

Research Article

Synthesis, Characterization, and Biological Study of New Schiff Base and their Complexes for Ni (II), Cu (II) Metal Ions

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Abstract:

This research describes the synthesis of new ligands Schiff base and their complexes (L), (N₂) derived from 5, 5-Dimethyl-cyclohexane-1, 3-dione with 2-Aminopyridine. The ligands were characterized by the elemental microanalysis, melting point, spectroscopic method (FT. IR, ¹H-NMR, UV-Vis spectrometer. Reaction of these ligands with some metal salts Ni (II) Cu (II), form a variety of complexes of the general formula: [(M) (L) Cl₂], with : M (II) = Ni, and Cu. They were characterized by FT. IR, ¹HNMR, UV/Visbil spectroscopy, elemental micro analysis (C.H.N.), magnetic susceptibility, molar conductivity, melting point. The above measurements suggested the following geometry for the prepared complexes demonstrated that the ligand L complexes are non-electrolyte. A study of the biological effectiveness of ligands and complexes was conducted with two types of bacteria and one type: A-An Tetrahedral geometry around metal ion for the Cu, Ni complexes with the ligand [L]. Molar conductivity measurements also of bacteria, and they had an effect on the species studied.

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INTRODUCTION

A Schiff base is such nitrogen analog of an aldehyde [R₁-CH=O] or ketone [R₁-C=O-R₂] substances where the carbonyl group [C=O] is condensed with a primary amine [R₃-NH₂]; hence, it has been substituted with an imine containing the azomethine group [R₂-R₁-C=N-R₃], R₁, R₂, and R₃ may be aryl, alkyl, heteroaryl, or cycloalkyl, among others Figure 1. The imine group [-C=N-] in Schiff bases provides a distinct function in generating these

molecules with wide biological activity (Holder et al., 2005; Santhanam, 1996; Kalyanasundaram & Gratzel, 1998; Dagotto, 2003; De Groot, 2012).

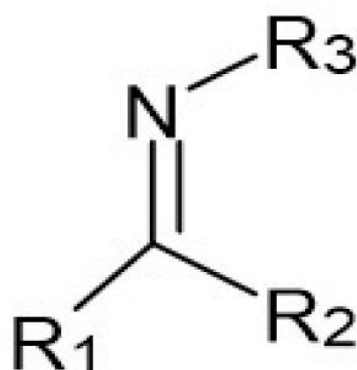


Figure 1. General Structure of Schiff Bases (R₁, R₂, and R₃ = alkyl or aryl).

Schiff bases are compounds which contain a carbon double bond with nitrogen atom connected to an aryl or alkyl group but not hydrogen atom. The first Schiff base was reported by Hugo Schiff by condensation of a carbonyl compound with primary amine in 1864 (Delaney et al., 2009). Schiff base is an analogue of a ketone or aldehyde in which the carbonyl group (C=O) has been replaced by an imine or azomethene group (De Medici, 2009). Schiff bases possess different names like azomethines, imine and arils, but in general are called aldimine derived from aldehydes and ketamine from ketons.

Application in Modern Technologies

Schiff bases, versatile compounds derived from the condensation of a primary amine and an aldehyde or ketone, find a multitude of applications across various scientific disciplines. Their diverse properties make them valuable in catalysis, medicinal chemistry, coordination chemistry, materials science, photovoltaics, sensing, corrosion inhibition, and environmental and industrial applications.

Photo- and thermo chromic properties of Schiff bases as well as their biological activity make them applicable in modern technology. Among others, they are used in optical computers, to measure and control the intensity of the radiation, in imaging systems, as well as in the molecular memory storage, as organic materials in reversible optical memories and photodetectors in biological systems (Diep, 2004).

Due to photo chromic properties, Schiff compounds could behave as photo stabilizers, dyes for solar collectors, solar filters. They are also exerted in optical sound recording technology (Di Matteo et al., 2005). Among others, worthy of interest in the properties associated with Schiff rules include: properties of liquid crystal (Efremov et al., 2004), chelating ability (Ehrenstein et al., 2006), thermal stability (Izatt et al., 1998), optical nonlinearity, and the ability to create the structure of a new type of molecular conductors using electrical properties to proton transfer. Because of its thermal stability Schiff bases can be used as stationary phase in gas chromatography. The optical nonlinearity of these compounds allows us to use them as electronic materials, opto-electronic (in optical switches) and photonic components (Saito, 2004). Due to the presence of the imine group, the electron cloud of the aromatic ring and electronegative nitrogen, oxygen and sulfur atoms in the Schiff bases molecules, these compounds effectively prevent corrosion of mild steel, copper, aluminum and zinc in acidic medium.

EXPERIMENTAL PART

Synthesis of organic ligands and their complexes

(1Z, 3Z) -5, 5-dimethyl 1-di (pyridin-2-yl) cyclohexane-1, 3-diimine

An ethanolic solution (15 ml) of 5,5-Dimethyl-cyclohexane-1,3-dione (1.4 g, 0.01mol) was added to a mixture containing an ethanolic solution (15 ml) of 2-Aminopyridine (1.88 g, 0.02mol), with glacial acetic acid as a catalyst. The mixture was heated in a water bath at 30–40 °C for 4hours, the resulting mixture was refluxed for 8 hours with stirring. A nutty light was formed and then re-crystallized from ethanol. The product was dried over anhydrous CaCl_2 in vacuum. Yield: 85% (0.8 g), mp. 200-201 °C.

RESULTS

Synthesis of L Complexes

Synthesis of Ni Complex

A solution of (L) (0.0.292 g, 0.001mol) in ethanol (20 ml), was added to a stirred solution of Ni (II) chloride (0.237 g, 0.001mol) in ethanol (20 ml). the mixture heated under reflux for 4 hours). Then the mixture was filtered and the precipitation was dried and washed with cold ethanol and water and dried at room temperature during (72 hours). A green product were obtained, m.p. (215-224°C), Yield: 83%.

Syntheses of Metals Cu (II)

Complexes

A similar method to that mentioned in (3.1.1) to prepare of Cu (II) complex. Table 1 shows the physical properties of the complexes and their reactant quantities.

Table 1. Some Physical Properties of the Prepared Ligand (L) and their complexes.

Comp. symbol	General Formula	Mwt g.mol-1	Color	Yield%
L	$\text{C}_{18}\text{H}_{20}\text{N}_4$	292.38	nutty light	85
LC_1	$\text{C}_{18}\text{H}_{20}\text{Cl}_2\text{NiN}_4$	421.98	brown	84
LC_2	$\text{C}_{18}\text{H}_{20}\text{Cl}_2\text{CuN}_4$	426.83	Deep yellow	80

Characterization of ligands (L) and its complexes

Schiff bases ligand was synthesized in double steps which in turn employed to prepare a new series of complexes for the next metal ions: Ni (II), and Cu (II), were characterization using different techniques: micro elemental analysis, FT-IR, UV-Vis, ^1H -NMR, F.A.A, molar conductivity magnetic moment, and thermal analysis.

FT-IR studies of ligand (L)

The infrared spectra of the ligand (L) and its complexes are recorded in the region 4000-400 cm^{-1} on KBr pellets. The FT-IR spectrum of ligand L is shown in Figure 2, describes the important characteristics absorption bands the strong absorption band at 3067 cm^{-1} which is assigned to the ν (C-H) aromatic stretching vibration, the bands at 2927 cm^{-1} and 2727 cm^{-1} which is assigned to the ν (C-H) aliphatic stretching vibration. The spectra show strong absorption band at 1663 cm^{-1} are assigned to the ν (C=N) of azomethine group stretching

vibration indicating the formation of the new Schiff base product, the strong band at 1527 cm^{-1} is assigned to $\nu(\text{C}=\text{C})$ aromatic stretching vibration of benzene ring, the bands at 1452 cm^{-1} is attributed to the $\nu(\text{C}=\text{N})$ stretching vibration of ring.

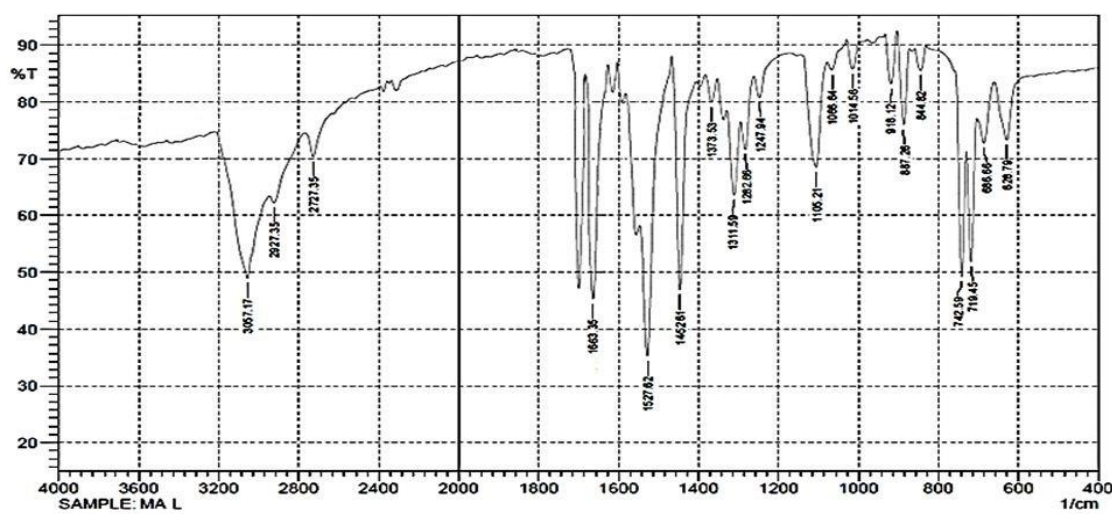


Figure 2. FT. IR Spectrum of ligand L

UV-Vis spectrum of the ligands (L)

UV-Vis spectrum of the ligand (L) in Figure 3, demonstrates the following electronic transitions: ($\pi \rightarrow \pi^*$) attributed to the presence of double bonds in the ligands and ($n \rightarrow \pi^*$) attributed to the presence of hetero atoms in the ligand's formula corresponded to (259 nm, 38610 cm^{-1}) for L and (213 nm, 43478.2 cm^{-1}), (260 nm, 38461.5 cm^{-1}) for L2 and (312 nm, 32051.2 cm^{-1}) for L and (297 nm, 33670.0 cm^{-1}) respectively (Belik, 2006).

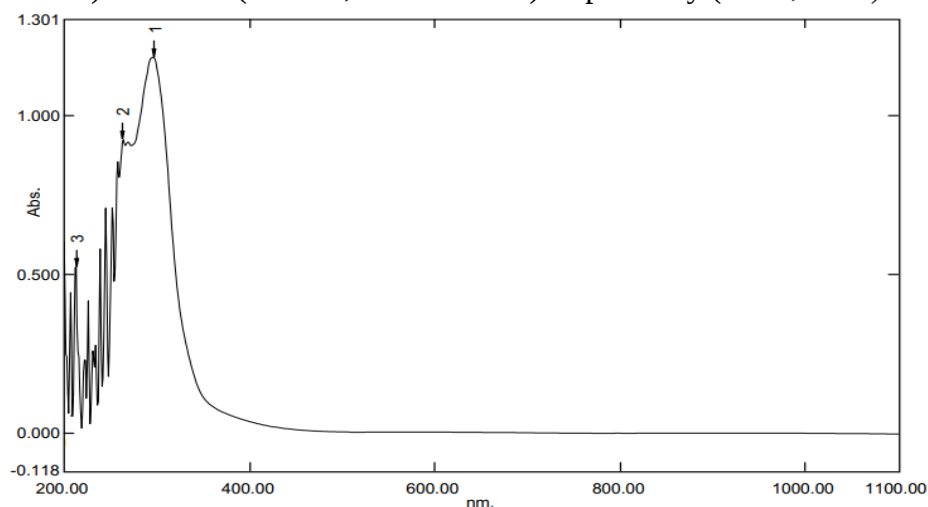


Figure 3. UV-Vis spectrum of Ligand L

Micro Elemental Analysis (C.H.N.) Metal Ratio and Chloride Content and Some Physical Properties

This technique is considered as one of the most important techniques used in the characterization of organic compounds and solid coordination complexes. The counting of the number of C, H and N atoms is carried out by this technique if included in the resultants. Flame atomic absorption FAAS technique is used to determine metal ratio and chloride content in the complexes. All the results are recorded in Table 2 All the theoretical results were so closer to the experimentally obtained results that prove the prepared mol ratios (1:1) (M: L) and supports the suggested molecular formula. The synthesized ligand and its

corresponded complexes were characterized by element microanalysis technique (C. H. and N) that gave compatible results with those that theoretically calculated (Berry, 1984).

Table 2. C. H. N Analysis % data metal ratio and chloride content: of the ligand (L) and its complexes.

No.	Molecular formula (M.wt) g.mol ⁻¹	Colour	m.p./°C	C	H	N	M
L	C ₁₈ H ₂₀ N ₄ 292.38	Nutty walnut	215	74.68 (73.94)	6.21 (6.8) 9	20.28 (19.16)	
LC ₁	C ₁₈ H ₂₀ Cl ₂ NiN ₄ 421.98	Brown	265	52.16 (51.23)	5.22 (4.78)	14.59 (13.28)	13.04 (13.91)
LC ₂	C ₁₈ H ₂₀ Cl ₂ CuN ₄ 426.83	Deep yellow	237	51.22 (50.65)	3.84 (4.72)	14.22 (13.13)	14.22 (14.89)

Nuclear magnetic resonance spectrum of ligand (L)

Nuclear magnetic resonance spectrum of the new Schiff base ligand was studied using dimethyl sulfoxide DMSO-d₆ as solvent and TMS as standard reference Figure 4 demonstrate the returns and chemical shifts of these spectra.

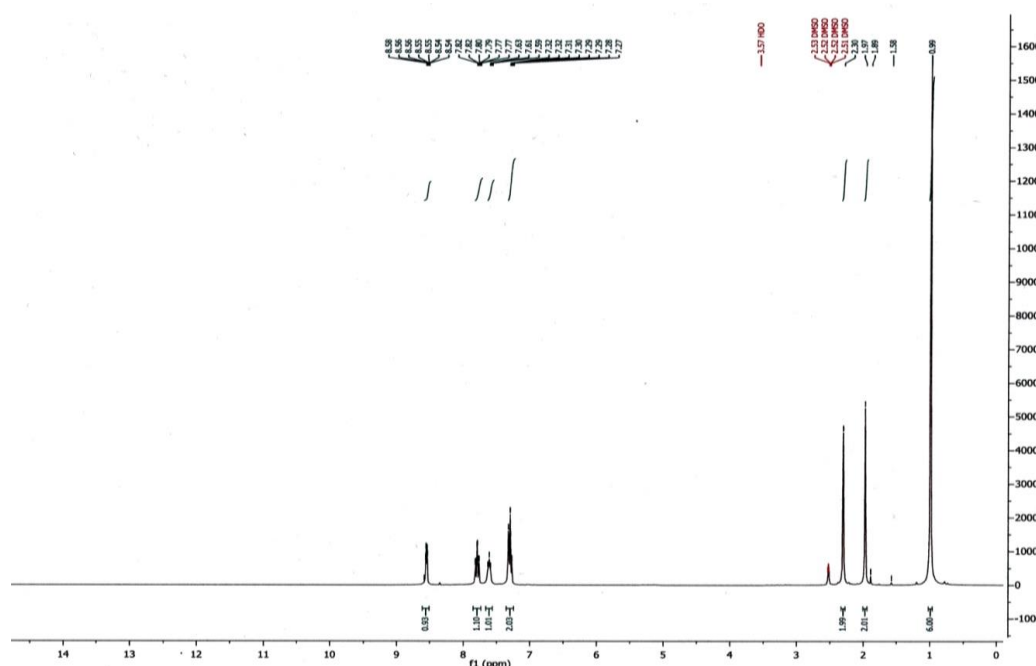


Figure 4. ¹H-NMR spectrum of ligand (L)

¹H-NMR spectrum of ligand (L)

- 0.99 ppm (2CH₃ (9 and 10), s, 6H)
- 1.89- 1.97 ppm (2CH₂ (7 and 11) , s, 4H)
- 2.30 ppm (CH₂ (13), s, 2H)
- 2.51-2.53 ppm (DMSO)
- 3.57 ppm (HDO)
- 7.27-7.63 ppm (4CH (2, 4, 15 and 17), m, 4H)

- 7.77-7.82 ppm (2CH (3 and 16), m, 2H,
- 8.54-8.58 ppm (2CH (5 and 18), d, 2H)

UV-Vis studies of compounds

Ultra violet-visible measurements are considered as important part in characterization of transition metal complexes through studying the obtained absorption bands in their electronic spectra, those complexes are distinguished by their colors because of the presence of partially occupied orbitals. The study of electronic spectra of the synthesized complexes aimed to determine the: geometries of these complexes, type and binding degree between ligand and metal ion which attaches to which through electronic effects present inside d-orbital of the metal ion or inside the molecular orbitals that formed between ligand and metal ion. Generally, electronic spectra of the transition metal complexes include the next absorption bands (Bersuker, 2006; Bersuker, 2010; Bhattacharjee et al., 2009; Biermann, 2005; Blanco-Canosa, 2009).

a- Organic compound transitions (Ln) : n = (1 and 2)

Involve peaks of high intensities in ultra violet region (200-380) nm which result from the available electronic transitions ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$) types (Blinc, 2011).

b- Charge transfer transitions (CT)

The intensity of which ranges from moderate to high because it is resulting from available electronic transitions occur between the ligand and the metal ion. Two types (L-M), (M-M), (L-L) and (M-L), (Bocquet, 1992).

c- d-d transition spectra

Spectra related with the metal are affected by the presence of the ligand, the spectrum shows wide peaks with low intensity in visible region, which result from the excitation of the electrons between two d-orbitals, this transition is not available according to Labort law, therefore the peaks are weak with low intensity. Depending on these bases, the electronic spectrum of the synthesized complexes in this research were deduced as illustrated in Table 3.

UV-Vis spectrum of Ni-complex

UV-Vis spectrum of Ni-complex (LC1), in Figure 3.17, demonstrates the next electronic transition peaks: at ultra violet region; ($\pi \rightarrow \pi^*$) at (298 nm, 33557 cm^{-1}) and at visible region the next transitions were observed, ($n \rightarrow \pi^*$) + (C.T) (M $\rightarrow$ L) electronic transition at (429 nm, 23310 cm^{-1}). And Ni-complex (LC1), in figure 3.18, demonstrates the next electronic transition peaks: at ultra violet region; ($\pi \rightarrow \pi^*$), ($n \rightarrow \pi^*$) and (C.T) at (206 nm, 48543.6 cm^{-1}), (292 nm, 34246.5 cm^{-1}) and (327 nm, 30581.0 cm^{-1}) respectively. The previous transitions resemble to those found in ligand itself and the noticeable shifting attributes to the occurrence of coordination with metal. Other electronic transitions attribute to the transitions in metal itself (d-d) transitions, those are as follows: $^3T_{1(F)} \rightarrow ^3T_{2(F)}$, $^3T_{1(F)} \rightarrow ^3A_2$ and $^3T_{1(F)} \rightarrow ^3T_{1(P)}$ observed at: (512 nm, 19531 cm^{-1}), (676 nm, 14792 cm^{-1}) and (778 nm, 12853 cm^{-1}) respectively which support the tetrahedral geometry for Ni-Complex (LC1), but transitions, those are as follows: $^3A_{1g} \rightarrow ^3T_{2g(P)}$, $^3A_{1g} \rightarrow ^3T_{1g}$ and $^3A_{1g} \rightarrow ^3T_{1g}$ observed at: (400 nm, 25000 cm^{-1}), (609 nm, 16420.3 cm^{-1}) and (623 nm, 16051.3 cm^{-1}) respectively which support the Octahedral geometry for Ni-Complex (LC1), (Bode , 2007; Cruickshank et al., 1975; Sharma et al., 2013; El-Sherif & Synthesis, 2009; Angelusiu et al., 2010; Rous et al., 2011).

UV-Vis spectrum of Cu-complex

UV-Vis spectrum of Cu-complex (LC2), demonstrates the next electronic transition peaks at Ultraviolet and visible region; ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$) + (C.T M $\rightarrow$ L) electronic transitions at (307 nm, 23573 cm^{-1}) and (493 nm, 20283 cm^{-1}). And Cu-complex (LC2), in figure 3.20,

demonstrates the next electronic transition peaks at Ultraviolet region; ($\pi \rightarrow \pi^*$), ($n \rightarrow \pi^*$) and (C.T M $\rightarrow$ L) electronic transitions at (213 nm, 46948.3 cm⁻¹), (289 nm, 34602.0 cm⁻¹) and (342 nm, 29239.7 cm⁻¹). The previous transitions are resembled to those that found in ligand itself and the noticeable shifting attributes to the occurrence of coordination with metal. Other electronic transition attributes to the transition in the metal itself (d-d) transitions; this transition is $^2T_2 \rightarrow ^2E$ at (729 nm, 13717 cm⁻¹) which support the tetrahedral geometry, but transitions, those are as follows: $^2E_g \rightarrow ^2T_{2g}$ observed at: (548 nm, 18248.1 cm⁻¹) respectively which support the Octahedral geometry for Cu-Complex (LC2), (Karimi-Jaberi & Takmilifard, 2013).

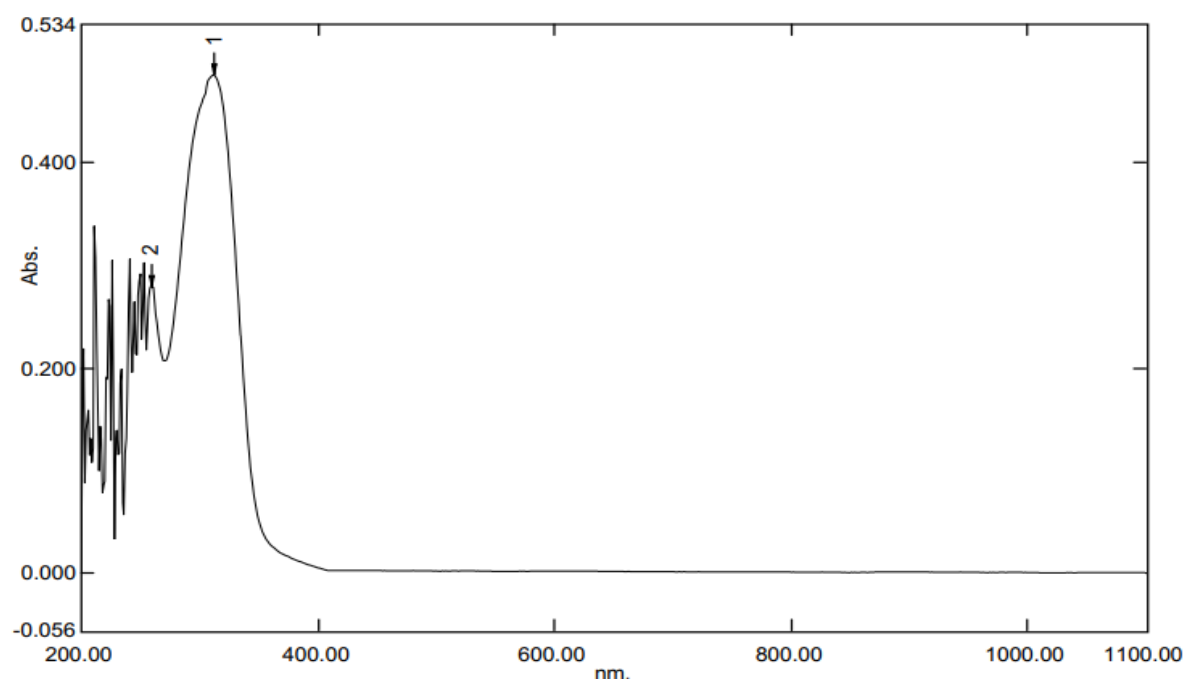


Figure 6. UV-Vis spectrum of Ligand L

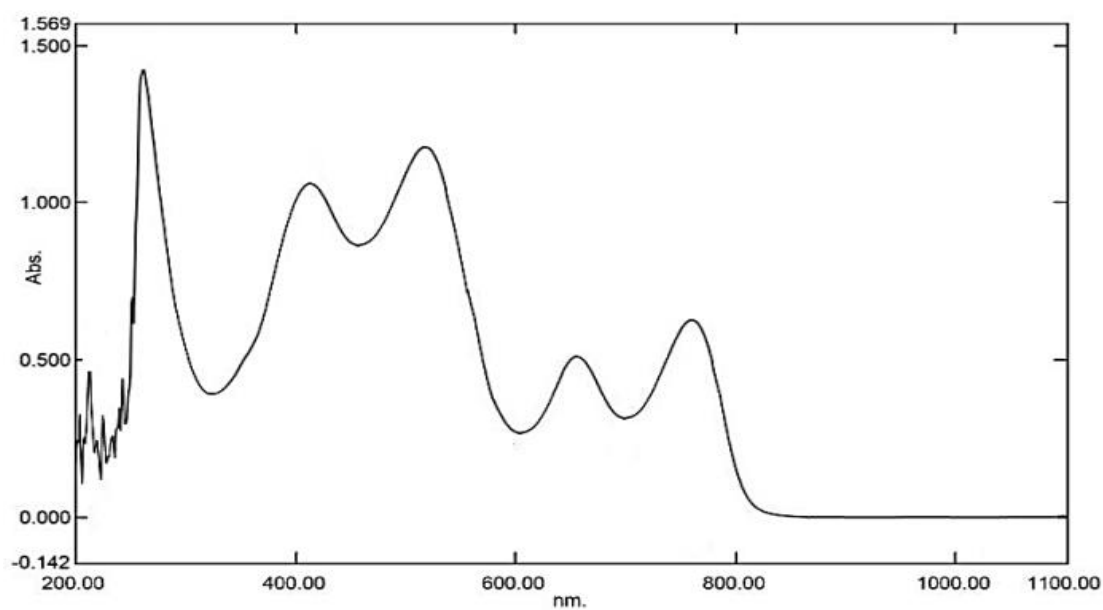


Figure 7. UV-Vis spectrum of Ni-complex (LC1)

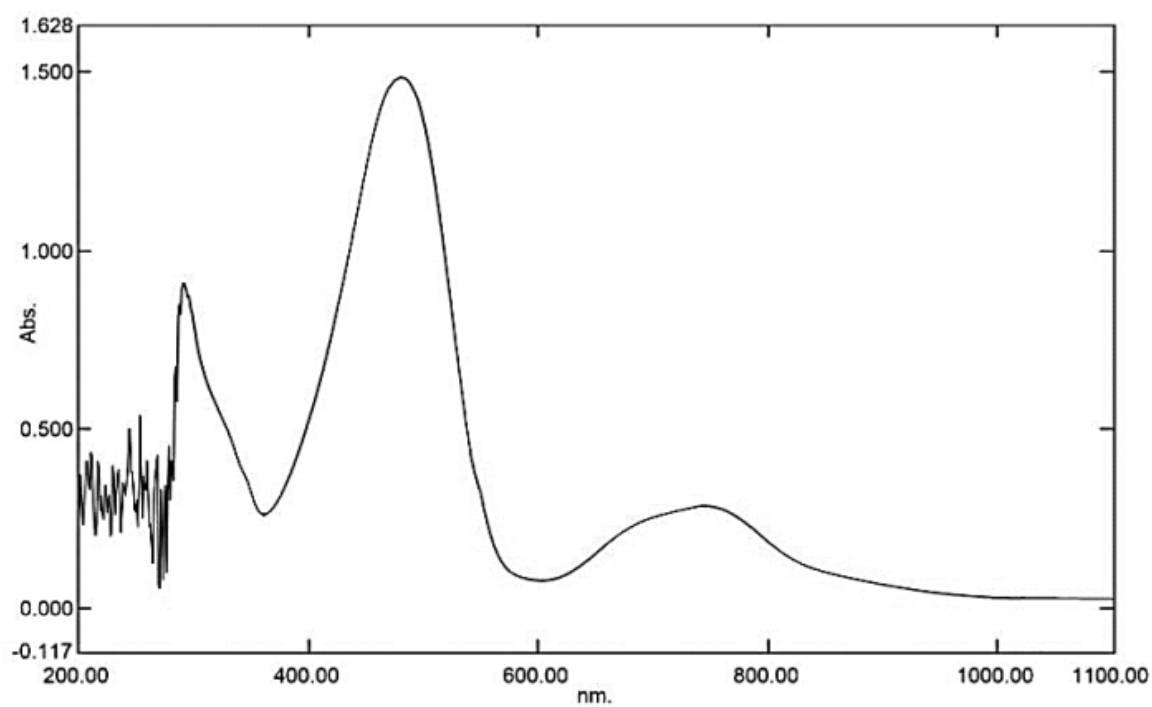


Figure 8. UV-Vis spectrum of Cu-complex (LC2)

Table 4. UV-Vis and conductance data of ligands and complexes.

Compound	λ_{max} (nm)	νcm^{-1}	ABS.	ϵ_{max} $\text{L cm}^{-1} \text{ mol}^{-1}$	Assignment
L	259 312	38610.0 32051.2	0.280 0.485	280 485	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$
Ni-complex (LC ₁) Tetrahedral	298 429 512 676 778	33557 23310 19531 14792 12853	1.423 1.093 1.203 0.505 0.614	1423 1093 1203 505 614	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^* + (\text{C.T}) (\text{M} \rightarrow \text{L})$ ${}^3\text{T}_1(\text{F}) \rightarrow {}^3\text{T}_2(\text{F})$ ${}^3\text{T}_1(\text{F}) \rightarrow {}^3\text{A}_2$ ${}^3\text{T}_1(\text{F}) \rightarrow {}^3\text{T}_1(\text{P})$
Cu-complex (LC ₂) Tetrahedral	307 493 729	32573 20283 13717	0.901 1.496 0.368	901 1496 368	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^* + (\text{C.T}) (\text{M} \rightarrow \text{L})$ ${}^2\text{T}_2 \rightarrow {}^2\text{E}$

FT-IR studies of ligand (L) and its complex

The infrared spectra of the ligand (L) and its complexes are recorded in the region 4000-400 cm^{-1} on KBr pellets. The FT-IR spectrum of ligand L is shown in Figure 9, table 6 describes the important characteristics absorption bands the strong absorption band at 3067 cm^{-1} which is assigned to the ν (C-H) aromatic stretching vibration, the bands at 2927 cm^{-1} and 2727 cm^{-1} which is assigned to the ν (C-H) aliphatic stretching vibration. The spectra show strong absorption band at 1663 cm^{-1} are assigned to the ν (C=N) of azomethine group stretching vibration indicating the formation of the new Schiff base product, the strong band at 1527 cm^{-1} is assigned to ν (C=C) aromatic stretching vibration of benzene ring, the bands at 1452 cm^{-1} is attributed to the ν (C=N) stretching vibration of ring (Ramon et al., 2003).

The infrared spectra of [Ni(L)Cl₂] complex is shown in figure 10. The important characteristics absorption bands show at 3106 cm^{-1} , 2924 cm^{-1} , 2864 cm^{-1} and 1450 cm^{-1} are attributed to the ν (C-H) aromatic, ν (C-H) aliphatic and ν (C=N) ring stretching vibration, respectively. The band attributed to ν (C=N) of azomethine group stretching vibration in the spectrum of ligand (L) was shifted to lower wave number in the metal complexes and appeared at 1631 cm^{-1} , these shift attributed to confirm the coordination of the ligand through the nitrogen of azomethine groups with metal ion, further, the new intensity band appeared in the 574 cm^{-1} , 530 cm^{-1} region assignable to ν (M-N). The infrared spectra of [Cu(L)Cl₂] complex is shown in figure 11. The important characteristics absorption bands show at 3174 cm^{-1} , 2924 cm^{-1} , 2854 cm^{-1} and 1448 cm^{-1} are attributed to the ν (C-H) aromatic ν (C-H) aliphatic and ν (C=N) ring stretching vibration, respectively. The band attributed to ν (C=N) of azomethine group stretching vibration in the spectrum of ligand L was shifted to lower wave number in the metal complexes and appeared at (1649 cm^{-1}), these shift confirm the coordination of the ligand through the nitrogen of azomethine groups with metal ion, The presence of new medium intensity bands appeared in the 597 cm^{-1} region assignable to ν (M-N).

Table 6. Infrared spectral data of the ligand (L) and their complexes.

compound	ν (CH) arom.	ν (C-H) aliph.	ν (C=C)	ν (C=N) azomethine	ν (C=N) ring	ν M-N
Ligand (L)	3067	2927, 2727	1527	1663	1452	-
[Ni(L)Cl ₂]	3193	2924, 2852	1504	1640	1448	588, 526
[Cu(L)Cl ₂]	3067	2927, 2833	1504	1639	1450	594, 526

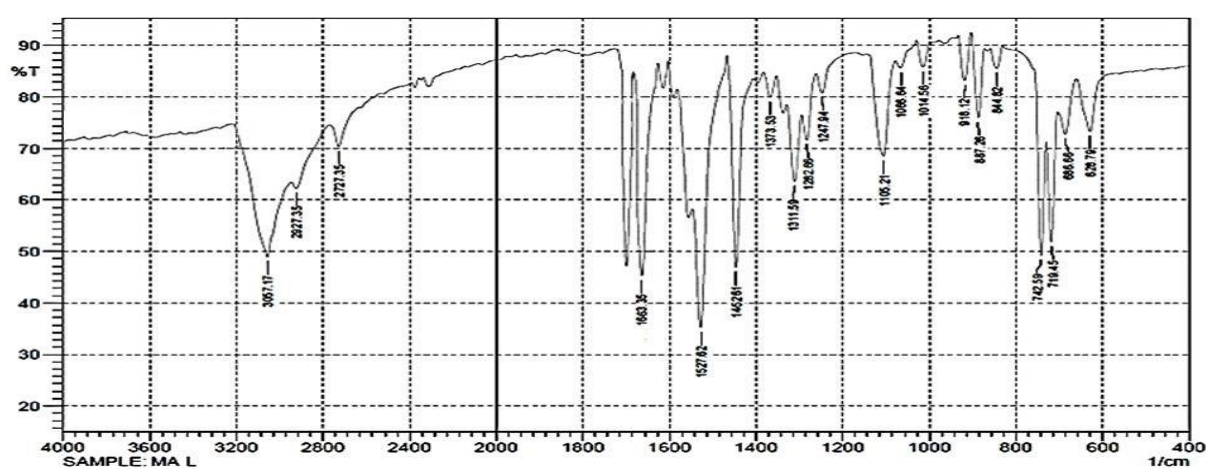


Figure 9. FT. IR Spectrum of ligand L

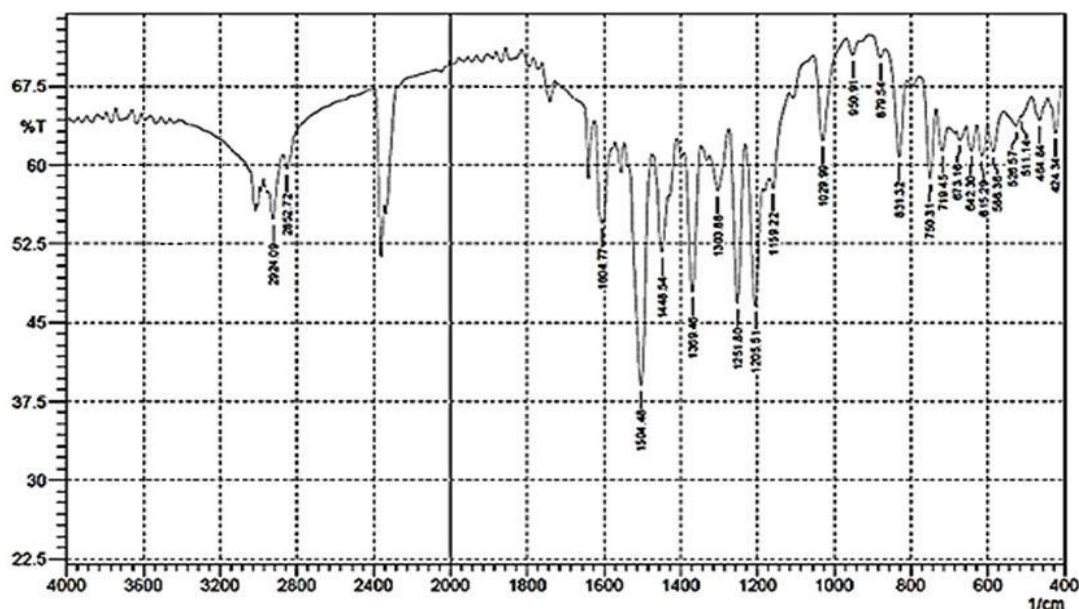


Figure 10. FT. IR Spectrum of Ni-complex

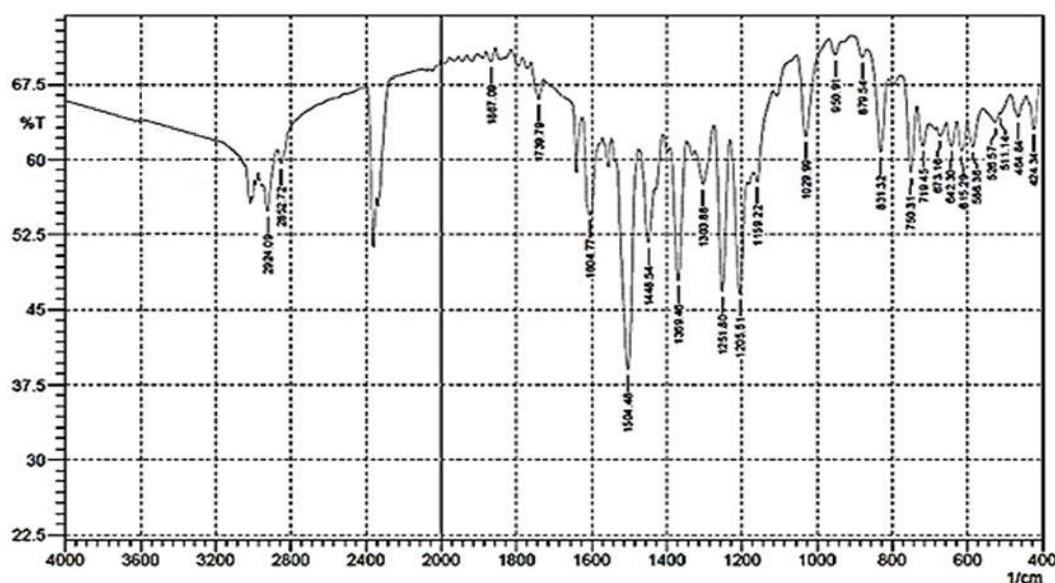


Figure 11. FT. IR Spectrum of Cu-complex

BIOLOGICAL ACTIVITY

The focus on complexes ions transitional elements and because of its effects in microbiology, requires knowledge of the reasons for its effectiveness and them to have their solutions ability to dissolve the outer cell wall leads to osmoses fluids This cell death is complexes that affect the cell wall of the more factors selective and relevant Index therapeutic high, due to the difference in composition between the wall of the host cell (Chandra et al., 2009). the possibility of establishing a single metallic items in the cell because of its association with the power of about derivative replaces the metal within a complex installation so that the cell deprived of one of its members metallic, it has been attributed Hits to osmoses cells influence where the membrane is affected half osmoses stopping Activity membrane Cytoplasm and then hinders the necessary vehicular traffic the metabolism, as well as the presence of active groups within the installation of effective biological ligand may lead to the formation of complexes with the items in the cell body, such as ions of both Ni (II), Cu (II), that need the bacterial cell and result in the loss of these items to cell death (Kale & Synthesis, 2012).

The method used for the spread of the complexes in the center Nutrient Agar was a way of drilling (wells) and proposed by the world (Egorove) in 1985 to test the sensitivity of the bacteria against compounds, where Action includes four drill diameter 8mm mediated piercing Faeli Cork Borer then measured the inhibition of Qatar brokered the ruler (Kamran et al., 2013). In this study take two type (bacteria and fungus).

E-Coli and Staph (bacteria)

E-Colli

Escherichia coli (E.coli) is a bacterium commonly found in the intestines of humans and warm-blooded animals. Most strains of *E. coli* are harmless. However, some strains, such as Shiga toxin-producing *Escherichia coli*, can cause severe foodborne illness. This bacterium is transmitted to humans primarily through the consumption of contaminated food, such as raw or undercooked ground meat products, raw milk, and contaminated raw vegetables and sprouts (Yousef et al., 2014).

This *Escherichia coli* produces a toxin known as Shiga toxin because it is similar to the toxin produced by the *Shigella dysentery* bacterium. Shiga toxin-producing *Escherichia coli* can grow at temperatures ranging from 7°C to 50°C, and the optimum temperature for its

growth is 37°C. Some species of *E. coli* that produce Shiga toxin can grow in acidic foods with a pH of 4.4 at most, and in foods with a water activity of 0.95 as a minimum (Alam et al., 2014).

Dr. Nipponi Rajapaksa says that *E. coli* bacteria can cause some symptoms in the stomach, such as abdominal pain and nausea. But it may get worse. “A specific type of *E. coli*, known as O157:H7, can cause bloody diarrhea and is associated with a condition that may cause kidney damage, especially in young children. Figure 12 explain the shape of staph (Al-Hamdani et al., 2015).

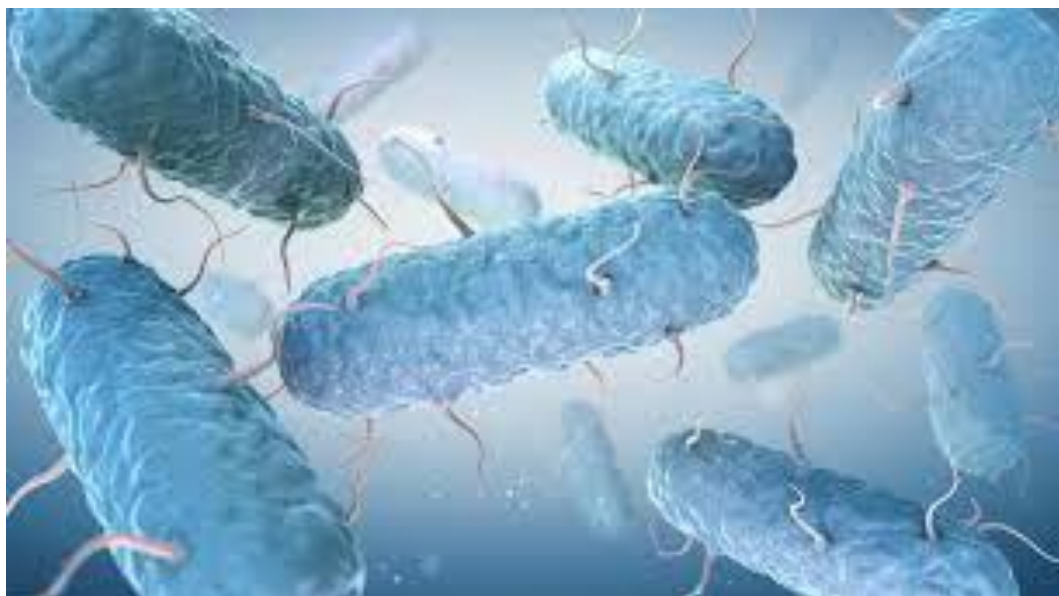


Figure 12. Colli (bacteria)

Staph (bacteria)

Staph infections are caused by staphylococcus bacteria. These types of germs are commonly found on the skin or in the nose of many healthy people. Most of the time, these bacteria cause no problems or cause relatively minor skin infections. But staph infections can turn deadly if the bacteria invade deeper into your body, entering your bloodstream, joints, bones, lungs or heart. A growing number of otherwise healthy people are developing life-threatening staph infections (Manolikakes, 2008).

Treatment usually involves antibiotics and cleaning of the infected area. However, some staph infections no longer respond, or become resistant, to common antibiotics. To treat antibiotic-resistant staph infections, health care providers may need to use antibiotics that can cause more side effects.

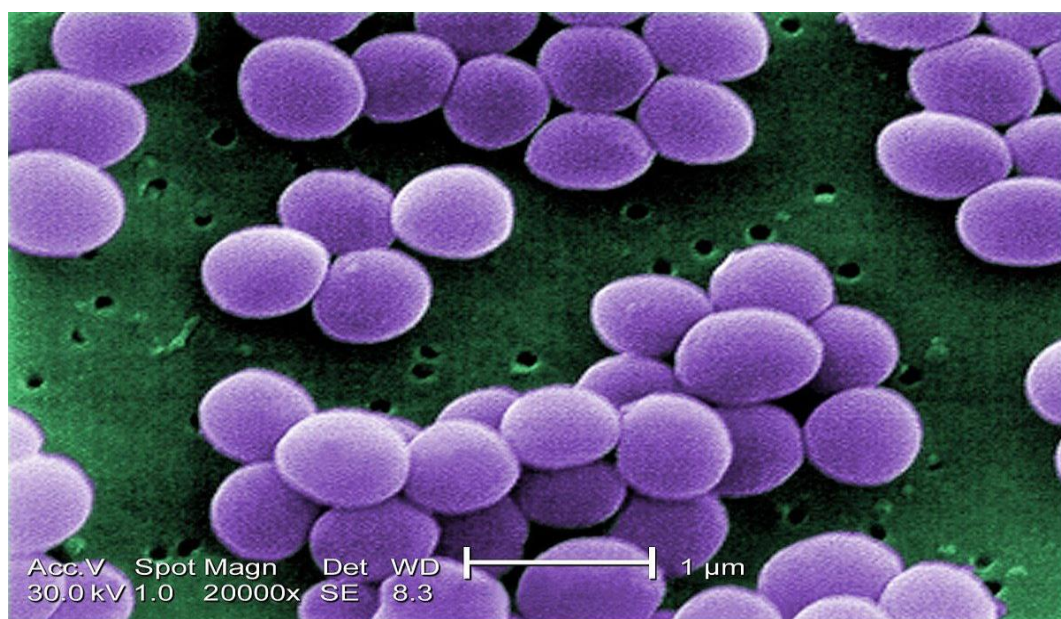


Figure 13. Staph (bacteria)

Table 7 explain the antibacterial activity of the ligand L and their complexes in different concentration (10mg/ml and 5mg/ml) with bacteria (E-Colli and Staph) and Aspergillus niger (fungus). The effective area in (L) show in Figure14.

Table 7. Antibacterial activity of the some ligand L and L₂ complexes.

Ligand and their complexes	10mg/ml effective area (E-Colli)	5mg/ml effective area (E-Colli)	10mg/ml effective area (staph)	5mg/ml effective area (Staph)
L	2.8mm	3.1mm	2.7mm	3mm
L ₁ Ni	1.3mm	-	3mm	2.5mm
L ₁ Cu	2.1mm	1.7mm	2.2mm	2.6mm

According to the zone of inhibition diameter in mm (% inhibition): staph in L and there complexes more effective than E-colli.

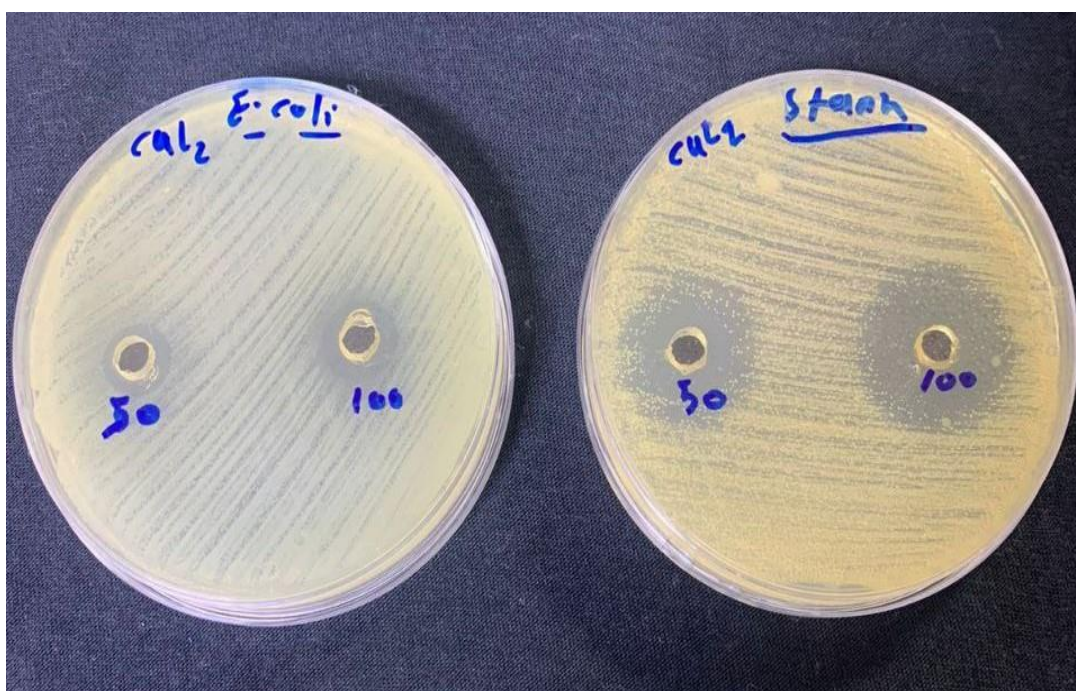
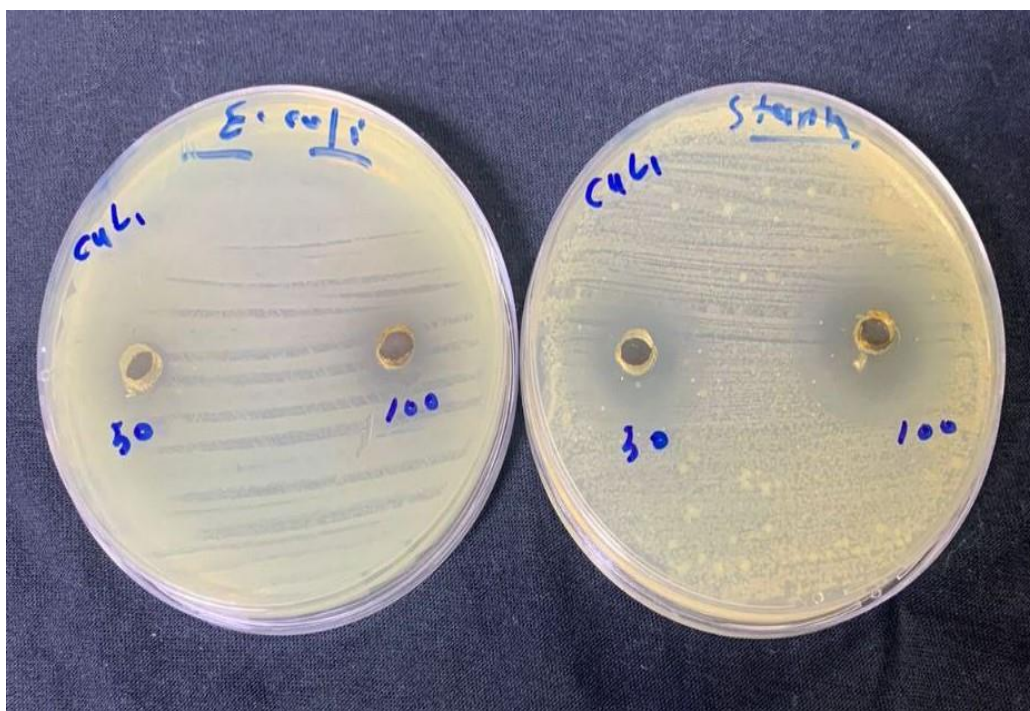


Figure 14. Bio activity for ligands and complexes

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