# "New Ligand a Type of Schiff Bases Derived from" 2- Mercaptobenzothizole and Its "Complexes with Some Metal Ions": Preparation, Characterization, and Evaluation of Biological Activity

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**Abstract**

In this study, a new Schiff base compound was synthesized and identified as a ligand (LH). A phenyl ketone with the molecular formula (C48H35N5O2S) -2-(2-(4-(1-(4'-("benzothiazole-2-ylamino)-[1, 1'-biphenyl]-4-yl) imino) ethyl) phenyl) imino)-1,2-diphenylethylidene) amino) benzoic acid . In order to generate complexes with metal ions pd (II), Hg(II), Co(II), and Ni(II), a newly synthesised ligand called LH was used. In addition, metal salts was utilised in order to react with a ligand. In all, there were four stages involved in the synthesis of ligand (LH). The product had a 79.85% yield and 100-102°C melting point. In order to determine the identities of ligands and the complexes they form, analytical techniques such as the FT-IR, UV-Vis, melting points, molar conductivity, CHN atomic absorption, magnetic sensitivity, XRD, and FESEM have been used. An investigation of the biological activity of Ligand and its complexes revealed that both were successful in inhibiting the growth of the bacteria under study.

Keywords: Benzothiazole, Schiff bases, Benzothiazole- Schiff bases complexes, biological activity of Benzothiazole.

## 1. Introduction

Organic compounds with benzene rings and thiazole rings are known as benzothiazoles [1]. benzothiazoles are heterocyclic organic compounds that have sulfur and nitrogen as their basic structure [2-4] Drug discovery and production rely on its wide range of applications in the synthesis of a wide range of biologically active compounds and antibiotics, such as those used to treat infections like tuberculosis and TB, as well as malignancies and fungal growths. Industrial and analytical fields use benzothiazole and its derivatives [5, 6]. As a result of these vital activities, and benzothiazole and its derivatives have anti-microbial, anti-cancer properties as well as antibacterial and anti-bacterial properties [7-9]. Schiff bases are organic compounds that contain the amine group (Azomethine) Hugo Schiff, a German chemist, first developed the Sheff rules in 1864 [10, 11]. It was made by combining an aldehyde or ketone with the primary amine. One of Schiff bases most important properties is its stability [12]. It's critical to the body's building process, and the Schiff bases are crucial to coordination chemistry because they're involved of the preparation of numerous complexes [13].

## 2. Materials and Methods

"Chemicals were imported from Sigma-Aldrich", Merck, MSDS, and BDH. The UV-visible spectra were recorded between 200-1000 nm using instruments from these companies (ShimadzuU.V-165PCS spectrophotometer). On the Fourier Transform Varian Spectrometer, The Shimadzu FTIR 8400S Spectrophotometer was used to record FTIR spectra in the 400-4000 cm-1 range (Japan). It was found that the Stuart melting point could be used to calculate the melting points of all compounds. At room temperature, the Balance Magnetic Susceptibility Model MSB-MKI was used in order to carry out measurements of magnetic susceptibility. The AA-6300 was used for the purpose of determining the metal percentage, and the EA-300.mth was utilized for the purpose of recording the elemental analysis.

### Preparation of the Ligand

#### Four steps were required to make a ligand:

Compound A was prepared from the reaction of (1.67 g, 0.01ml) of the compound 2-mercpto-benzothiazole dissolved in (25 ml) of "absolute ethanol as a solvent" in a round flask capacity (100 ml), to which a solution was added consisting of dissolving ("1.84 g, 0.01mol") of benzidine in absolute ethanol., and the reaction was followed up using lead acetate paper. When the lead acetate paper started to turn black, this indicated that the reaction had completed. After being cooled to "room temperature", "the solution was filtered and then cooled again". Using hot absolute

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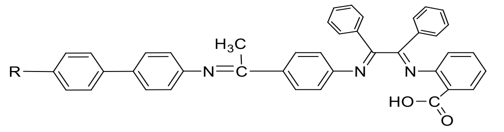
ethanol, the non-reacting materials were removed from the sediment by recrystallizing it. Allow the sediment to dry before collecting and weighing it. (yield% = 70%) and (m.p.190-102 °C)
In a round flask with a capacity of 100 ml, compound (B) was prepared by reacting 2.1 g of compound (A), 0.01 moles, with 25 ml of absolute ethanol as a solvent, and then adding a solution of dissolving 1.37 g (0.01 moles) of anthranilic acid in "25 mL of absolute ethanol". Five to six drops of glacial acetic acid were added to the mixture. They refluxed for eight hours, cooled, filtered, and recrystallized. The precipitate was collected and gave a product percentage (65%), and the melting point ranged between 90 and 92 degrees Celsius.
Compound (C) was prepared in the same way as before by dissolving (1.59 gm, 0.005 mol) of the compound (A) and reacting with (1.35 gm, 0.005 mol) of the compound 4-aminoacetophenon. After the reflux process, precipitation was observed, after which, the precipitate was filtered, dried and recrystallized from absolute ethanol. Finally, the precipitate was collected, yielding sixty percent and a temperature of one hundred to ninety-eight degrees Celsius.
Preparation of the ligand (LH) by dissolving 1.65 g, 0.005 mol ("of compound" (B) in (25 ml) of absolute ethanol by continuous stirring and adding to it a solution of 2.17 g, 0.005 mol (of compound (C) dissolved in (15 ml) of absolute ethanol) [5, 6] drops of glacial acetic acid were added to the solution of compound (B). The mixture was raised for [8] hours, cooled further, by cooling the mixture, where it was observed that a precipitate was filtered and dried, re-crystallized from absolute ethanol, and then the precipitate was collected, giving a result of (79%). Melting point (102-100°C).




2-((2-((4-(1-((4'-(benzo[*d*]thiazol-2-ylamino)-[1,1'-biphenyl]-4-yl)imino) ethyl)-phenyl)imino)-1,2-diphenylethylidene)amino)benzoic acid (LH)
$C_{48}H_{35}N_5O_2S$

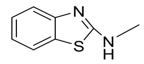
R: 


Figure (1): The chemical structure of the ligand 2-((2-((4-(1-((4'-( benzotheazol-2-ylamino )- [1, 1'-biphenyl]-4-yl) imino) ethyl) phenyl) imino)-1, 2-diphenylethylidene) amino) benzoic acid (LH).

## Synthesis of the metallic Complexes

The complexes of the ligand (LH) were prepared according to the following general method:
The complexes were prepared in different percentage, as the cobalt (II) and nickel(II) complexes were prepared in a ratio of (2:1) (ligand: metal) and a ratio of (1:1) (ligand: metal) for the complexes of palladium (II) and mercury (II), (0.74 g, 0.1 mm) of the ligand (LH) dissolved in 10 ml absolute ethanol and added to it the weights shown in Table (1) for the salts of the metal ions cobalt (II) and nickel(II) dissolved in 5 ml absolute ethanol, and dissolved (0.37 g, 0.05 mmol) of the ligand (LH) of the same volume as above and adding to it the weights shown in the table (1) for the salts of the metal ions palladium (II) and mercury (II), the mixture was refluxed for two hours to form a colored precipitate, collected, dried and recrystallized from absolute ethanol. Table (2) shows some physical properties of Ligand (LH) and its metal complexes

Table (1): weights (gm) taken from the prepared ligand (LH) and the metal salts used in the preparation of the complexes

| | [12], [L:M] | | | [11], [L:M] | | |
|---|---|---|---|---|---|---|
| Compound | (LH) | CoCl2.6H2O | NiCl2.6H2O | (LH) | PdCl2 | HgCl2 |
| Wt. (g) | 0.74 | 0.06 | 0.07 | 0.37 | 0.09 | 0.14 |

Table (2): " Physical" properties of the ligand (LH) "and its complexes".

| Compounds | Color | M.P °C | Yield (%) | M.wt gm/mole | "Elemental microanalysis" (%) Calcd. (Found) | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | C% | H% | N% | S% | M% |
| Liggand (LH) | yellow | 100-102 | 79.85 | 744.90 | 77.32 (77.26) | 4.62 (4.23) | 9.39 (9.59) | 4.30 (4.23) | |
| [Co(L)2]. H2O | dark yellow | 107-109 | 55 | 1568 | 73.66 (73.52) | 4.55 (4.45) | 8.84 (8.89) | 4.08 (4.07) | 3.76 (3.66) |
| [Ni(L)2]. H2O | yellow | 115-117 | 55.6 | 1568 | 73.66 (73.56) | 4.54 (4.44) | 8.84 (8.87) | 4.08 (4.04) | 3.76 (3.63) |
| [Pd(L)Cl]. H2O | brown | 170-173 | 82.5 | 903.5 | 63.72 (63.22) | 3.98 (3.79) | 7.74 (7.87) | 3.54 (3.32) | 11.71 (11.62) |
| [Hg(L)Cl] | lead | 140-142 | 82 | 979.5 | 58.81 (58.62) | 3.47 (3.46) | 7.14 (7.56) | 3.26 (3.16) | 20.4 (——) |

## Antibacterial activity

S.aureus and E.coli were used to test ligands and metallic complexes biological activity. These microorganisms are disease-causing. The ligand and its metallic complexes were dissolved in dimethyl sulfoxide (DMSO) and prepared at 250 and 500 mg/L for biological activity. Mueller-Hinton agar (38 g) was dissolved in 1 liter of distilled water per company instructions. Loopful was used to disseminate bacteria on the plates that contained Mueller-Hinton agar, and an alcohol-"sterilized Corkborer was used in order to drill holes of 6 millimetres in diameter". The inhibitory zone width was measured in millimetres after being kept in the incubator for 24 hours at 37 degrees Celsius.

# 3. Results and Discussion

Infra-red Spectra of the Synthesized Ligand "and their metal complexes"
The following table provides a listing of the values of the IR spectra of the ligand and the complexes that include them [3]. Comparing and taking into account

the bands that may be seen in the spectra of both the complexes and the ligand is part of the process. The azomethine group υ (C=N) belonging to the Schiff base, which appeared at (1674 cm-1) in the spectrum of the free ligand shifted towards lower or higher frequencies compared to the complex spectra, as it gave a shift of (7-11 cm-1) after coordination and appeared at (1664, 1667, 1685, 1685 cm-1) in the spectra of the complexes of cobalt (II), nickel (II), palladium (II) and mercury (II), respectively [14].The disappearance of the absorption band of the hydroxyl group of carboxylic acid after losing its proton as a result of coordination with the metal ion and the appearance of new bands in the range (455-476 cm-1) belonging to the υ (M-O) group in the spectra of the complexes of cobalt (II), nickel (II), palladium (II) and mercury (II) ().New bands appeared at (506-586 cm-1), these bands belongs to the υ (M-N) group, as a result of the coordination of the ligand with the metal ions through the nitrogen atoms of the azomethine group [15].The appearance of wide bands at (3409, 3374, 3425 cm-1) belongs to the hydroxyl group of the water of crystallization molecules in the spectra of the complexes of cobalt (II), nickel (II) and palladium (II), respectively [16].

Table (3): "The Infrared frequencies" (in cm-1) "data of ligand and its complexes".

| Compounds | υ(O-H) Carboxylic υ(O-H) (H2O) | υ(N-H) 2oamine | υ(C-H) aromatic | υ(C-H) aliphatic | υ(C=O) Carboxylic acid | "υ(C=N)" Imine | υ(C=N) Benzothiazole ring | υ(C=C) aromatic | υ(M–N) υ(M–O) |
|---|---|---|---|---|---|---|---|---|---|
| Ligand (LH) | 3363 - | 3255 | 3047 | 2962 2893 | 1697 | 1674 | 1658 | 1596 1527 | — |
| [Co(L)2].H2O | - 3409 | 3255 | 3047 | 2962 2893 | 1697 | 1664 | 1653 | 1596 1527 | 506 474 |
| [Ni(L)2 ].H2O | - 3374 | 3255 | 3047 | 2970 2839 | 1697 | 1667 | 1658 | 1596 1519 | 532 457 |
| [Pd(L)Cl].H2O | - 3425 | 3255 | 3085 | 2970 2869 | 1697 | 1685 | 1658 | 1596 1527 | 586 476 |
| [Hg(L)Cl] | - 3409 | 3255 | 3062 | 2970 2839 | 1697 | 1681 | 1658 | 1596 1527 | 532 455 |

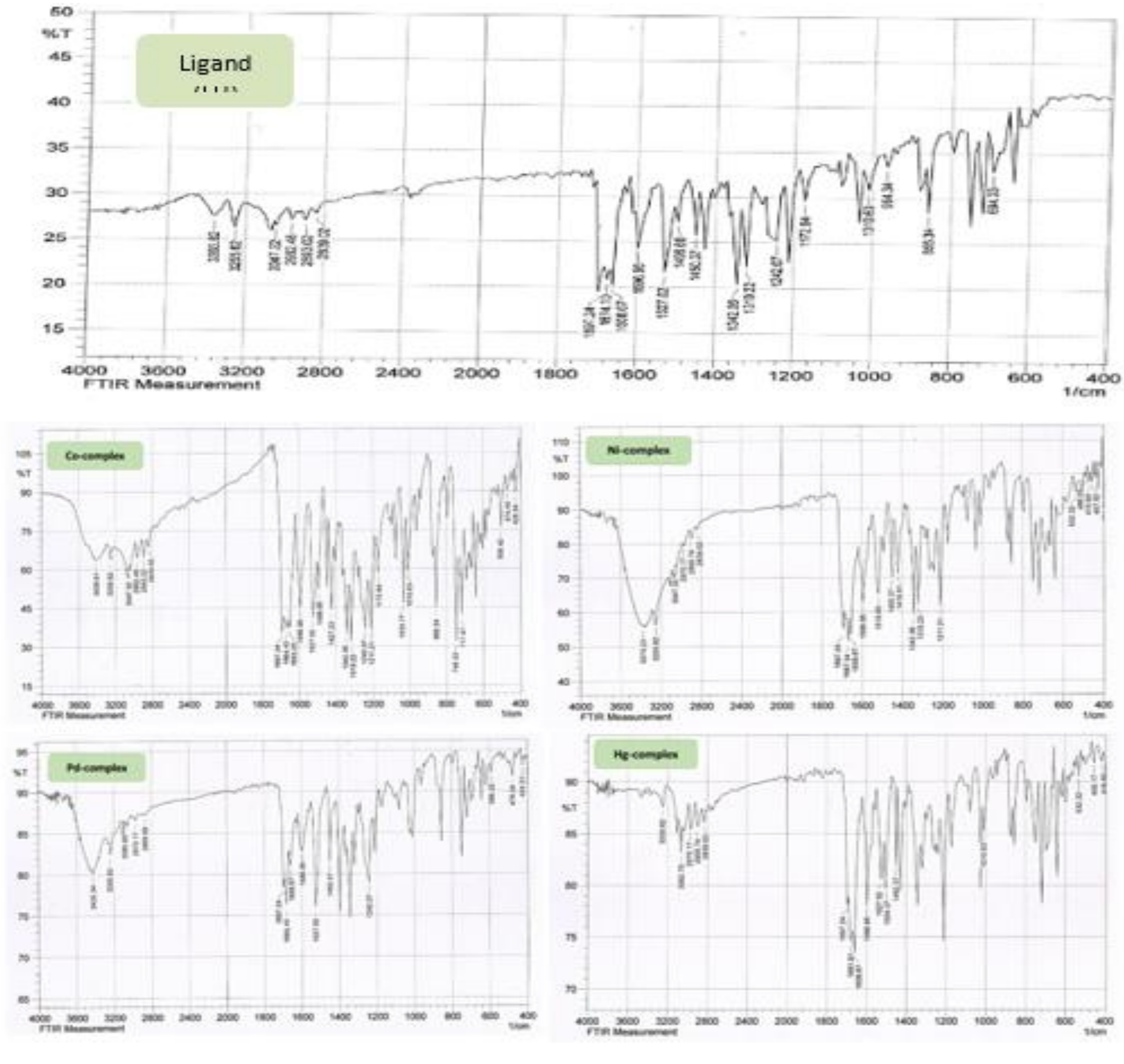



*Figure (2): IR Spectrum of ligand and its metal complex.*

## Electronic Spectra

There was a wide diversity of band types seen in the UV–vis spectra of both the ligand and their complexes. The presence of bands served many purposes, including encouraging the creation of ligands, facilitating the interaction of metal ions with ligands, and indicating the presence of different transitions in the sorts of compounds that were present. At room temperature, UV-Vis spectra were acquired for each of the compounds, and then those spectra were scanned throughout a wavelength range that extended from 200 to 1000 nm. The ethanol was used at a concentration of one hundred percent while making the solutions of the compounds [16-18]. The data on the compounds' spectra can be found in Table (4), and the findings can be shown in Figure (3).

| Table(4): "Electronic spectra", "magnetic moments", "geometry and hybridization" | | | | | |
|---|---|---|---|---|---|
| Compounds | λ(nm) | υ-(cm-1) | Transitions | μeff (B.M) | Geometry |
| Ligand (LH) | 207<br>258<br>322 | 48309<br>38760<br>31056 | π-π*<br>π-π*<br>n-π* | —— | —— |
| [Co(L)2].H2O | 219<br>278<br>335<br>521<br>663 | 45662<br>35971<br>29851<br>19194<br>15083 | Ligand Field<br>Ligand Field<br>Ligand Field<br>4T1g(F) →4T1g (P)<br>4T1g(F) →4T2g(F) | 3.82<br>(Para.) | Octahedral<br>sp3d2<br>distorted |
| [Ni(L)2 ].H2O | 220<br>293<br>352<br>463<br>616<br>746 | 45455<br>34130<br>28409<br>21598<br>16234<br>13405 | Ligand Field<br>Ligand Field<br>Ligand Field<br>3A2g (F) →3T1g (P)<br>3A2g (F) →3T1g(F)<br>3A2g (F) →3T2g(F) | 2.83<br>)Para.( | Octahedral<br>sp3d2<br>Regular |
| [Pd(L)Cl].H2O | 225<br>276<br>341<br>432<br>601<br>654 | 44444<br>36232<br>29326<br>23148<br>16639<br>15291 | Ligand Field<br>Ligand Field<br>Ligand Field<br>1A1g→1Eg<br>1A1g →1B1g<br>1A1g →1A2g | (Dia.) | Square planar<br>dsp2 Regular |
| [Hg(L)Cl] | 226<br>300<br>375 | 44248<br>33333<br>26667 | Ligand Field<br>Ligand Field<br>Charge transfer(MLCT) | (Dia.) | Tetrahedral<br>sp3Regular |

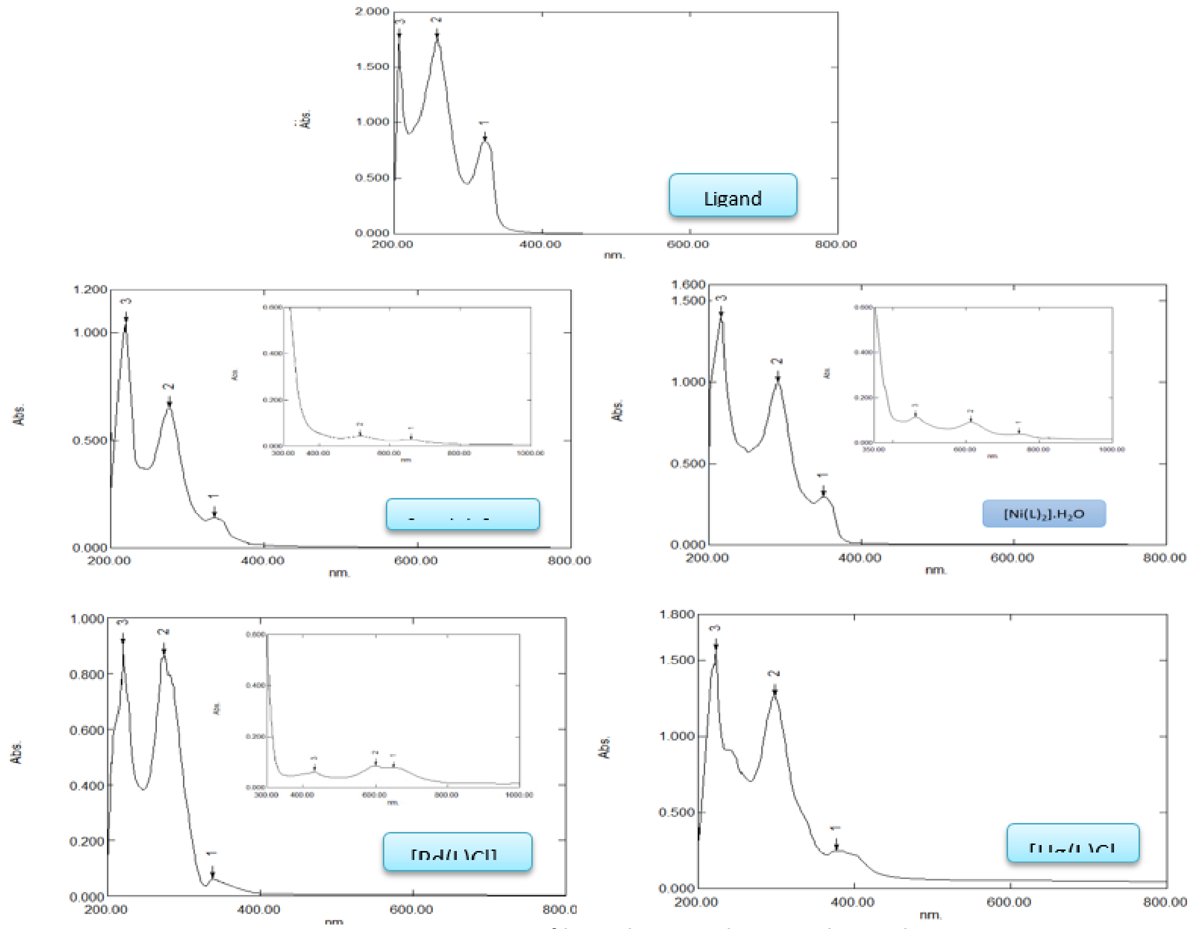



*Figure (3): VU-V-Spectrum of ligand (LH) and its metal complexes.*

## Molar Conductance Measurements

In order to get an accurate reading of the molar conductivity, we employed absolute ethanol with a concentration of 10-3 M at room temperature. Based on the fact that the complexes had molar conductivity values of (12.32-30 ohm-1 cm-2 mol-1), this suggests that the complexes do not possess any ionic characteristics.

"XRD Analysis"

By employing XRD, we were able to record the diffraction patterns of both the ligand and the complexes within the range of 5–80 for the value of 2. The presence of prominent diffraction peaks in the ligand is suggestive of the presence of a crystalline structure. In order to determine some of the structural characteristics, such as the microstrains of crystalline size, the crystalline structure, and the dislocation density, it is necessary to record their purity and any flaws in the crystalline structure while simultaneously converting the ligand to the metallic complex. For the purpose of determining the diameters of the crystallites, the Scherrer equation is used.[19]:

D = "kλ/βCosθ" (1)

where D is the average grain size, b is the line broadening at half the maximum intensity measured in radians, and k is Blanks' constant, which is 0.891. [20, 22]. According to the research, the crystalline size of the ligand and a few of the complexes is less than 100 nm, which brings them well inside the domain of the nanoscale. These results, on the other hand, add validity to the FESEM study that we carried out in the course of our prior research using a field-emission scanning electron microscope. The X-ray diffraction patterns of the ligand are shown in Figure 4, and a summary of the crystallographic data for the ligand and the complexes is provided in Table 5. (5).

Table (5): - "Crystal data of ligand" (LH) "and metal complexes"

| "Compound" | No. | Peak Position °2θ | Peak Width (FWHM) | D Crystallite size(nm) | Rel. Int. [%] | Lattice Strain |
|---|---|---|---|---|---|---|
| (LH) | 1- | 17.72 | 0.179 | 48.65 | 100% | 0.0048 |
| | 2- | 25.22 | 0.194 | 43.85 | 58% | 0.0038 |
| | 3- | 22.04 | 0.230 | 42.22 | 40% | 0.0032 |
| | 4- | 28.32 | 0.432 | 35.87 | 25% | 0.0025 |
| [Co(L)2] H2O | 1- | 25.204 | 0.117 | 72.67 | 100% | 0.0023 |
| | 2- | 34.686 | 0.110 | 70.30 | 93% | 0.0015 |
| | 3- | 27.381 | 0.139 | 66.50 | 51.8% | 0.002 |
| [Ni(L)2] H2O | 1- | 25.282 | 0.059 | 68.30 | 100% | 0.0012 |
| | 2- | 24.291 | 0.103 | 84.61 | 88% | 0.0021 |
| | 3- | 18.396 | 0.400 | 21.04 | 26% | 0.0104 |
| [Pd(L)Cl] H2O | 1- | 24.431 | 0.116 | 73.16 | 100% | 0.0024 |
| | 2- | 17.639 | 0.078 | 89.72 | 5.4% | 0.0022 |
| | 3- | 13.543 | 0.125 | 66.84 | 3.2% | 0.0048 |
| [Hg(L)Cl] | 1- | 24.449 | 0.076 | 71.38 | 100% | 0.0029 |
| | 2- | 12.172 | 0.088 | 62.33 | 47% | 0.0018 |
| | 3- | 21.923 | 0.173 | 53.83 | 36% | 0.0032 |

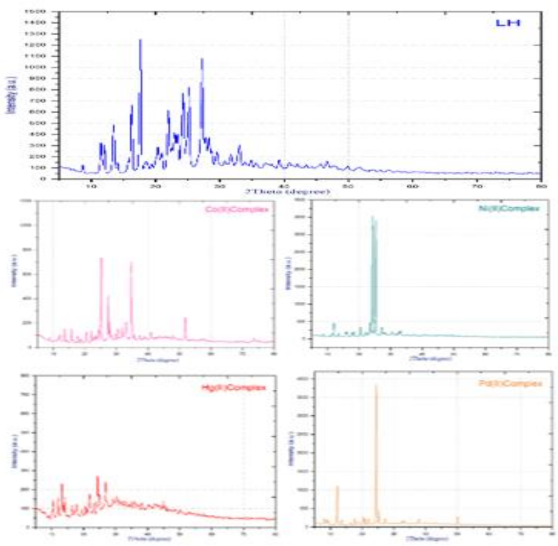



*Figure (4): "XRD patterns of Ligand" (LH) "with some complexes".*

FESEM ("Field emission Scanning Electron Microscope") analysis

As can be seen in Figure (5), both the ligand and the complexes exhibited distinctively diverse morphologies. This method is utilised to investigate "the surface morphology and" form of "the particles", as well as the aggregation that results from the ligand and complexes .[22].

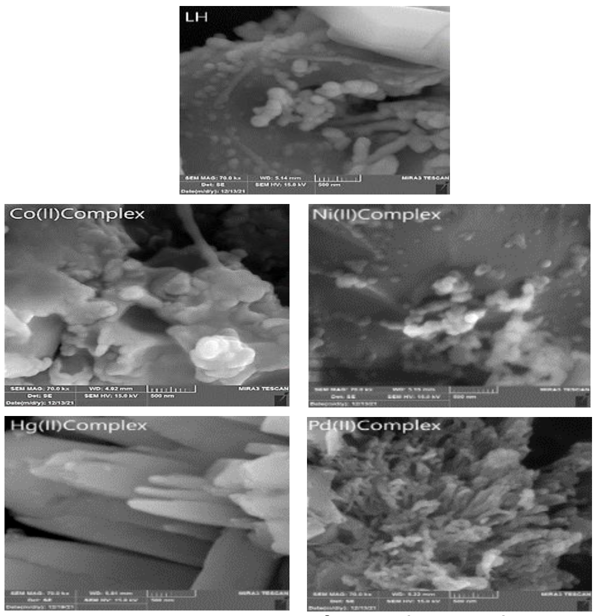



*Figure (5): FESEM images of Ligand (LH) with some complexes*

## Biological Activity

The use of benzothiazole compounds makes the process of inhibiting the development of a broad range of bacterial species a lot less difficult. The reason for this is due to the fact that their products have the power of destroying the outer cell wall, which, in the end, causes the cell's fluids to become depleted and causes the cell to die [19]. The hybrid atom is nitrogen, and it forms complexes with other elements by binding to specific elements in the body of the bacterial cell, such as copper ions, cobalt, iron, zinc, monovalent manganese, and monovalent potassium. Other elements that can be bound include iron, zinc, cobalt, manganese, and potassium. In order for the bacterial cell to perform its functions correctly, certain components are necessary. inevitably leads to the death of the cell as a consequence of the exhaustion of these components in its last stage of development. In this body of study, both the effectiveness of the ligands that were under scrutiny and the metallurgical complexes that those ligands formed with ions were examined. During the course of the study, we used two distinct strains of Staphylococcus as well as Escherichia coli [20, 21]. In order to determine whether or not these kinds had an inhibiting effect, the growth of these organisms was assessed utilizing these kinds. Table contains information that may be obtained there on the antibacterial activity of the recently produced compounds [6, 24]. Our experiment revealed that the ligand, along with a few transition metal complexes, has antibacterial activity that was efficient against the bacteria that were under investigation. Therefore, since benzothiazole compounds are used in the treatment of more severe clinical illnesses, they may have an advantage over bacteriostatic drugs. This is owing to the fact that benzothiazole compounds may inhibit the growth of bacteria.

Table (6): "biological activity data of synthesis compounds"

| Complex | Corresponding | | | |
|---|---|---|---|---|
| | S. aureus | | E.coli | |
| | 250 ppm | 500 ppm | 250 ppm | 500 ppm |
| Ligand (LH) | 6 | 10 | 10 | 11 |
| [Co(L)2] H2O | 7 | 8 | 11 | 13 |
| [Ni(L)2] H2O | 8 | 11 | 7 | 10 |
| [Pd(L)Cl] H2O | 21 | 21 | 20 | 22 |
| [Hg(L)Cl] | 3 | 16 | 11 | 14 |

## 4. Conclusion

Additional information on the ligand molecule and its complexes was gleaned via the use of microelement analysis, melting point measurements, molar conductivity tests, and atomic absorption spectroscopy. Field emission scanning electron microscopy (FESEM) and infrared and ultraviolet (IR and Vis-UV) spectroscopy were also used to study the ligand molecule and its complexes. A comparison of the infrared spectra of the ligand (benzothiazole) and its counterpart (azomethine, a Schiff base) reveals that the metals are freely flowing across the nitrogen atoms amine, and azomethine groups. The data reported above provide evidence that the complex structures designed in the synthesised chemicals are efficient in combating the study microorganisms.

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