_1) (Figure 5, Table 3): δ = 2.50 (1H, OH in benzoin), 2.49 (DMSO), 3.39 (1H, -CH-OH), 7.21-8.01 (7H, m, C_6H_5) [20]

^{1}H-NMR (DMSO-d_6) of (L_2) (Figure 6, Table 4): δ =2.47 (DMSO), 6.62-7.56(6H, m, C_6H_5), 10.99 (1H, NH in isatin) [21]

Table 3. ^{1}H-NMR data for ligand (L_1) evaluated in DMSO-d_6 as well as chemical shift in ppm (δ)

(OH) in benzoin	DMSO	N=CH-OH	(C=CH) arom.
2.50	2.49	3.39	7.21-8.01

Table 4. ^{1}H-NMR data for ligand (L2) evaluated in DMSO-d_6 as well as chemical shift in ppm (δ)

(N-H) in Isatin	DMSO	(C=CH) arom.
10.99	2.50	7.56-6.62

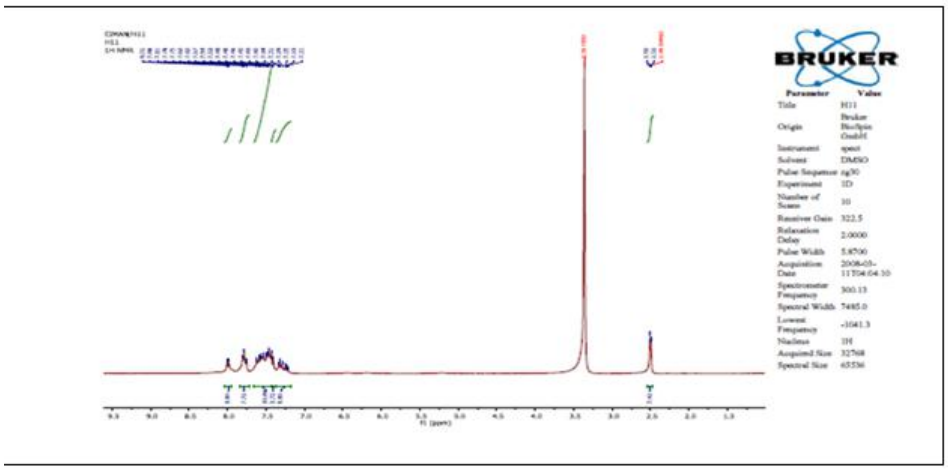


Figure 5. The^{1}H-NMR for the ligand (L_1)

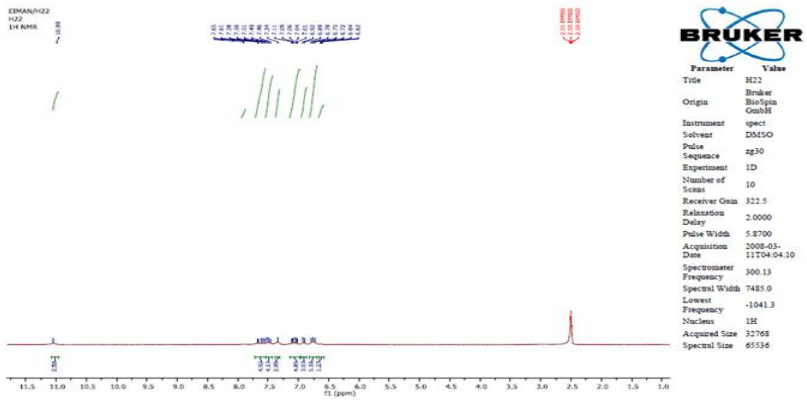


Figure 6. The ^{1}H-NMR for the ligand (L_2)

3.3 Conductivity measurements, magnetic susceptibility and electronic spectra

Electronic spectra for ligands in addition to their complexes were shown in (Figure 7, Table 5), together with the magnetic moments. Also, the molar conductivities are indicating that all metal complexes were electrolytes [22]. The absorption spectrum regarding copper complex, showed band at about (17574 cm^{-1}) attributed to($^5E \rightarrow {}^5T_2$) transition of a Cu complex with tetrahedral geometry. The magnetic moment (μeff) for such complex was 2.3 B.M. for each Cu ion that was in range for tetrahedral copper complex [23].

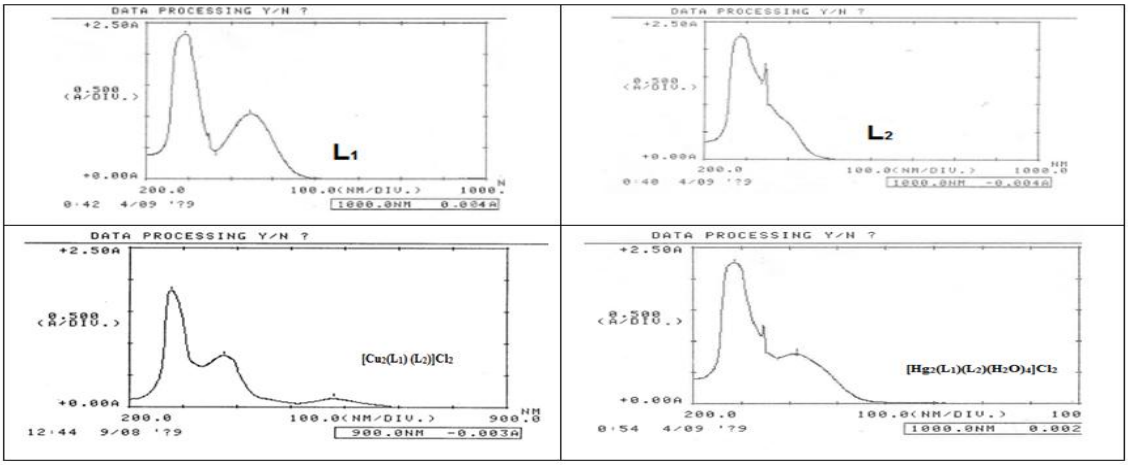
The absorption spectrum related to Hg complex, showing absorption band at about (24038cm^{-1}), due to (charge transfer) transition, which was found to be diamagnetic as expected for d^{10}system [24,25], where d-d transitions are excluded [26]. Based on these data, geometry have been assigned to the Cu (II) as well as Hg (II) complex. According to elemental analysis, FT-IR and TGA.

Table 5. electronic spectral data of the ligands and their metal complexes

geometry	μ (BM)	Λm (S.mol-1.cm2)	Transitions	Ɛmax (molar-1 .cm-1)	υ' cm-1	λ nm	Compounds
_	-	-	π→π* n→π*	2320 1041	34129 22371	293 447	(L1)
_	-	-	π→π* n→π*	2232 1689	34965 28901	286 346	(L2)
tetrahedral	2.3	78.4	L.F C.T 5E→5T2	1836 418 1302	35971 28818 17574	278 347 569	[Cu2(L1) (L2)]Cl2
octahedral	-	72.6	L.F L.F C.T	2286 1748 808	34722 28735 24038	288 348 416	[Hg2(L1) (L2)(H2O)4] Cl2

Figure (7) Electronic spectrum of the ligands and their complexes

3.4 Thermal decomposition of $[Hg_2(L_1)\ (L_2)\ (H_2O)_4]\ Cl_2$ complex



TGA thermal analysis curves for $[Hg_2(L_1)\ (L_2)\ (H_2O)_4]\ Cl_2$is shown in (Figure 8), and data have been listed in (table 6). The Hg^{II} complex decomposes in three steps. The first stage is the loss ($C_{40}H_{30}N_2$+2H_2O) with mass losses of 36.3217% (calc. = 36.85890232 %) within a temperature range of 30-293.596^{0}C, the decomposition of the complex in the 30-293.596^{0}C range is indicated by exothermic processes at

91.7^{0}C ,the decomposition of the complex in the 30-293.596^{0}C range is indicated by endothermic at 166.9^{0}C and 216.7^{0}C and 255.8^{0}C,the second step involves the loss of the organic fraction , ($C_8H_5NO+2H_2O$) within the temperature range of

293.596-415.602^{0}C, with mass losses of 10.3935% (calc. =10.7237573%),the decomposition of the complex in the 293.5960-415.602^{0}C range is indicated by exothermic at 373.9^{0}C . The third step involves the loss of the organic fraction, ($C_{20}H_{13}N_3O$) weight of the compound observed at 20.093% (calc. =19.97058994%) within a temperature range of 415.602-595.661^{0}C. The difference in the calculations in observed of the residue weight may be related to the sublimation upon thermal decomposition, the difference in the calculations in observed of the residue weight may be related to the sublimation upon thermal decomposition [27].

Table 6. Thermo analytical result (TG and DSC) of $[Hg_2(L_1)\ (L_2)\ (H_2O)_4]\ Cl_2$ complex

Compounds	Mass loss temp./°C	Mass loss Theoretically	Mass loss Practically	DSC
$[Hg_2(L_1)\ (L_2)\ (H_2O)_4]\ Cl_2$	30-293.596	36.85890232	36.3217	91.7(exo) 166.9(endo) 216.7(endo) 255.8(endo)
	293.5960-415.602	10.7237573	10.3935	373.9(exo)
	415.602-595.661	19.97058994	20.093	-

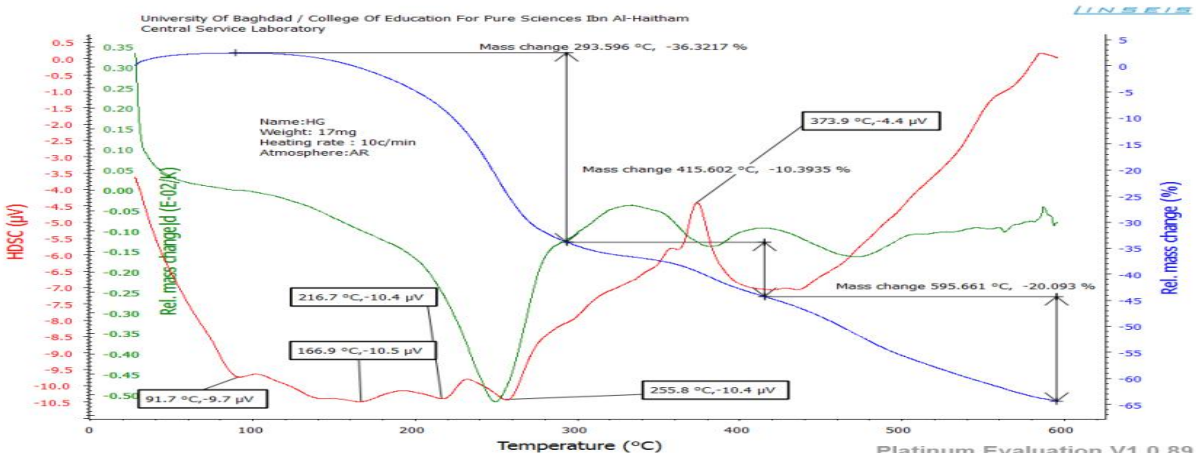


Figure 8. (TG/DTG and DSC) Thermo gram of $[Hg_2(L_1)(L_2)(H_2O)_4]Cl_2$

4-Biological Activities

Biological Activities related to ligands as well as their complexes were conducted against pathogenic bacteria such as (E. coli and s. aureus) and fungi such as (Candida tropicalis and Canidia albicans) utilizing nutrient agar medium via disc diffusion approach [28]. Also, the test solution is prepared in DMSO, after that soaked in filter paper with diameter of 5mm and thickness of 1mm. Discs are placed on already-seeded plates and subjected to incubation for 24 hours at a temperature of 37 Celsius. In addition, the diameter of the zone of inhibition around each one of the disks was evaluated following 24 hours. Also, the synthesized complexes showing considerable biological activity against the fungi and bacteria as shown in (Table 7, Figure 9), whereas the antimicrobial results showing that the activity of Schiff base ligands were pronounced in the case when coordinated to metal ions $[Hg_2(L_1)(L_2)(H_2O)_4]Cl_2$ showing high antibacterial activity against the (E. coli and S. aureus) and fungi (Candida tropicalis and Canidia albicans).Therefore, the overall potency related to free ligand was improved on coordination with the metal ion. Furthermore, the overall order regarding antimicrobial activity was $[Hg_2(L_1)\ (L_2)\ (H_2O)_4]\ Cl_2$ > $[Cu_2(L_1)\ (L_2]\ Cl_2$ >L_1 >L_2.The improvement in the anti-microbial activity of complexes compared to free Schiff base might be specified based on Tweedy chelation theory [29].

Table 7. Inhibition circle diameter in millimeter for the ligands and their complexes

Compounds		Inhibition Zone Diameter (mm)			
		Escherichia Coli (G^-)	staphylococcus aureus(G^+)	Canidiaalbicans	Candida tropicalis
1	(L_1)	-	16	-	-
2	(L_2)	-	11	-	-
3	$[Cu_2(L_1)\ (L_2)]\ Cl_2$	22	13	-	-
4	$[Hg_2(L_1)\ (L_2)\ (H_2O)_4]\ Cl_2$	14	21	15	16
5	DMSO	-	-	-	-

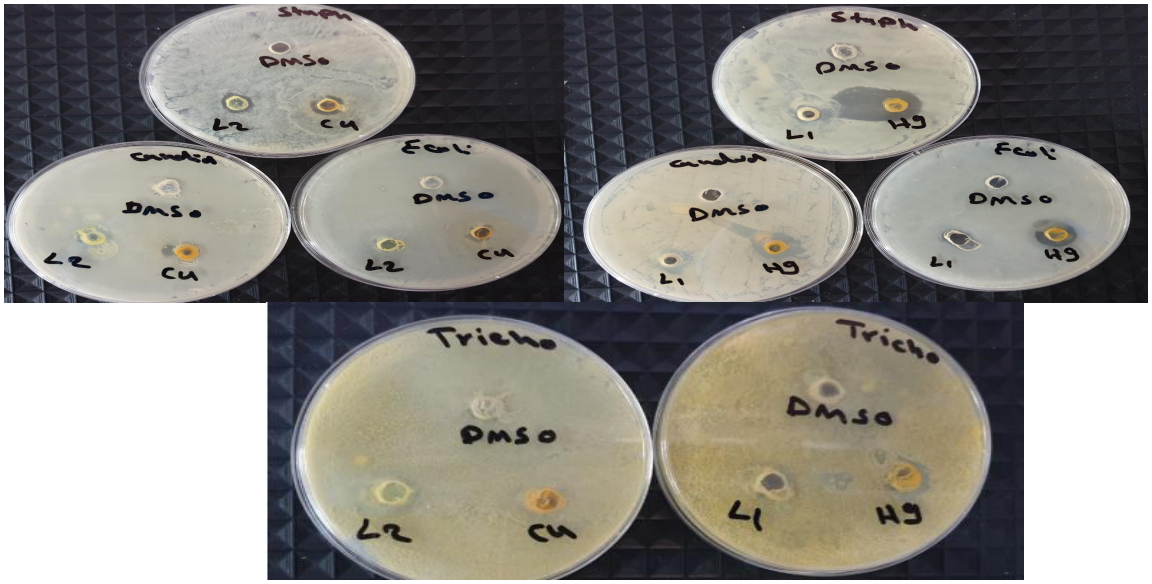


Figure (9) The biological activities of the ligands and their complexes

5-Conclusion

In the presented study, the characterization and synthesis of tetra dentate Schiff base ligands: ligand(L_1) obtained from benzidine and benzoine, also ligand (L_2) obtained from benzidine and isatin, their interactions with the metals are indicated, while the metal complexes created were electrolytic in nature. In addition, the synthesized systems have been specified via TG analysis, FT-IR, microanalysis, UV spectroscopy and molar conductance values. Furthermore, the spectral and analytical data suggesting tetrahedral geometry for complex $[Cu_2(L_1)\ (L_2)]\ Cl_2$ as well as octahedral geometry for complex $[Hg_2(L_1)\ (L_2)\ (H_2O)_4]\ Cl_2$. On the basis of the already-mentioned results, the structure of coordination compounds under study are expressed in (Figure 2).

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