

Article

Preparation and characterization of a new ligand derived from the compounds 2-hydrazinobenzothiazole and 2-amino-6-methoxy benzothiazole and its complexes with some metal ions, evaluating its antibacterial activity, and studying its nanoscale properties.

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Abstract

New-type Schiff bases were prepared: [2-(benzoxazol-2-yl)-hydrazinyl diene][2,1-diphenyl-N-(6-methoxybenzothiazol-2-yl)-2,1-diphenylethane]. -1-amine (L) of the mixture. 2-mercaptobenzoxazole was dissolved with hydrazine in absolute ethanol as a first stage to form compound A. while the ligand was obtained in its final from the reaction of compound A with both benzal and 2-amino-6-methoxybenzothiazole. The complex of Ni(II) and Pd(II) was prepared by reacting palladium chloride and nickel chloride with ligand L. The structure of the prepared ligand and its complexes was diagnosed using several spectroscopic techniques, such as FT-IR, UV-Visible, and ¹H-NMR, in addition to FE-SEM, atomic absorption, molar conductivity, and elements. Microanalysis and measurements of magnetic susceptibility and melting points Molar conductivity measurements of palladium (II) and nickel (II) complexes showed that they have an ionic property of (1:2). It was found that the azomethine in the benzoxazole and benzothiazole rings, as well as the nitrogen atoms in the two azomethine groups in the Schiff base, coordinate with the metal ion in the FT-IR spectra of the compound. Based on measurements of sensitivity to ultraviolet, visible, and magnetic radiation, the complexes were shown to have geometries such as palladium (II) being square and planar and nickel (II) being octahedral. It can be used to study its antibacterial effect, and it showed good results.

Keywords: benzoxazole. Benzothiazoles. Benzoxazole-benzothiazole complexes. Antibacterial. Nanoparticles.

Introduction

Benzoxazole, also known as 1,3-benzoxazole, is an aromatic heterocyclic organic compound consisting of an oxazole ring containing one oxygen atom and one nitrogen atom attached to the benzene ring ⁽¹⁾. It was first discovered by chemist Haritzsch in 1887 AD. Its molecular formula is (C₇H₅NO), molar mass 119.12 g/mol, melting point 27-30 degrees Celsius, and boiling point 182 degrees ^(3,2). It is white to light yellow in color with a pyridine-like odor, insoluble in water, and at the same time completely soluble in organic solvents such as ethanol ⁽⁴⁾. In general, benzoxazole and its derivatives are used in coordination chemistry ⁽⁵⁾ to produce complexes as well as optical applications such as photoluminescence, bleaching, and dyeing ⁽⁶⁾.

Benzoxazole belongs to a large family of heterocyclic chemicals ⁽⁷⁾. Due to their diverse biological and pharmacological properties, benzoxazole and its derivatives have a wide range of applications. (It has anti-inflammatory, antibacterial, anti-tuberculous, antispasmodic, antidepressant, and cardiovascular stimulant properties.) Benzoxazole derivatives are frequently used in drug development programs, and many of the medicinal compounds are used in pharmaceutical applications ^(9,8). They are also used in the manufacture of bioactive compounds such as beta-lactams ⁽¹⁰⁾, and benzoxazoles are used to counteract oxidative stress. For the prevention of many diseases, including cancer, it is used in a variety of therapeutic procedures ⁽¹¹⁾.

Benzothiazoles are weak bases and are heterocyclic organic compounds with a distinct binary ring system containing a benzene ring linked to a pentameric thiazole ring containing a nitrogen and a sulfur atom. It is a colorless, slightly viscous liquid. Its melting point is (2) Celsius and its boiling point is (228-227) C°. It is slightly soluble in water ⁽¹²⁾. Its molecular mass is (19.135) g/mol and its density is 1.24 g/ml. It is present in the scent of cocoa beans and coconut. It was first extracted in 1967 from volatile materials from cranberries, and since then it has now been extracted from various sources, as it was found in the tail gland of deer. It also extracts volatile substances from French oak wood ⁽¹³⁾.

Benzothiazole is a heterocyclic compound. It has various biological activities and is a weak base. It is of great importance nowadays. Benzothiazoles and their cyclic derivatives are widely used in many fields, such as medicinal chemistry and life. It is included in the composition of some medications used to treat many diseases. It is

used as an antimicrobial, including both Gram-negative and Gram-positive bacteria, and as an anti-inflammatory, antifungal, antihelminthic, and anticancer, as well as all types of diabetes. The benzothiazole compound and its derivatives are used in several fields, including analytical and industrial ^(15,14).

Experimental

Chemicals and Methods

Chemical reagents are supplied by Sigma-Aldrich, Merck, MSDS, and BDH companies.

Ultraviolet spectra were obtained in the wavelength from 200 to 1000 nm using a Shimadzu U.V-165PCS Spectrophotometer. ¹H-NMR spectrum was recorded on a Varian transform Fourier spectrometer, running at 300 MHz with tetramethyl silane as the internal reference standard in DMSO-d₆ solvent. FT-IR spectra were recorded in the range (400-4000 cm⁻¹) employing an FTIR 8400S Shimadzu Spectrophotometer (Japan). The melting points of prepared samples were obtained employing the Stewart melting point. The magnetic susceptibilities at room temperature were measured with a Balance Magnetic Susceptibility Model MSB-MKI. The atomic absorption spectrophotometry was performed employing a Shimadzu AA-6300 instrument to determine the metal content. The electronic scanning measurements in the emitted field (FE-SEM) of the prepared compounds were recorded using the (MIRA3 TESCAN, Czech), while the (XRD) measurements were made using the (D2Phaser Bruker AXS GmbH) with an angular range of 2θ (20-80°). Conductivity were measured at a concentration of (10⁻³M) and at laboratory temperature, using the Digital Conductivity Series Ino device. Cond 3110 SET1 using ethanol as a solvent. Lastly, elemental analysis was performed using the EA-300.mth instrument.

Synthesis of the ligand (BOHPTEI)

BOHPTEI was manufactured in the following two stages:

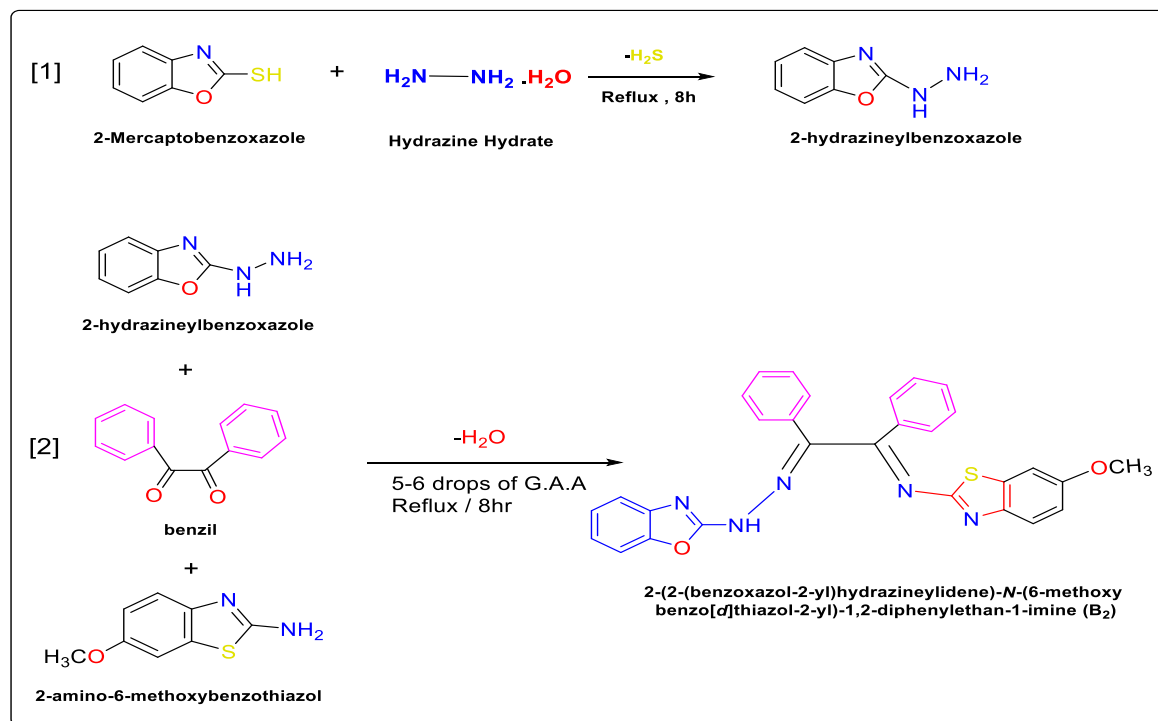
Preparing the chemical 2-hydrazinylbenzoxazole (A) is the first step.

The compound 2-hydrazinylbenzoxazole (A) was prepared by reacting (1.5 g, 0.01 mol) of 2-mercaptobenzoxazole with (25 ml) of absolute ethanol as a solvent in a round flask (250 ml), then adding (5 ml, 0.01 mol) of the solution Aqueous hydrazine with (15 ml) of absolute ethanol with constant stirring. The solution was continuously refluxed for eight hours, after which it was cooled to room temperature and filtered.

The precipitate is then collected and dried to produce yellow crystals, and the final product is recrystallized from ethanol to remove any remaining unreacted materials. The precipitate was allowed to dry for use in the next step, giving the product 2-hydrazinylbenzoxazole (35%), with a melting point (102 °C).

The second step: Synthesis of the ligand (BOHPTEI)

Preparation of ligand (L) from the reaction of (1.5 g, 0.01 mol) of compound (A) dissolved in (25 ml) of absolute ethanol as a solvent that produced ligand (L) to which it was added with continuous stirring at a temperature of (40-50) Celsius. Next, a solution consisting of (2.1 g, 0.01 mol) of benzil and (1.8 g, 0.01 mol) of 2-amino-6-methoxybenzothiazole is dissolved in (25 ml) of absolute ethanol, and acidified by adding (5- 6 drops) of glacial acetic acid. The mixture is refluxed continuously for 8 hours. The solution was cooled, and a precipitate was observed after filtering and drying. It is made by recrystallizing 100% ethanol, collecting the precipitate (yield: 79%), and measuring it at 102-105°C. Scheme (1) showed the preparation of the compound (BOHPTEI).



Scheme (1): preparation of the ligand (L)

Synthesis of the palladium (II) complex

Add (0.423 g, 0.001 mol) of the ligand (L) to 10 mL of absolute ethanol, which has already been used to dissolve 0.177 g, 0.001 mol of palladium(II) chloride and 0.238 g (0.001 mol) of aqueous nickel chloride. After two hours of reflux with stirring, the mixture cools. From absolute ethanol, the precipitate is produced, filtered, dried, and then recrystallized. The compound precipitates as a clear, colored substance. Table 1 includes some physical and chemical properties of the prepared compound (L) as well as the properties of its palladium complex.

Table (1): Several of the physicochemical characteristics (BOHPTEI) ligand and its palladium complex.

Compounds	Color	M.p (C°)	Yield%	M.Wt
Ligand (BOHPTEI)	Light yellow	102-105	73	423.49
[Pd(BOHPTEI)]Cl ₂	Dark brown	159 - 164	90	600.81
[Ni(BOHPTEI)(H ₂ O)Cl]Cl	Light brown	132 - 135	74	651.19

Bioactivity

The biological activity of ligands and complexes was studied on two types of bacteria found in nature, Gram-positive bacteria (*Staphylococcus aureus*) and Gram-negative bacteria (*E. schriachia coli*), using diffusion technology on the surface of culture media as antibacterial agents (10). The biological activity solutions were prepared at a concentration of 500 mg / L by taking the weight of (0.005 g) of the ligand or the complex separately, and it was dissolved in (10 ml) of dimethyl sulfur oxide (DMSO). The compounds were incubated at 37 °C for one day, and the compounds were measured. The results were processed by measuring the diameter of the inhibition zone.

Results and Discussion

The palladium complex is dark brown and the nickel light brown, while the ligand (BOHPTEI) is represented by pale yellow crystals. Many solvents, including ethanol, methanol, DMSO, and DMF, are soluble in the compound and its palladium-nickel complex, but pure water and ether are insoluble. The ligand-to-metal ratio was confirmed to be (1:2):(M:L) using the molar ratio method developed by June ⁽¹⁶⁾.

Molar conductivity Measurements

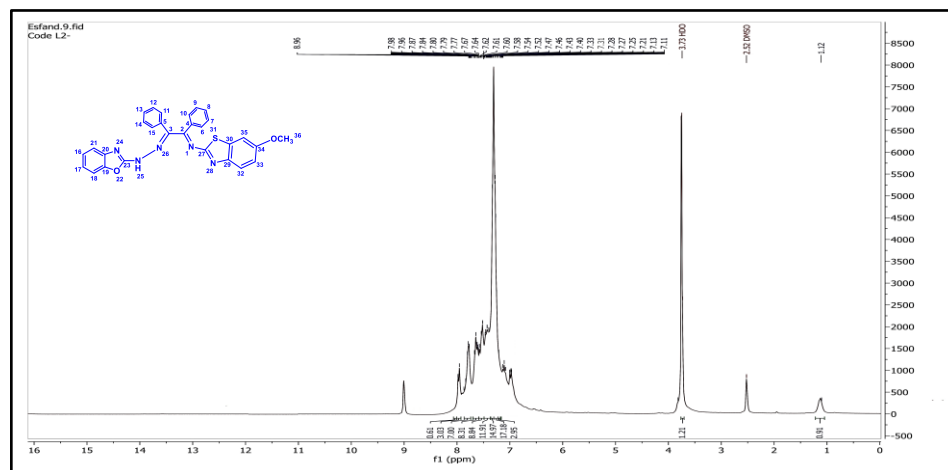
To evaluate the molar conductivity at laboratory temperature and concentration (10^{-3}M), ethanol was employed as a solvent. The palladium-nickel complex has a molar conductivity of ($78\text{ ohm}^{-1}\cdot\text{cm}^2\cdot\text{mol}^{-1}$), indicating that it is ionic. In a 1:2 ratio ⁽¹⁷⁾

The ^1H -NMR spectrum of the ligand (BOHPTEI)

To measure the NMR spectrum of the new prepared ligand (L), I used DMSO- d_6 as a solvent and TMS as an internal standard reference at laboratory temperature. The ligand spectrum in Figure (1) showed a multiple signal when the ligand spectrum showed a multiple signal at (M, 4H, $\delta = 7.40\text{--}7.67\text{ ppm}$) dating back to the protons of the benzoxazole ring ⁽¹⁸⁾, as well as multiple signals.

The other one at (M, 10H, $\delta = 7.77\text{--}7.98$) concerns the protons of the two rings (phenylbenzoyl).

Multiple signals appear at (M, 3H, $\delta = 7.11\text{--}7.31\text{ ppm}$) related to the protons of the benzothiazole ring. The single signal at (S, 1H, $\delta = 8.97\text{ ppm}$) is due to the proton of the secondary amine group (-NH) belonging to the benzoxazole ring, while A single signal appeared at the chemical shift (S, 1H, $\delta = 1.12\text{ ppm}$) due to the protons of the methyl group (C-H) belonging to the benzothiazole ring, and a single signal appeared at the chemical shift (M, 6D, $\delta = 2.52\text{ ppm}$) due to the protons of the solvent (DMSO- d_6) ^(20,19).



Figure(1): ^1H -NMR spectrum of the ligand (L)

Table (2) Signal values of the (^1H -NMR) spectrum of the ligand (L)

Signals (ppm)	Assignment
M, 4H, $\delta = 7.37 - 7.49$	C-H (Benzoxazole ring)
D, 2H, $\delta = 7.50 - 7.75$	C-H (Thiazole ring)
M, 10H, $\delta = 7.80 - 7.97$	C-H (phenyl rings of benzil)
S, 1H, $\delta = 8.92$	-NH (2° amine)
M, 6D, $\delta = 2.52$	DMSO- d_6

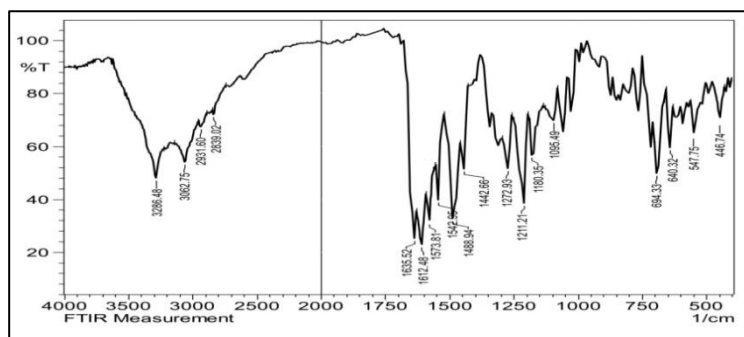
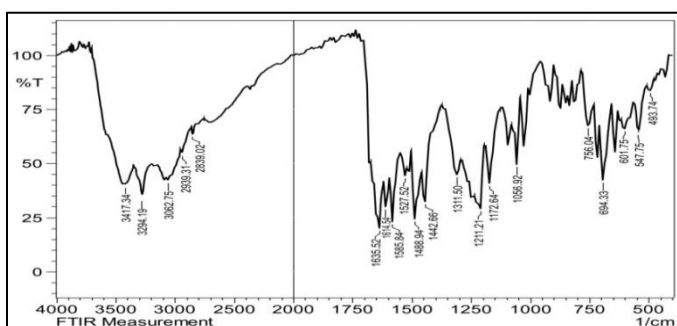
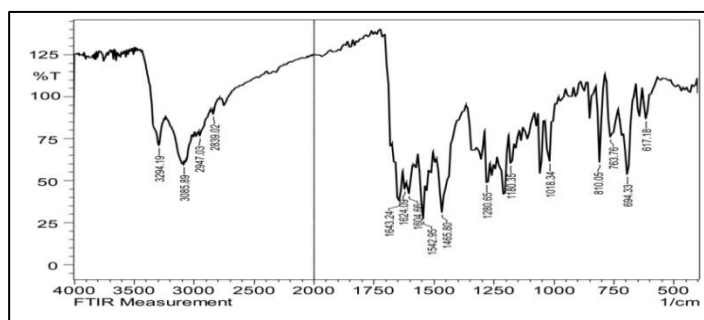
Infrared spectra of BOHPTEI and its palladium(II) and nickel(II) complexes

The infrared spectrum was measured to determine the most important active groups present in the free ligand. The FT-IR spectrum of the free ligand (L) showed a number of bands, the most important of which were bands at wavenumbers (3294 cm^{-1}) and (3085 cm^{-1}), which belong to the secondary amine group $\nu(\text{N-H})$ and the aromatic group $\nu(\text{C-H})$, respectively ⁽²¹⁾. The appearance of an important band in the spectrum of the ligand, the appearance of which indicates the formation of the ligand, is represented by the azomethine group (C=N) ν belonging to the Schiff base, which appeared at (1643 cm^{-1}), while the band of the carbonyl group present in the reactants before the reaction disappeared ⁽²²⁾. As for the band that appeared at (1604 cm^{-1}), it belongs to the azomethine group $\nu(\text{C=N})$ the benzoxazole ring ⁽²³⁾. As for the azomethine ν group (C=N) of the benzothiazole ring, it gave a band at (1624 cm^{-1}), and the aromatic ν group (C=C) gave bands at ($1465\text{--}1542\text{ cm}^{-1}$). Finally, another band appeared at $2839\text{--}2947\text{ cm}^{-1}$, which belongs to aliphatic $\nu(\text{C-H})$ groups ⁽²⁴⁾. It was discovered by examining the spectra of the resulting palladium(II) and nickel(II) compound and comparing it with the spectrum of the free compound:

In the spectrum of the prepared complex, the azomethine group $\nu(\text{C=N})$ of the Schiff base moved towards a lower frequency and appeared at (1635 cm^{-1}) ⁽²⁵⁾. The frequency of the two azomethine groups belonging to the benzothiazole and benzoxazole ring was also changed to a lower frequency, and it occurred at ($570\text{--}547\text{ cm}^{-1}$) in the spectrum of the prepared complex. The interaction of the ligand with palladium and nickel ions via the azomethine groups of the nitrogen atoms is strongly supported by these transitions ⁽²⁶⁾. A new band appears at (547 cm^{-1}) defining the group (M-N) ⁽²⁷⁾.

Table 3: Important spectral bands of Ligand(BOHPTEI), Pd(II), and Ni(II) complex in the infrared

Sample	ν (N-H) 2° Amine	ν (O-H) Hydrate	ν (C-H) Aliphatic	ν (C-H) Aromatic	ν (C=N) Imine	ν (C=N) BenzoThiazole	ν (C=N) Benzoxazole	ν (C=C) aromatic	ν (M-N) ν (M-O)
Ligand (L)	3294		2947 2839	3085	1643	1624	1604	1542 1465	— —
[Pd(L)]Cl ₂	3286	—	2931 2839	3062	1635	1612	1573	1542 1488	547 -
[Ni(L) Cl ₂].H ₂ O	3417	3294	3062	2939 2839	1635	1614	1585	1527 1488	547 493



Figure(4): IR spectrum of the Pd(II) complex

Electronic Spectra

Three absorption peaks could be seen in the ligand's electronic spectra, as illustrated by Figure (5), the first two peaks at 204 nm (49020 cm^{-1}) and 222 nm (45045 cm^{-1}) belong to the transition ($\pi\text{-}\pi^*$), while the third peak at 263 nm (38023 cm^{-1}), due to the ($n\text{-}\pi^*$) transition of the azomethine group ($\text{C}=\text{N}$) ⁽²⁸⁾.

The spectrum of the nickel (II) compound showed six absorption peaks, three of which belong to the ligand domain, namely (46296cm^{-1}), (43103cm^{-1}) and (31348cm^{-1}). While the next three absorption peaks at (19455cm^{-1}), (17182cm^{-1}) and (14164cm^{-1}) indicate the following transitions (${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{P})$), (${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{F})$) and (${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{2g}(\text{F})$, respectively ⁽²⁹⁾. The palladium (II) complex showed five absorption peaks, namely (47393 cm^{-1}), (37313 cm^{-1}), (18315 cm^{-1}) and (16611 cm^{-1}). (14409 cm^{-1}); two belong to the ligand domain and three belong to the transitions ${}^1\text{A}_{1g} \rightarrow {}^1\text{E}_g$, ${}^1\text{A}_{1g} \rightarrow {}^1\text{B}_{1g}$, and ${}^1\text{A}_{1g} \rightarrow {}^1\text{A}_{2g}$, respectively^(31,30). Shows the electronic transitions, the geometric shape, as well as the magnetic susceptibility. It turns out that the geometric shape of the nickel (II) complex is octahedral, except for the Pd (II) complex, which is a planar square.

Table (4): Peaks Absorption Values, Magnetic Momentum and Expected geometry for ligand (BOHPTEI) and Pd(II) complex

Compounds	λ (nm)	ν (cm^{-1})	Transitions	μ_{eff} (B.M)	Geometry
Ligand(BOHPTEI)	204 222 263	49020 45045 38023	$\pi-\pi^*$ $\pi-\pi^*$ $n-\pi^*$	—	—
[Pd(BOHPTEI)]Cl ₂	211 268 546 602 694	47393 37313 18315 16611 14409	Intra Ligand Intra Ligand ${}^1\text{A}_{1g} \rightarrow {}^1\text{E}_g$ ${}^1\text{A}_{1g} \rightarrow {}^1\text{B}_{1g}$ ${}^1\text{A}_{1g} \rightarrow {}^1\text{A}_{2g}$	(Dia.)	Square planar dsp^2
[Ni(BOHPTEI)(H ₂ O)Cl]Cl	216 232 319 514 582 706	46296 43103 31348 19455 17182 14164	Intra Ligand Intra Ligand Intra Ligand ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{P})$ ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{F})$ ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{2g}(\text{F})$	2.83 (Para.)	Octahedral sp^3d^2 Regular

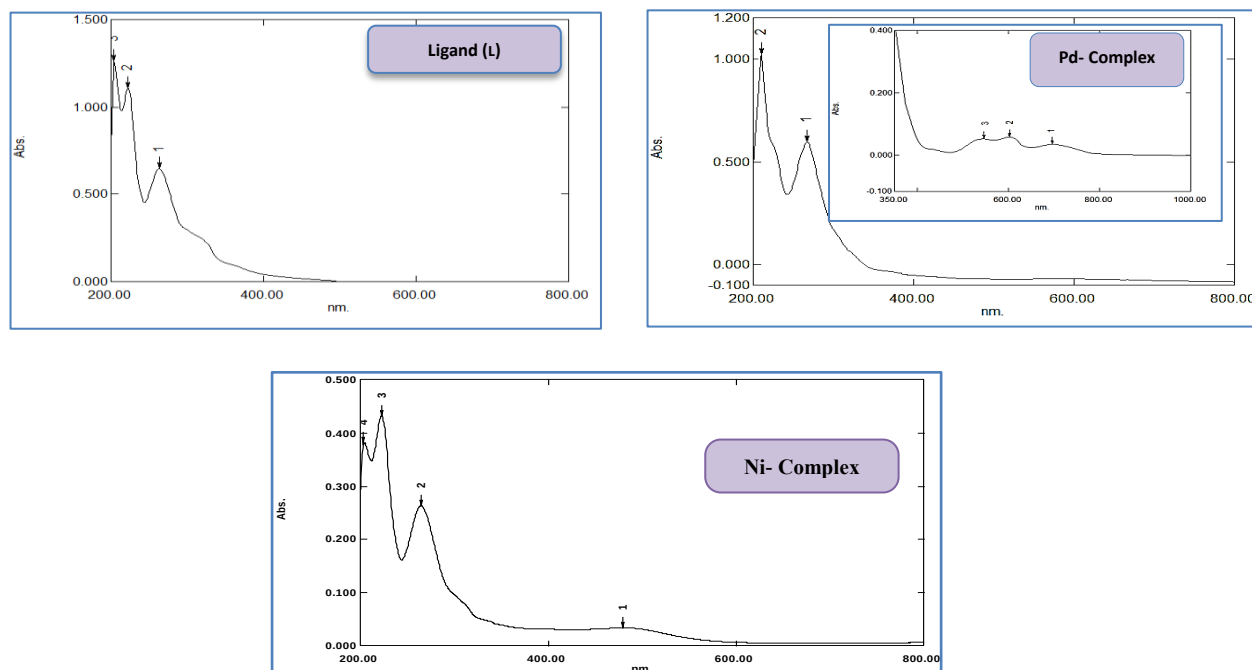


Figure (5): UV-Vis. Spectrum of the BOHPTEI ligand and its palladium(II) and nickel complexes(II).

X-ray diffraction (XRD) study

X-ray diffraction was used to study the crystal structure of (BOHPTEI) and its metal complexes palladium (II) and nickel (II) in their solid state in order to determine several structural parameters, such as crystal shapes and crystal size, which were also estimated. Their degree of purity, defects that develop in crystalline nature when bonds are converted into metal complexes, microstrains, and dislocation density were also determined. There are some diffraction peaks that may be visible for a variety of reasons, such as micro-strains resulting from crystal distortions, such as the absence of lattice distortion and cracks, the domain size of the crystal, and finally the domain size distribution ⁽³²⁾. The XRD spectrum revealed sharp peaks, indicating the formation of a crystalline network, and may have broad peaks, indicating an amorphous structure. The intensity of these peaks is determined by the crystal structure, crystal lattice properties, crystallographic planes, and other factors ⁽³³⁾. The crystal size of the investigated (BOHPTEI) , palladium (II) metal, and nickel (II) complex was determined using the Debye-Scherer formula as follows:

$$D = \frac{k\lambda}{\beta \cos \theta}$$

where:

D = average crystal size

k = shape factor, which is usually about 0.9

λ = the wavelength of the X-ray whose value is $\text{CuK}\alpha = 1.54056 \text{ \AA}$,

β = total width at half height FWHM

θ = the deviation angle

Also, the following equation was used to calculate the microcompliance :-

$$S = \beta \cos \theta / 4$$

S = microstrains

β = total width at half the maximum height

The density of dislocations is estimated using the formula that follows the equation.

$$\delta = 1/D^2$$

δ = density of dissolutions

D = average crystal size

The difference in the distance between the crystallographic degrees d of the composite metal complex, as well as the melt density, was clearly different when analysed using X-ray spectra, supporting the existence of a coordination pathway between the complex and the complex (II), palladium (II), and nickel (II). It was also found that there is an inverse relationship between crystal size, microcompatibility, and melt density, meaning that as the crystal size increases, microcompatibility decreases and the melt density decreases, and thus crystal defects decrease⁽³⁴⁾. X-ray diffraction measurements of the ligand (BOHPTEI) showed a crystalline size of 64.85 nm. Also, the palladium complex had a crystal size of 42.03 nanometers, while the nickel complex had a crystalline size of 77.2 nanometers. These values indicate that the size of the prepared complex (BOHPTEI), metallic palladium (II), and metallic nickel (II) is less than 100 nm, meaning that they are within the nanoscale range⁽³⁵⁾.

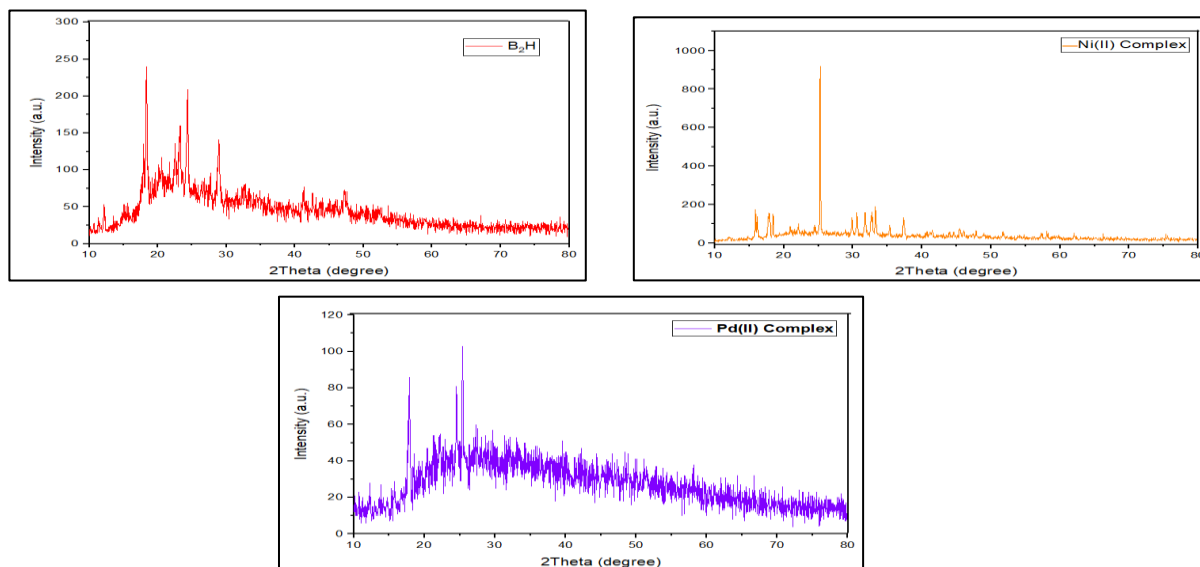


Figure (6): XRD patterns of ligand (B) and its prepared metal complexes

Table(5): Interplanar distances and the 2θ value of each peak, relative intensity for ligand and complex

Compound	No.	Pos. $^{\circ}2\theta$. (Radian)	Width FWHM	d-spacing $\text{\AA}^{\circ}$	D Crystallite size(nm)	Intensity In	Rel. Int [%]
Ligand(BOHPTEI)	1-	20.3121	0.1080	4.3685	78.08	159	100%
	2-	24.2719	0.1080	3.664	78.61	156	86.46%
	3-	31.840	0.228	2.81057	37.86	155	64.85%
[Pd(BOHPTEI)]Cl ₂	1-	17.1860	0.236	4.9786	36.50	93	54%
	2-	24.529	0.242	3.6291	35.39	90	100%
	3-	25.394	0.1574	3.5074	54.20	98	42.03%
[Ni(BOHPTEI)(H ₂ O)Cl]Cl	1-	19.1388	0.1080	4.63359	77.94	56	77.2%
	2-	26.8919	0.6912	3.31272	12.37	97	100%
	3-	34.2447	0.1080	2.61639	80.42	73	77.7%

The electronic scanning measurements in the emitted field (FE-SEM)

Field emission scanning electron (FE-SEM) measurements were used to analyze the surface properties of BOHPTEI and its metal complexes prepared from palladium (II) and nickel (II), in terms of shape, molecular size, and cross-sectional distance distribution. 200 nm and magnification power = 135kx.

Through FE-SEM image analysis, the ligand (BOHPTEI) was shown to be large, homogeneous crystals with a particle size of 237.79 nm, while FE-SEM image analysis of the palladium(II) complex showed it to have irregular, heterogeneous oval shapes with an average size of 43.93 nm. The FE-SEM image of the nickel (II) complex also showed that it is in the form of heterogeneous agglomerated grains, with an average particle size of ($D = 305.9$ nm). The above results showed that the prepared compound and the nickel (II) complex are characterized by a particle size larger than 100 nm, i.e. Outside the nanoscale, the palladium(II) compound has a particle size of less than 100 nm, i.e. within the nanoscale. These results enabled us to study the compound ligand, palladium (II) and nickel (II) in the field of medicine and their ability to eliminate many types of pathogenic bacteria, including (*Escherichia coli* and *Staphylococcus aureus* bacteria), and the possibility of using them as medicine, and this will be clarified later in this study^(38,36).

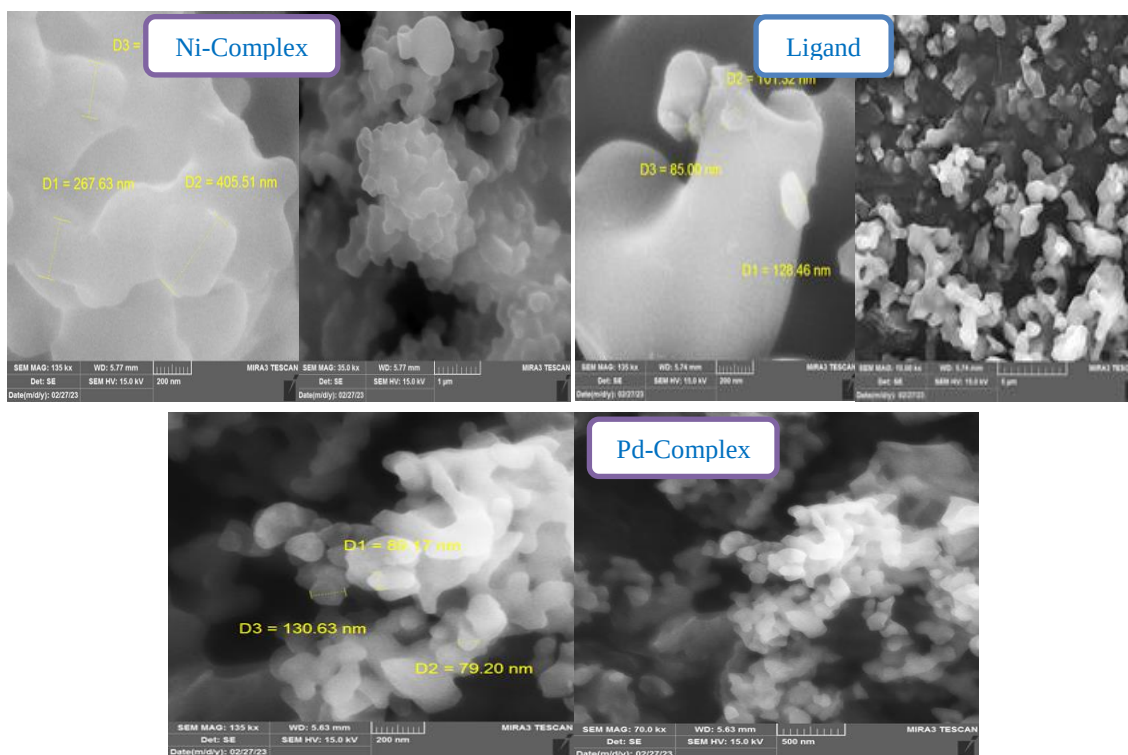


Figure 7: FE-SEM images of the prepared ligand (L) and its metal complexes

Pharmacology Results

Antibacterial Activity

In this research, the biological activity of the ligand and its complexes prepared from their solutions dissolved in dimethyl sulfoxide (DMSO) was studied against two types of pathogenic bacteria, Gram-positive and Gram-negative. It responds to this dye and absorbs it, but does not secrete it outside the cell wall. It is characterized by the presence of gram-positive bacteria. Its wall is very thick and contains magnesium compound and high amounts of RNH. When washed it remains stable. As for those that do not respond to this dye, they throw the dye out of the cell wall, and here they are negative in this direction. Stain: Negative bacteria have a thinner wall than positive bacteria and contain a higher percentage of fats. This difference is due to the nature of its external walls. Therefore, two types of bacteria were used: the first is sensitive and negative to the Gram stain, which is (*Escherichia coli*), and the second is sensitive and positive to the Gram stain, which is (*Staphylococcus aureus*), where the activity of the compounds palladium (II) and nickel (II) at a concentration of (500ppm) was highly effective against bacteria (*S.aureus*) with the percentage of ligand (L) at the same concentration showed less activity against the same type of bacteria compared to the above complexes against the same bacteria under study. The results obtained showed that the nickel (II) complex, at the same concentration, had relatively moderate activity compared to the ligand (L) against *E.coli* bacteria, while the palladium (II) complex gave very high activity against the bacteria under study.

Table (6) shows the inhibition range of each of the L2H ligand and its complexes against some bacteria, size (mm)

No.	Compounds	Corresponding	
		<i>S. aureus</i>	<i>E.coli</i>
		500 ppm	500 ppm
1	Ligand(BOHPTEI)	2.6 cm	4.4 cm
2	[Ni(BOHPTEI)(H ₂ O)Cl]Cl	7 cm	5 cm
6	[Pd(BOHPTEI)]Cl ₂	8.7cm	6 cm

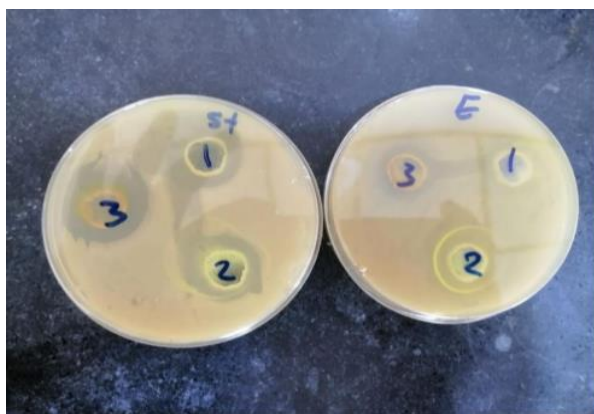


Figure (8) represents (1) for ligand (L) and the first ligand includes 3.2 nickel and palladium complexes, respectively. It shows the effect of ligand (L) and its complexes at a concentration of (500ppm) in inhibiting the growth of staphylococcal and coliform bacteria.

Conclusions

In this research, the ligand (BOHPTEI) was prepared from 2-mercaptobenzoxazole and 2-amino-6-methoxybenzothiazole, and the palladium (II) and nickel (II) complexes of this ligand were prepared by reacting with divalent palladium chloride and divalent aqueous nickel chloride. The ligand and its complexes were characterized in a variety of spectroscopic analyses. By physical methods, infrared spectra confirmed the formation of the azomethine group. These spectra also showed that the compound is coordinated with palladium and nickel ions through the nitrogen atoms of the two azomethine groups of the Schiff base, in addition to the two azomethine groups of the benzoxazole and benzothiazole rings. Upon coordination, the azomethine group shifted towards lower frequencies than in the free compound, indicating the formation of metal complexes.

The electronic spectrum of the palladium (II) complex indicates that the complex has a square, planar geometric shape, while the nickel (II) complex indicates an octahedral geometric shape. Molar conductivity measurements showed that palladium (II) and nickel (II) have an ionic nature. XRD measurements and FE-SEM analysis demonstrated that the ligand and its complexes are nanosized. Also, by testing the bacteria for each of the ligands, palladium (II) and nickel (II) compounds against two types of bacteria, negative and positive Ekram stain Both compounds showed promising antibacterial activity.

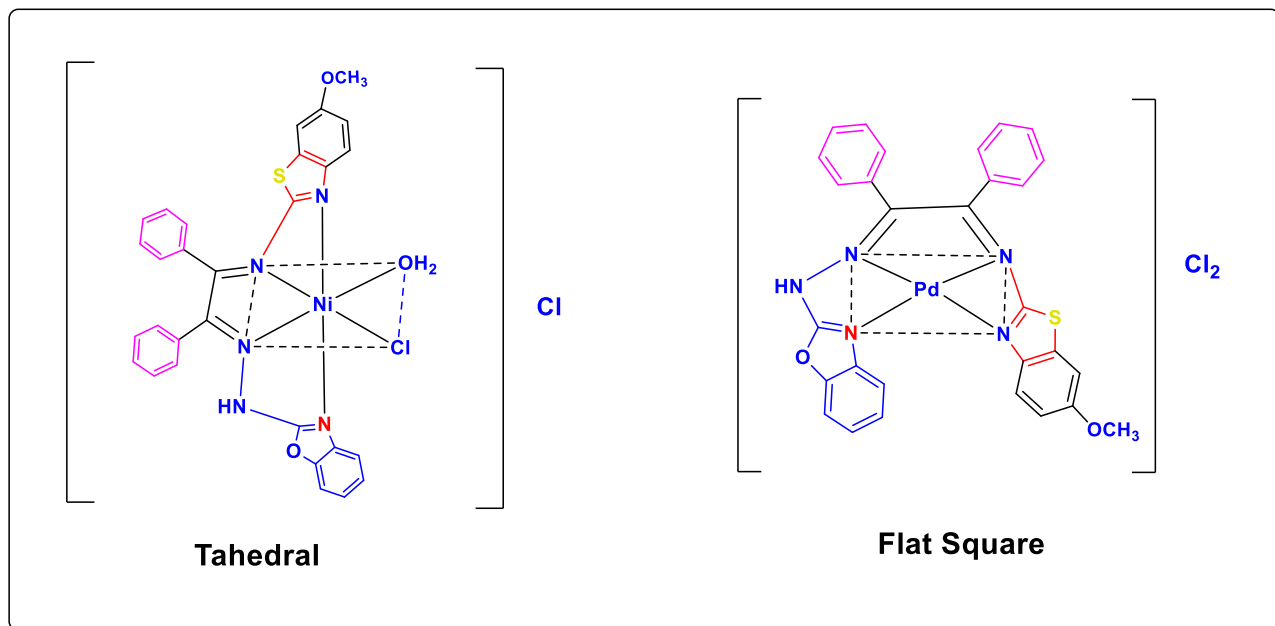


Fig (9): Proposed geometry for the prepared palladium (II) complex

References

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