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Synthesis, Characterization and Biological Activity Study for Some New Metals Complexes with (3Z,3'E)-3,3'-(((2E,5E)-hexane-2,5-diylidene)bis(hydrazine-2,1-diylidene))bis(indolin-2-one)

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Abstract

Our recent work displays the successful preparation of Schiff_bases that carried out between hexane-2,5-dione and 2 moles of (Z)-3-hydrazineylideneindolin-2-one forming in Schiff-bases-(L), Which in turn allowed combining with each of the next metal ions: (M^{2+}) = Ni, Mn, Zn, Cu and Co forming complexes_ in high stability. The formation of resulting Schiff_ bases (L) is detected spectrally using LC_Mss which gave approximately matching results with theoretical incomes, ^{1}HNMR proves the founding of doublet signal of (2H) for 2NH, FTIR indicates the occurrence of two interfered imine bands and UV-VIS mean is also indecates the formation of ligand. On the other hand, complexes-based-Schiff were characterized using the same spectral means that relied Schiff-bases_(L). Those means gave satisfactory results and proved the distinguishable geometries that suggested. Finally, and according to the antibiotic feature of Schiff-bases and its metal ions we have also examined such character against (-Bacteria and +Bacteria) giving an acceptable inhibition efficiency.

Key words: Hexadion ,Isatin-3-hydrazone, Schiff_bases complexes, biological activity .

1. Introduction

Schiff bases are organic compounds originating from the condensation of a primary amine with an aldehyd e or a keton, to give imine_groups. Electronic and steric effects can be easily adjusted by the proper choice of the amine and substituted carbonyl compound, to provide Schiff bases with great structural variability [1, 2]. Schiff bases constitute an important class of ligands with wide application in coordination chemistry: these bases can serve as bis-, tris-, tetra-, penta- or polydentate ligands for transition and representative metals and ions.[3,4] The presence of other functional groups in the structure of the starting carbonyl and/or amine used to synthesize the Schiff base provides the imine with two or more different donor atoms,[5] predominantly nitrogen, oxygen and sulfur,[5, 6] but also phosphorus, selenium and tellurium, to chelate metals and ions.[7] As a result of their stability, facile preparation, structural variability and versatile coordinating capabilities, Schiff bases play a key role in coordination chemistry, and their coordination complexes have several applications.[8,9] All these advantages have led Schiff bases to be often called “Privileged Ligands.” [10, 11] In 1864, Hugo Schiff [12] was the first to synthesize Schiff bases. Although, this class of compounds has long been known, they are still extensively studied in diverse research fields. For example, numerous reviews have dealt with structures and reactions of Schiff base complexes [13] and their catalytic [14-16], biological, magnetic, electrochemical [17] and cancer treatment applications, which often depend on the Schiff base structure.

2. Materials and method

2.1 Methodology

The starting materials including ($MnCl_2.4H_2O$,$CoCl_2.6H_2O$,$NiCl_2.6H_2O$, $CuCl_2.2H_2O$ and $ZnCl_2$) were commercially abundant in addition to the employed catalysts including (dimethyl sulfoxide, abs. ethanol. and diethyl-ether) that supplied from F-897ewluka, sigma aldrich. The employed FTIR apparatus operates in the range (200-4000) cm^{-1} Shimadzu-3800 model. Electronic spectral inform were accomplished depending on Shimadzu160-meter. LC/MSS incomes are also established by Mass100P_Shimadzu contribution. Pyrolysis diagnosis were carried out depending on perkin_Elmer_pyris Diamond D_S_C/TG. Proton-NMR was published using Bruker 400-MHz-meter and elemental micro analysis were done on a perkin_Elmer_automatical instruments model_240B. minerals were determined obeying a Shimadzu_(A-A)_680G AA_spectrometer. The Cl combination were estimated by conductivity measurements. Magnetic features were measured using balance magnetic susceptibility model MSR-MK

2.2 Organic ligand (Schiff-bases-(L)) and Metal complexes synthesis

This ligand has been synthesis using the general strategy that used in Schiff base synthesis and carried out in round bottomed flask of 100 ml in volume. At which (1g, 2mol) of main substance (Z)-3-hydrazineylideneindolin-2-one is dissolved in 10 ml of EtOH. With continuous stirring and heating to perform the dissolution of the main substance. Then, (0.342g, 1mol) of hexane-2,5-dione is added onto the main substance solution. Finally, adding two drops of glacial acetic acid with reflux and continuous stirring about 4 hours resulting in olive precipitate as in **Figure 1** below the (Schiff-bases_ (L)) synthesis. The former is filtered and dried in oven.

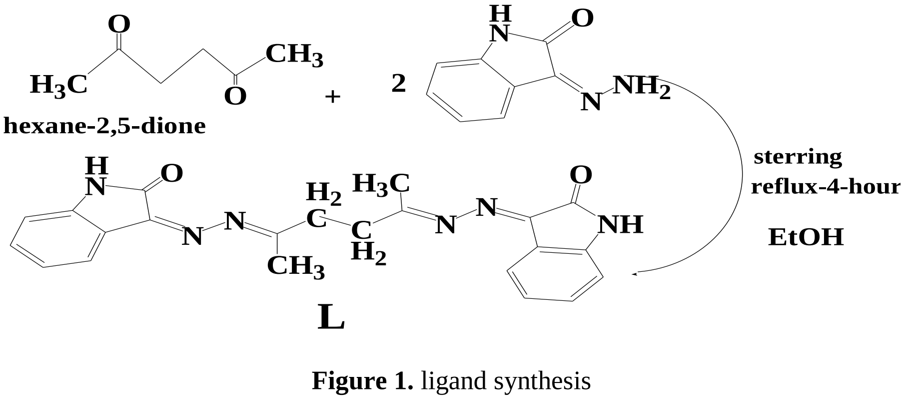

Figure 1. ligand synthesis

2.3 METAL complexes synthesis

Cobalt complex of the obtained (Schiff-bases_ (L) has been prepared using the following approach: dissolving (0.08 g, 1 mol) of ligand_(L) in 10 ml of MeOH with continuous stirring and heating to perform the dissolution, Then adding (0.073 g, 0.001 mol) of Co-salt ($CoCl_2.6H_2O$) that dissolved in MeOH, then reflux and continuous stir ring for 4 hours. After completely reflux, mixture is filtered while its heat, dried, washed and adding a few drops of diethylether. Resulting in light orange precipitate with (215-219)ºC. Other complexes of the following metal salts: $NiCl_2.6H_2O$ (0.06 g, 1 mol), $CuCl_2.2H_2O$ (0.08 g, 1 mol), $MnCl_2.4H_2O$ (0.05g, 1 mol) and $ZnCl_2$ (0.05g, 1 mol) , were prepared using the same approach that used in Co-complex synthesis Resulting in: light olive precipitate with (228-232)ºC, brown precipitate with (215-220)ºC, red precipitate with (275-279)ºC and orange precipitate with (195-198)ºC for each complex respectively.

3. Result and discussion 3.1 FT-IR studies

FT-R spectrum of newly obtained ligand L in **Figure 1**. Displays a distinguishable absorption band at 1658 cm^{-1} contributes to azomethine formation, which can be strong evidence about ligand (L) synthesis. In addition ,the absent of asymmetrical absorption band of NH_2-amino group. This can be strong indication that proves the formation of ligand through the interaction between carbonyl group of hexane-2,5-dione and amino group of isaten. It’s important to note the absent of C=O absorption band of hexane-2,5-dione which can also support the formation of ligand through this group. Other absorption bands were detected at (3155, 2900, 3356, 1683 and 1463) cm^{-1} that belonging to the stretching vibrational mode of the following functional groups: C-H aromatic, C-H aliphatic, N-H amine, C=O of amide and C=C of alkene respectively [18]. As demonstrated in **Table 2** and figure 2 for Copper(II) complex $[Cu(L)(Cl)(H_2O)]Cl$ in **Figure 2**. Demonstrates many changes including shifting in stretching vibrational mode of C=N group because of the occurrence of coordination through N of both C=N groups, to be detected at 1618 cm^{-1}. In addition, appearing new absorption bands at (580, 482 and 254) cm^{-1} attributed to the vibration of M-N, M-O and M-Cl respectively. Besides the bands of coordinated water molecule that observed at (3450, 1527 and 754) cm^{-1}. Those new bands can strongly prove the formation of complex and the presence of H_2O aqua inside coordination sphere [19, 20]. Other complexes in **Figures 3, 4, 5** and **6** also display individually such modifications that happened in Copper complex as mentioned in **Table 2**.

Table 1. FT-IR spectral data of ligand (L) and its complexes

Compound	C-H Alph.	C-H Atom	C=N Imine	N-H	C=O amide	C=C	M-N	M-O	M-Cl	H_2O aqua
$C_{22}H_{20}N_6O_2$ L	2900 (w-m)	3155 (s)	1658 (s)	3356 (m,b)	1683	1463 (m-w)	--	--	--	--
[Cu(L)(Cl)(H_2O)]	2985 (w-m)	3109 (w)	1618 (s)	3195 (m,b)	1691 s	1463 (m-s)	580 m	482 w- m	254 s	3450 1527 754
[Ni(L)Cl_2(H_2O)$_2$]	2956 (w-m)	3161 (s)	1618 (s)	3215 (m,b)	1687 s	1465 (m-s)	501 m	445 w- m	256s	3359 1552 748
[Zn(L)Cl_2]	2966 (w-m)	3064 (m)	1618 (s)	3201- 3218 (m,b)	1681 s	1463 (m-s)	541 m	497 w- m	223 S	--
[Mn(L)Cl_2(H_2O)$_2$]	2958 (w-m)	3050 (w)	1614 (s)	3163- 3215 (m,b)	1685 s	1465 (m-s)	520 m	499 w	297 s	3361 1552 748
[Co(L)Cl_2(H_2O)$_2$]	2990 (w-m)	3050 (m)	1656 (s)	3161- 3217 (m,b)	1687 s	1465 (m-s)	520 w	499 w	239 S	3361 1554 748

s= strong, b= broad, m= moderate, w= weak

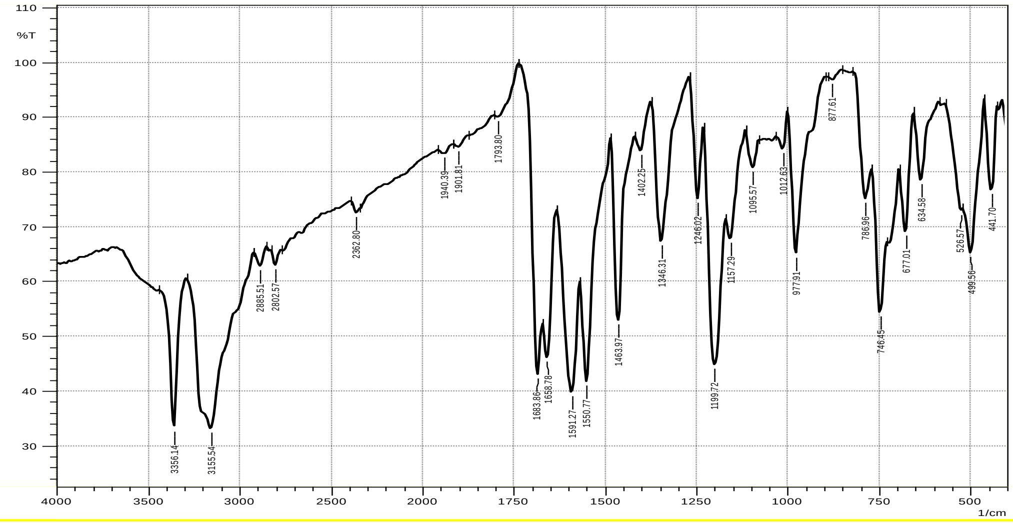

Figure 1. FT-IR spectrum of ligand (L)

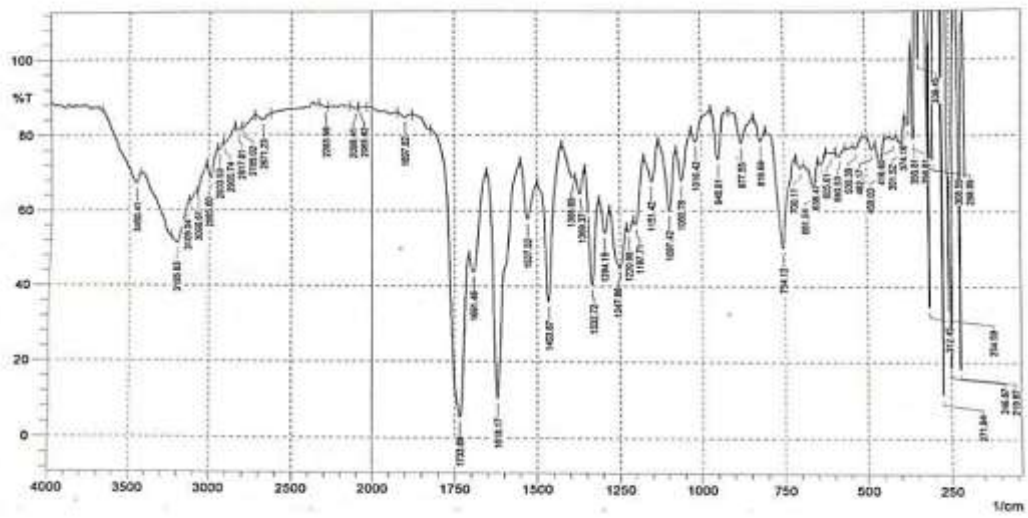

Figure 2. FT-IR spectrum of Copper complex

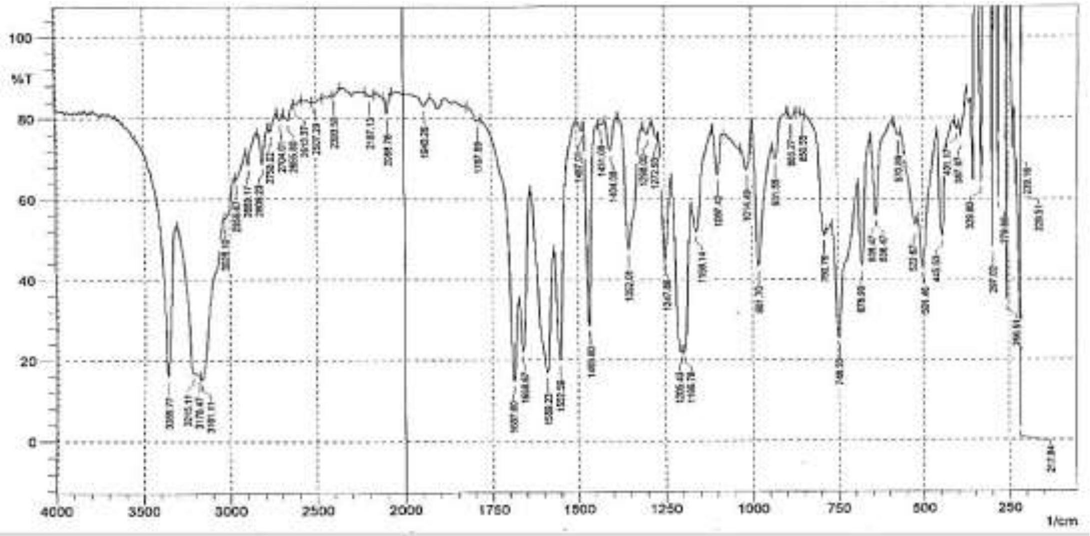

Figure 3. FT-IR spectrum of Nickle complex

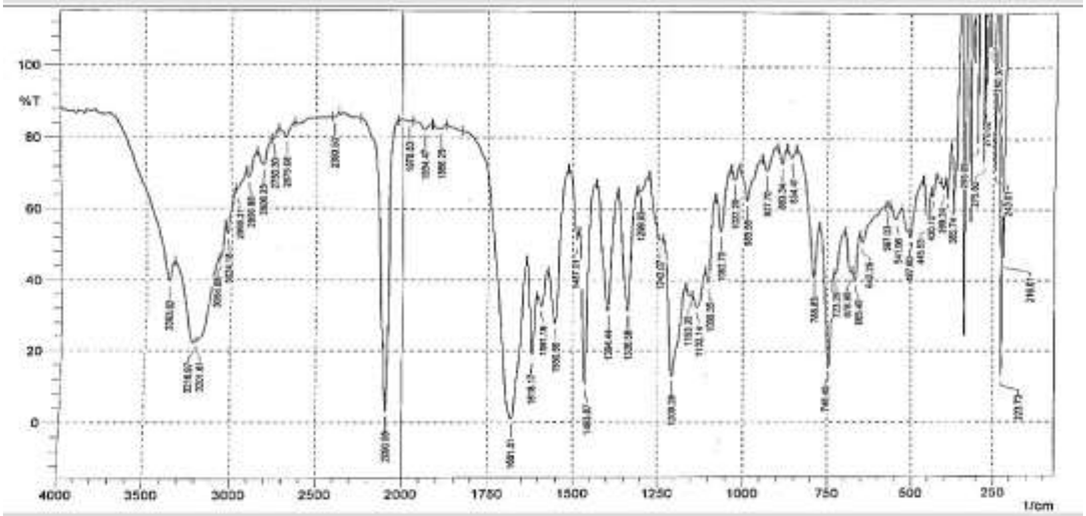

Figure 4. FT-IR spectrum of Zinc complex

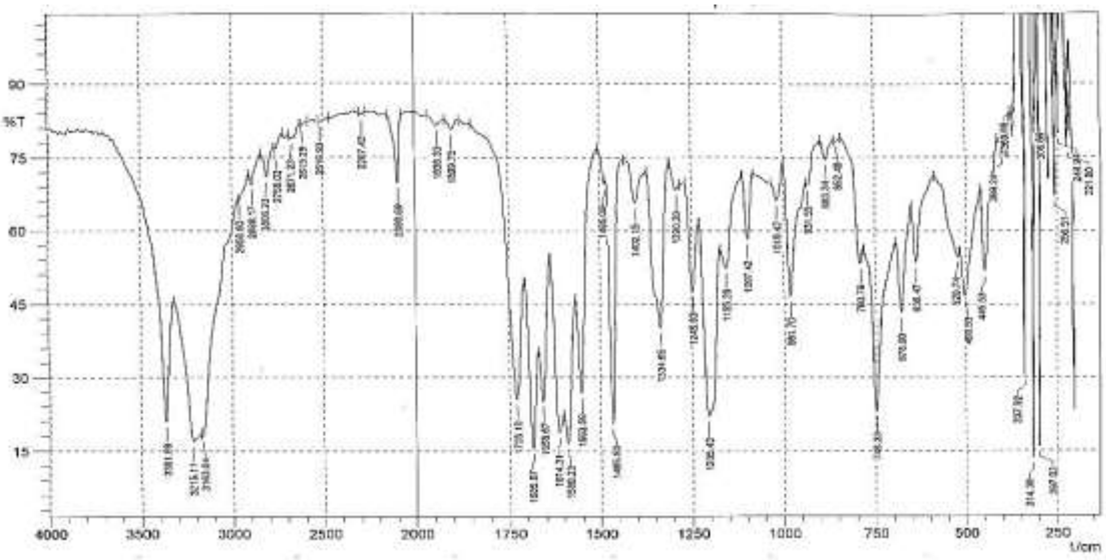

Figure 5. FT-IR spectrum of Manganese complex

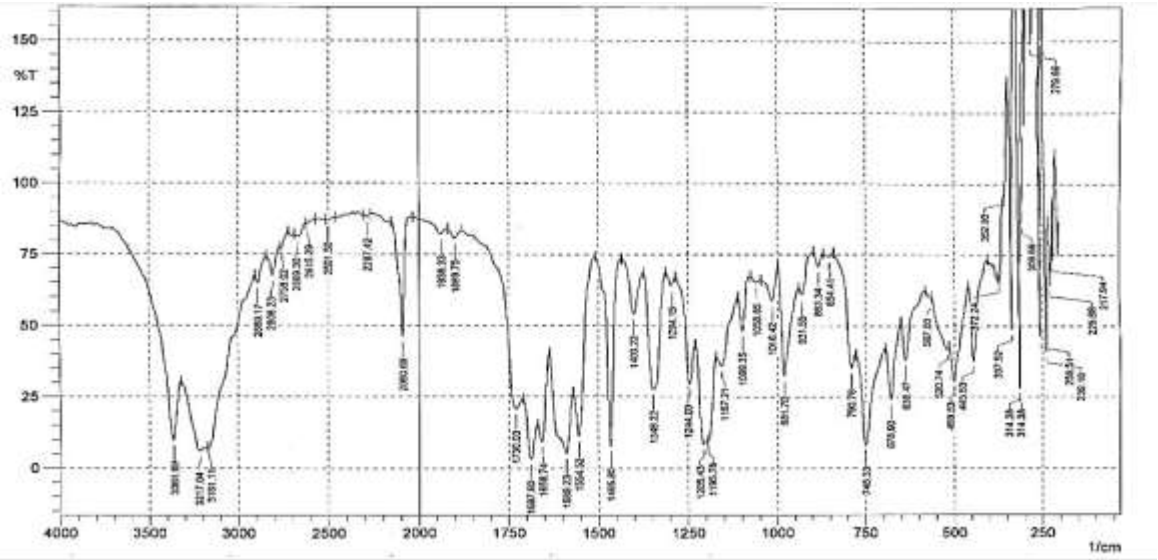

Figure 6. FT-IR spectrum of Cobalt complex

3.2 UV-Vis studies

Figure 7, demonstrates uv-vis spectrum of ligand (L), at which two $\pi \rightarrow \pi^*$ transitions occurred at (236 nm, 42372.881cm^{-1}) and (265 nm, 37735.849 cm^{-1}). These transitions attributed to the presence of unsaturated bonds and aromatic rings in ligand's structure. Other electronic transition that occurred in ultraviolet region is $n \rightarrow \pi^*$ electronic transition at (316 nm, 31645.569 cm^{-1}). This transition causes by the presence of heteroatoms in ligand's structure such as (-N-) that contains nonbonding electrons [21]. **Figure 8** Illustrates UV-Vis spectrum of Copper-L complex in diluted form at which ultraviolet transitions that referred to as $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ were shifted compared to the same transitions that observed in ligand's spectrum to be observed at (265 nm, 37735.849 cm^{-1}) and (304 nm, 32894.736 cm^{-1}) for both transitions respectively. This modification causes by the occurrence of coordination with metal ion through both Schiff base groups. Moreover, single transition observed at visible region (978 nm, 10224.948 cm^{-1}) in its concentrated form **Figure 9**, denoted as ${}^2T_2 \rightarrow {}^2E$ the transition that found in metal itself. This transition and the magnetic moment [3.81B.M] can support (td) geometry of the complex [22]. By the same approach, we can apparently discuss the electronic transitions for the rest complexes that displayed in **Figures 10, 11, 12** and **13** and **Table 2**, **Figure 11**. Illustrates UV-Vis spectrum of Cobalt-L complex at which ultraviolet transitions that referred to as 2 ($\pi \rightarrow \pi^*$) (202 nm, 49504.950 cm^{-1}), (245 nm, 40816.326 cm^{-1}) and $n \rightarrow \pi^*$ (316 nm, 31645.569 cm^{-1}). Were shifted compared to the same transitions that found in ligand's spectrum. This modification causes by the occurrence of coordination with metal ion through both Schiff base groups. Moreover, the transitions that observed at visible region are as follows: ${}^4A_2g \rightarrow {}^4T_2g_{(F)}$ at (412 nm, 24271.844 cm^{-1}), ${}^4A_2g \rightarrow {}^4T_1g_{(F)}$ (461 nm, 21691.973 cm^{-1}) and ${}^4A_2g \rightarrow {}^4T_1g_{(P)}$ at (482 nm, 20746.887cm^{-1}) those transitions are found in metal itself. Those transitions and the magnetic moment [2.79 B.M] can support (Oh) geometry of the complex [23].

Table 2. UV-Vis spectral data of ligand (L) and its complexes

Compound	Abs.	ε_{max} L.mol^{-1}.cm^{-1}	λ_{max} nm	μ_{eff} B.M	ύ cm^{-1}	Transition
$C_{22}H_{20}N_6O_2$ L	3.115	3115	236	--	42372.881	$\pi\rightarrow\pi^*$
	3.070	3070	265		37735.849	$\pi\rightarrow\pi$*
	1.317	1317	316		31645.569	n$\rightarrow\pi$*
[Cu(L)(Cl)(H_2O)]	1.213	1213	265	1.62	37735.849	$\pi\rightarrow\pi^*$
	0.663	663	304		32894.736	n$\rightarrow\pi$
	0.002	2.00	978		10224.948	$^2T_2\rightarrow{}^2E$
[Zn(L)Cl_2]	4.000	1333	299	diamagnetic	40322.581	$\pi\rightarrow\pi^*$
	3.950	321	310		37037.037	n$\rightarrow\pi$
	4.000	488	406		30303.030	C.T(M→L)
[Co(L)Cl_2(H_2O)$_2$]	2.857	2857	202	3.12	49504.950	$\pi\rightarrow\pi$*
	0.282	282	245		40816.326	n $\rightarrow\pi$*
	4.000	4000	316		31645.569	n $\rightarrow\pi$*+ (C.T)
	4.000	4000	412		24271.844	$^4A_2g\rightarrow{}^4T_2g$ (F)
	4.000	4000	461		21691.973	$^4A_2g\rightarrow{}^4T_1g$ (F)
	4.000	4000	482		20746.887	$^4A_2g\rightarrow{}^4T_1g$ (P)
[Mn(L)Cl_2(H_2O)$_2$]	0.952	952	212	2.79	47169.811	$\pi\rightarrow\pi$*
	4.000	4000	352		28409.090	n $\rightarrow\pi$*
	4.000	4000	401		24937.655	$^6A_1\rightarrow{}^4T_{1G}$
	0.004	4.000	908		11013.215	$^6A_1g\rightarrow{}^4A_1g+{}^4E_G$
[Ni(L)Cl_2(H_2O)$_2$]	1.997	1997	303	3.81	33003.300	$\pi\rightarrow\pi$*
	1.660	1660	325		30769.230	n $\rightarrow\pi$*
	0.002	2.000	978		10224.948	$^3T_{1F}\rightarrow{}^3T_{2F}$

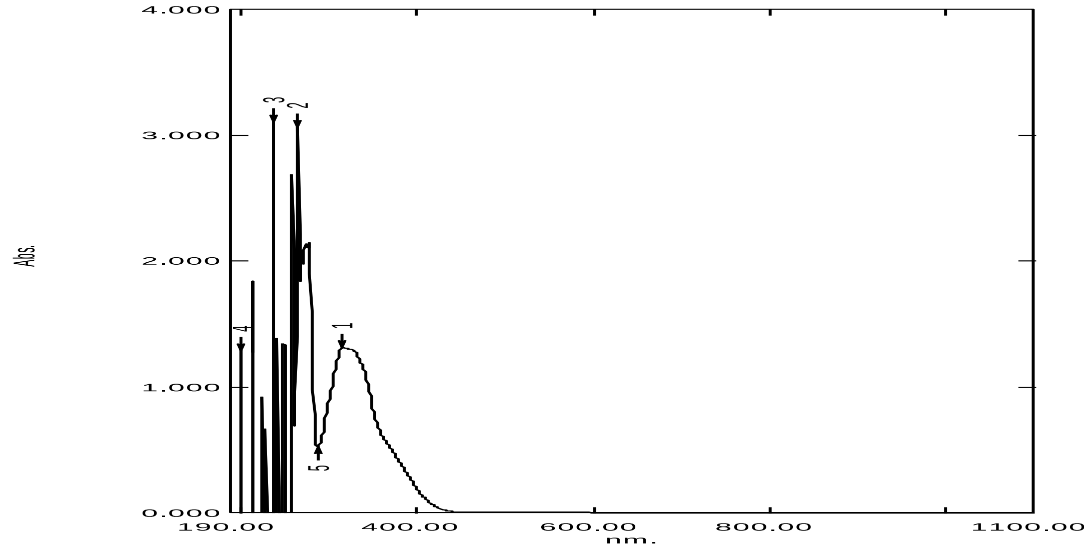

Figure 7. UV-Vis spectrum of ligand

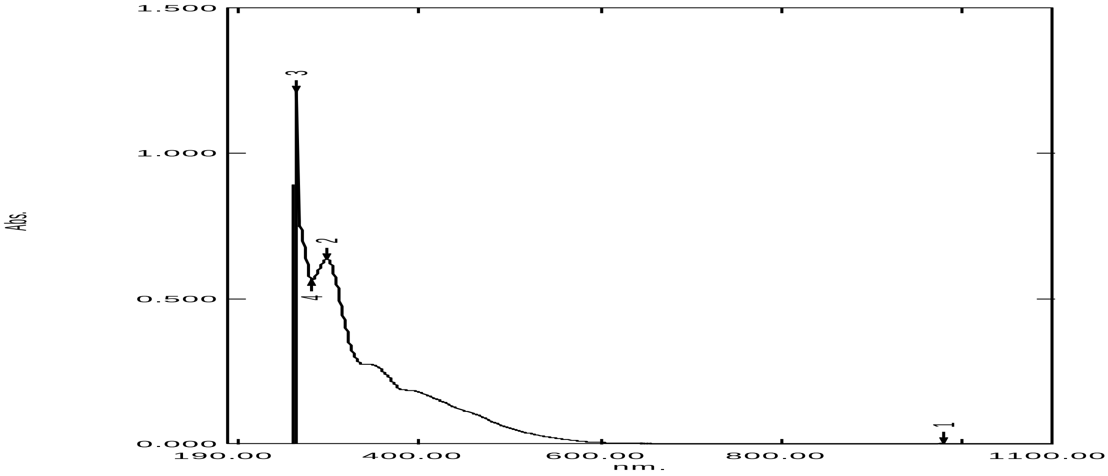

Figure 8. UV-Vis spectrum of dill. Copper complex

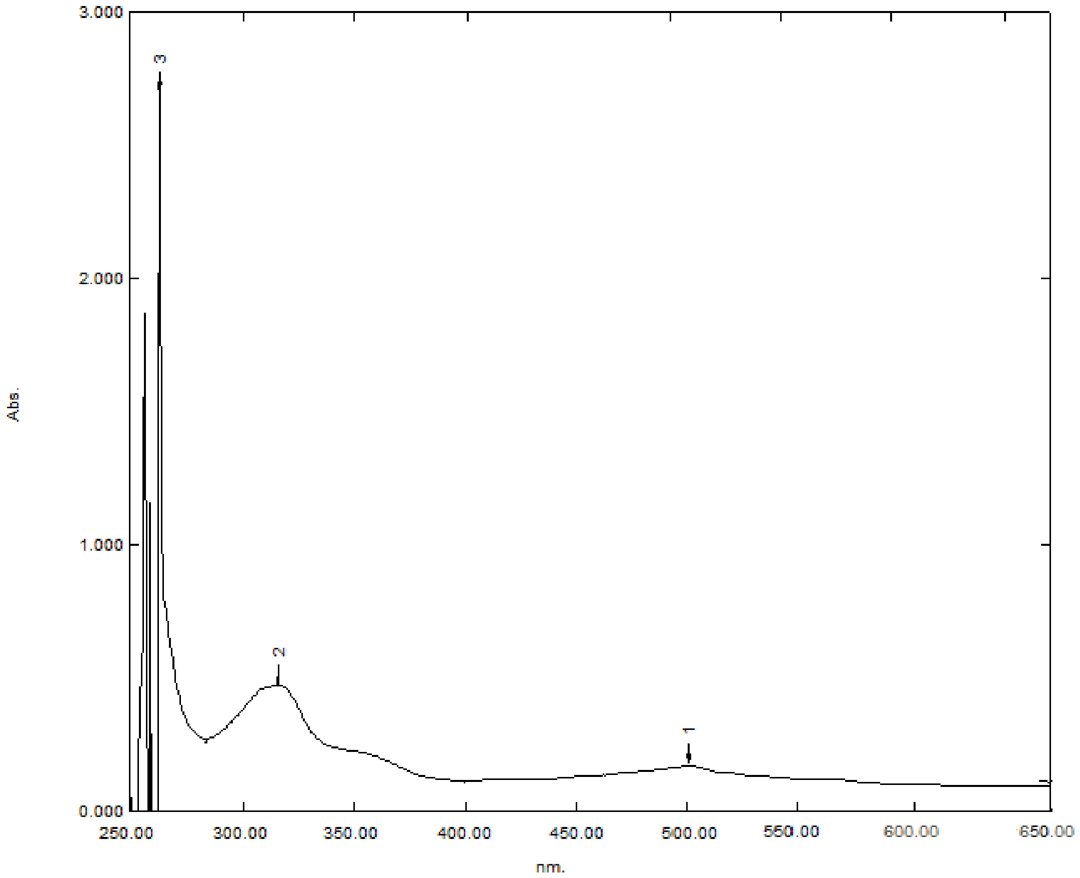

Figure 9. UV-Vis spectrum of conc. Copper complex

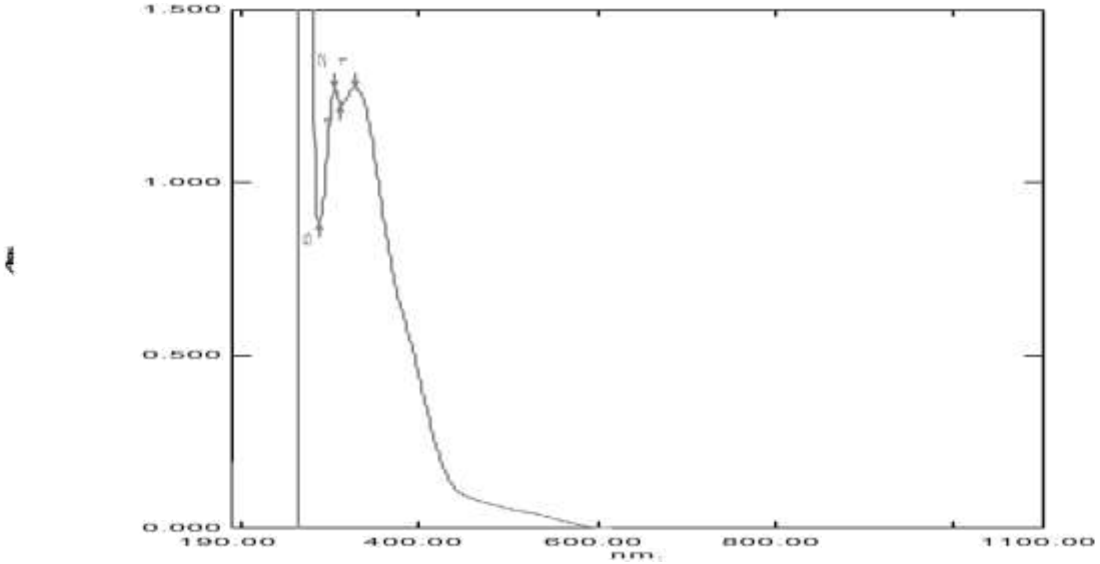

Figure 10. UV-Vis spectrum of Zinc complex

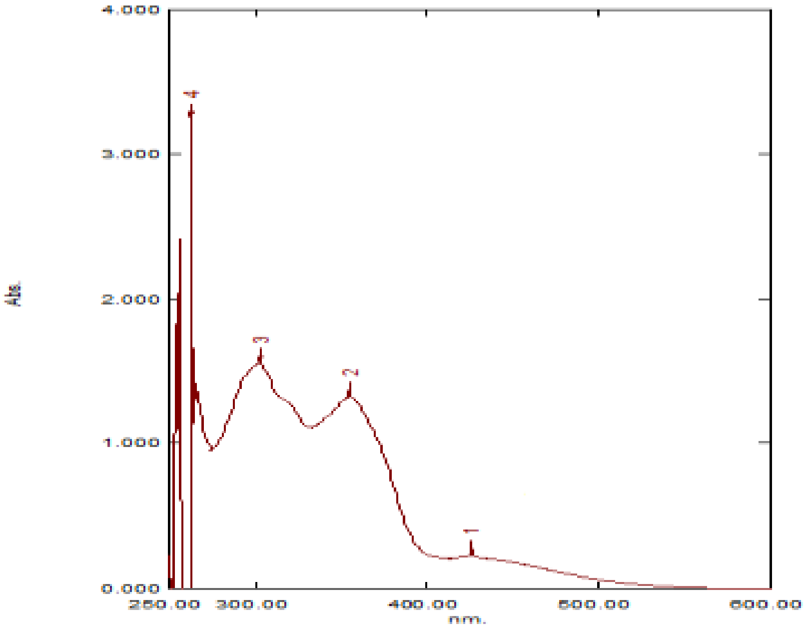

Figure 11. UV-Vis spectrum of Cobalt complex

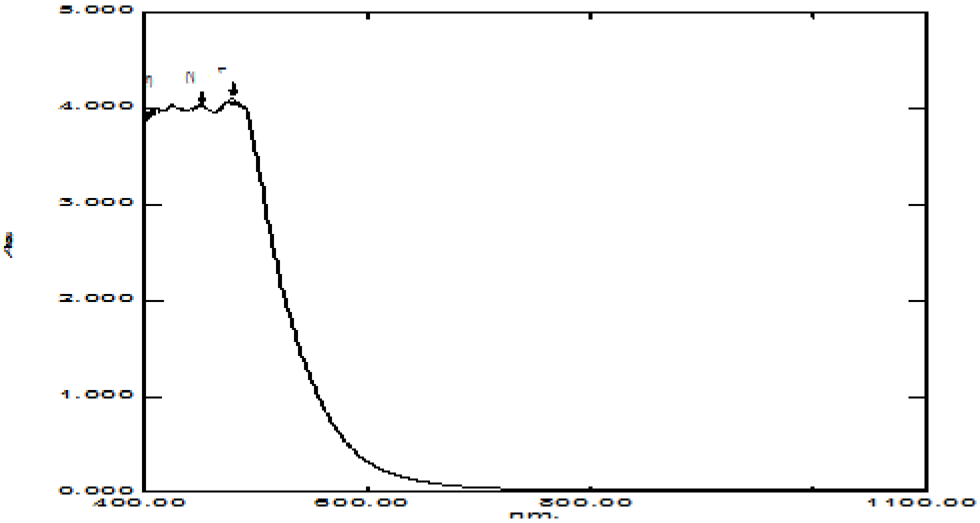

Figure 12. UV-Vis spectrum of Manganese complex

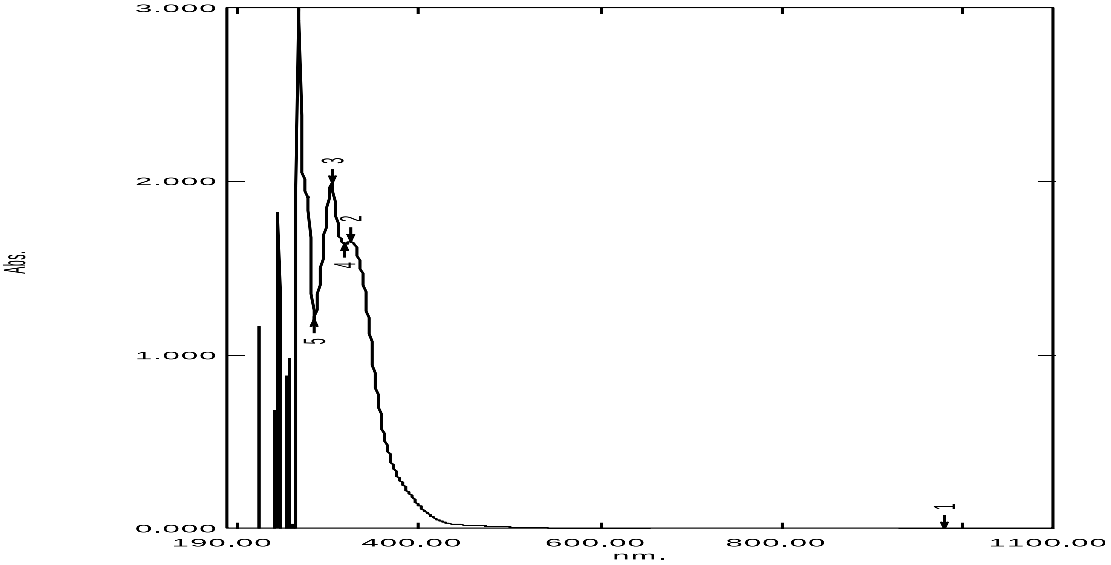

Figure 13. UV-Vis spectrum of Nickel complex

3.3 Nuclear magnetic resonance spectrum of ligand ^{13}C-NMR & ^{1}H-NMR

^{1}H-NMR spectrum in **Figure 14** and **Table 4** Demonstrates the next signals: two singlet signals at δ (2.5 and 3.5) ppm belongs to (2H) $2CH_2$ and (3H) $2CH_3$, Doublet signal at δ (9.55-9.60) ppm belongs to (2H) 2NH group, multiplet signal at (6.8-7.4) ppm belongs (8H) 2Ar- H. This measurement was carried out using DMSO-6 as solvent, which in turn gave a signal at (2.31-2.64). TMS is used as reference [24, 25]. ^{13}C-NMR spectrum in **Figure 15** demonstrates the next signals : (100.622 MHz, DMSO-d6) that gave a signal at 40 δ *ppm*: (110-130) δ *ppm* belong to (C_3-C_7) of aromatic ring, 140 δ *ppm* belong to (C_8 and C_9), 160 δ *ppm* belongs to C_{10} and C_{11} [26].

Table 4. ^{1}H-NMR spectral data of ligand (L)

Compound	^{1}H-NMR	δ *ppm*
$C_{22}H_{20}N_6O_2$ **L**	Singlet (2H) $2CH_2$	2.5
	Singlet (3H) $2CH_3$	3.5
	Doublet (2H) 2NH	9.55-9.60
	Multiplet (8H) 2Ar- H	6.8-7.4

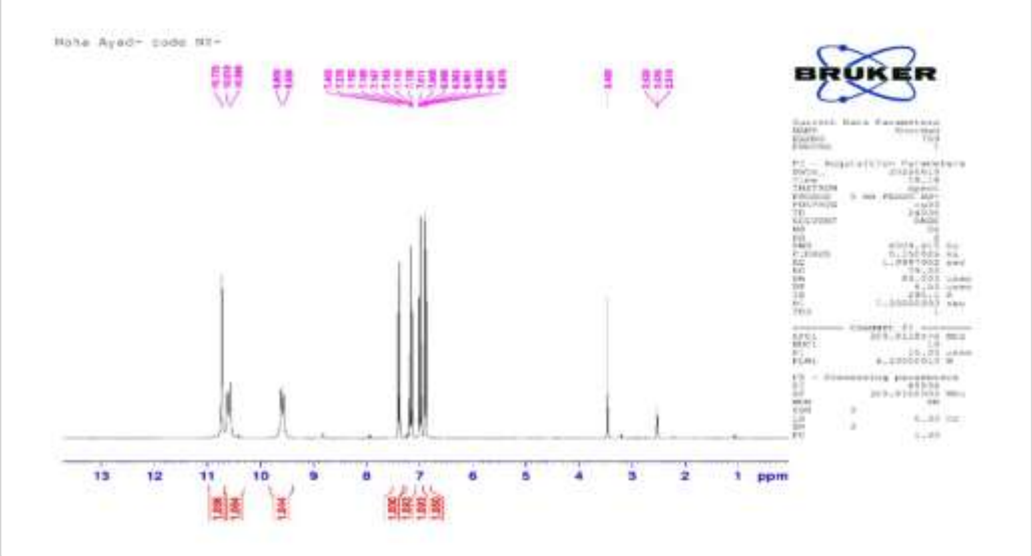

Figure 14. ^{1}H-NMR spectrum of ligand (L)

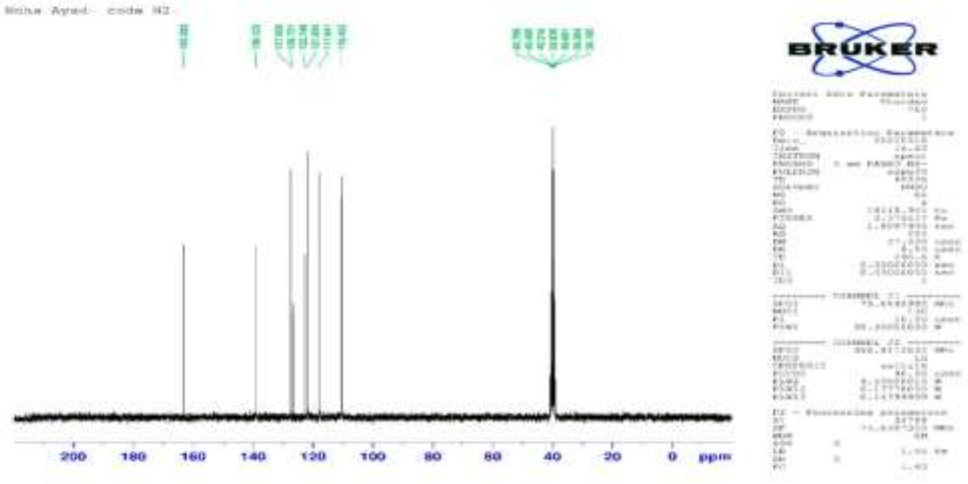

Figure 15. ^{13}C-NMR spectrum of ligand (L)

3.4 Mass Spectrum of Ligand

In coordination chemistry, mass spectroscopy is widely used as a potent structural characterization tool. Mass spectra fragmentation analogues for free Schiff base ligand [$C_{22}H_{20}N_6O_2$] were acceptable compared to its structure in scheme1. Characterized the mass spectrum an intense peak at 401.287 m/z that approximately matching its calculated molecular weight 400.44 m/z [27].

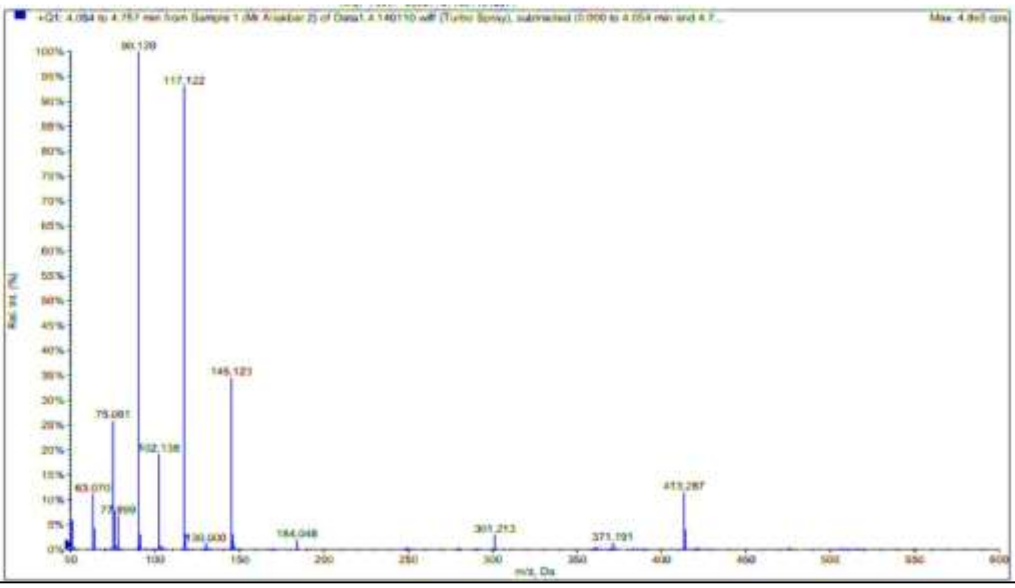

Figure 16. LC-MSS spectrum of ligand (L)

3.5 Bioactivity evaluation of the Ligand (L) and its complexes

Two types of bacteria were tested, (-Bacteria *(Escherichia_coli)* and +Bacteria *(Staphellococcus_aureus)*). The effect of the synthesized Schiff-species and its complexes on the mentioned bacteria were tested and compared in 0.001M (DMSO-solvent) as control and the results are recorded in **Table 4**. These results indicate that, the Schiff-species (ligand L) and Nickle complex has negative inhibitory action toward *Staphylococcus_aureus* bacteria besides, whereas the rest compounds were effective toward both types of bacteria [28]. All the details are demonstrated in **Figure 17**.

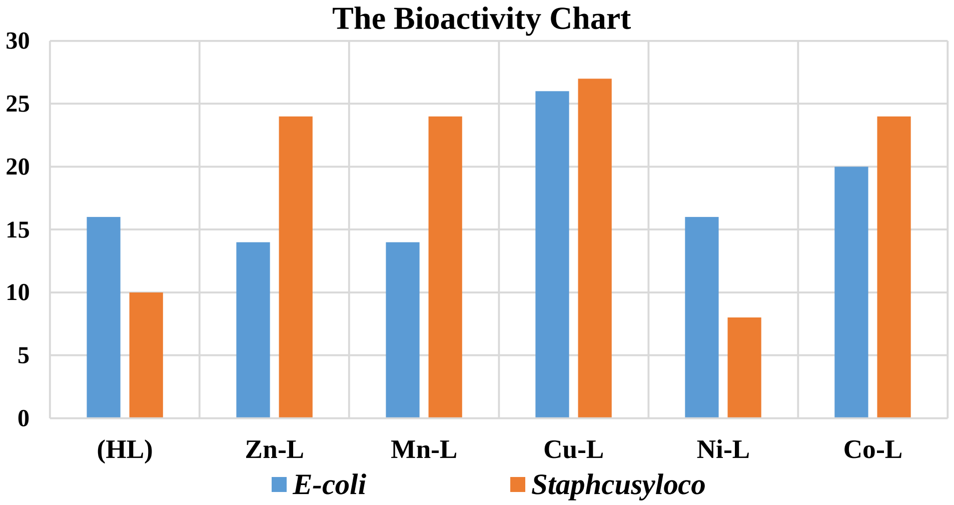

Figure 17. Biological evaluation-incomes

Table 4. Bioactivity results of Schiff-species-(L) and its complexes

Compound	*Escherichia_coli*	*Staphylococcus_aureus*
$C_{22}H_{20}N_6O_2$	16mm	10mm
$[Zn(L)Cl_2$	14mm	24mm
$[Mn(L)Cl_2(H_2O)_2]$	14mm	24mm
$[Cu(L)(Cl)(H_2O)]$	26mm	27mm
$[Ni(L)Cl_2(H_2O)_2]$	16mm	8mm
$[Co(L)Cl_2(H_2O)_2]$	20mm	24mm

4. Conclusion

Recently synthesized Schiff-bases_(L) (3Z,3'E)-3,3'-(((2E,5E)-hexane-2,5-diylidene) bis(hydrazine-2,1-diylidene)) bis(indolin-2-one) was successfully obtained using common condensation combination between hexane-2,5-dione and 2 moles of (Z)-3-hydrazineylideneindolin-2-one, Which forms massive stability Schiff-bases-(L)-complexes in specific conditions of preparation. Spectrally and using FTIR spectrometry this ligand and coordinates with employed metal ions (M^{2+}) = (Ni, Mn, Zn, Cu and Co) through both azomethine functional groups. The information of such apparatus indicating the formation of M-N and M-O belongs to aquatic water molecule that attaches to metal ions except for (M^{2+}) = Zn. Electronic spectra and conductivity measurements are also proved the distinguishable geometries of these complexes, which gave tetrahedral geometry for Zinc and Copper complexes and octahydral for the rest complexes. Additionally, we have also examined antibiotic character of Schiff-bases-(L) and complexes against (-Bacteria and +Bacteria) giving an acceptable inhibition efficiency as discussed in the article.

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