American Journal of Bioscience and Clinical Integrity
https://biojournals.us/index.php/AJBCI
ISSN: 2997-7347

Article

Preparation and Diagnosis of New Cd(II) Complexes using S-(thiophen-2-yl) 2-((2-hydroxyphenyl)amino)ethanethioate ligand Plus Phosphinate Group

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Citation: Salih, M. M. Preparation and Diagnosis of New Cd(II) Complexes using S-(thiophen-2-yl) 2-((2-hydroxyphenyl)amino)ethanethioate ligand Plus Phosphinate Group. American Journal of Bioscience and Clinical Integrity 2024, 1(9), 48-58.

Received: 28th Aug 2024
Revised: 4th Sept 2024
Accepted: 10th Sept 2024
Published: 18th Sept 2024

Abstract: This study investigates the use of phosphines in conjunction with the S-(thiophen-2-yl) 2-((2-hydroxyphenyl)amino)ethanethioate ligand to synthesize and characterize novel Cd(II) complexes. As sulfur-containing aromatic compounds, thiophenes are important in synthetic and natural chemistry, although there are limitations to their ability to bind metals. Through complexation, which was assessed using methods including FTIR, NMR, and SEM, regular nanostructures in tetrahedral geometries were discovered. This research attempts to address this through complexation, evaluated using techniques such as FTIR, NMR, and SEM, which revealed regular nanostructures in tetrahedral geometries. Promising outcomes were observed in conductivity measures and biological activity tests conducted against bacterial strains. The results provide fresh insights into coordination chemistry and the production of nanomaterials, and also point to the possible applications of these complexes in areas including hydrogen storage and antimicrobial treatments.

Keywords: Cd(II) complexes, FTIR, NMR, SEM

Introduction

The most basic sulfur-containing aromatic molecules are called thiophenes; they are found in many natural goods and fossil fuels [1], and they are essential in determining the unique smells that are significant to food chemistry [2]. We examined the chemical bonding in a new sulfur-containing thiophene-2-thiol by combining density functional theory with sulfur X-ray absorption spectroscopy [3]. We demonstrate that substantial amounts of the thion tautomer are present in thiophene-2-thiol solutions, which may be significantly favored by 5H-thiophene-2-thione or the more accessible 3H-thiophene-2-thione [4].

Chemicals and Equipment

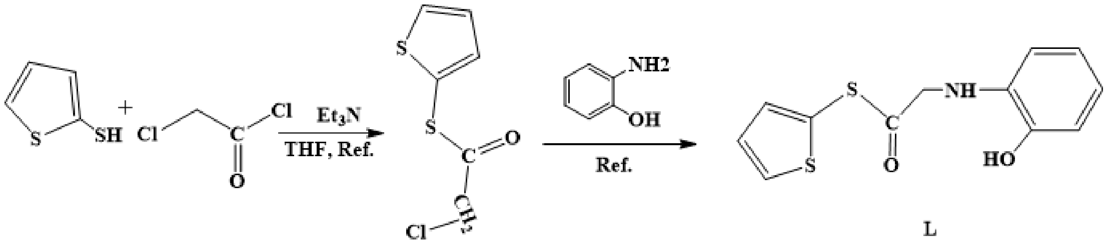
Without being purified, all of the ingredients, reagents, and solvents needed to synthesize the compounds were supplied. In this study, the following phosphines were employed: 1,2-(diphenylphosphine)ethane DPPE, 1,3-(diphenylphosphine)propane DPPP, 1,1,1-ferrocene (diphenylphosphine) DPPF, triphenylphosphine PPh3.

An automatic melting point instrument (SMP30) was used to record the melting point of the produced compounds. Using a Starter 3100c digital conductivity meter, the molar conductivity of the complexes' freshly made 10-3 M DMSO solution was determined. Using an Elementar vario El III CHN analyzer, the elements carbon, hydrogen, nitrogen, and sulfur were microanalyzed. A Shimadzu FTIR 8400S spectrophotometer (400–4000 cm–1) was used to record the complexes' infrared spectra as KBr disks. Using DMSO-d6 as the solvent, the NMR spectra were acquired using a Bruker 400 MHz spectrometer.

Materials and Methods

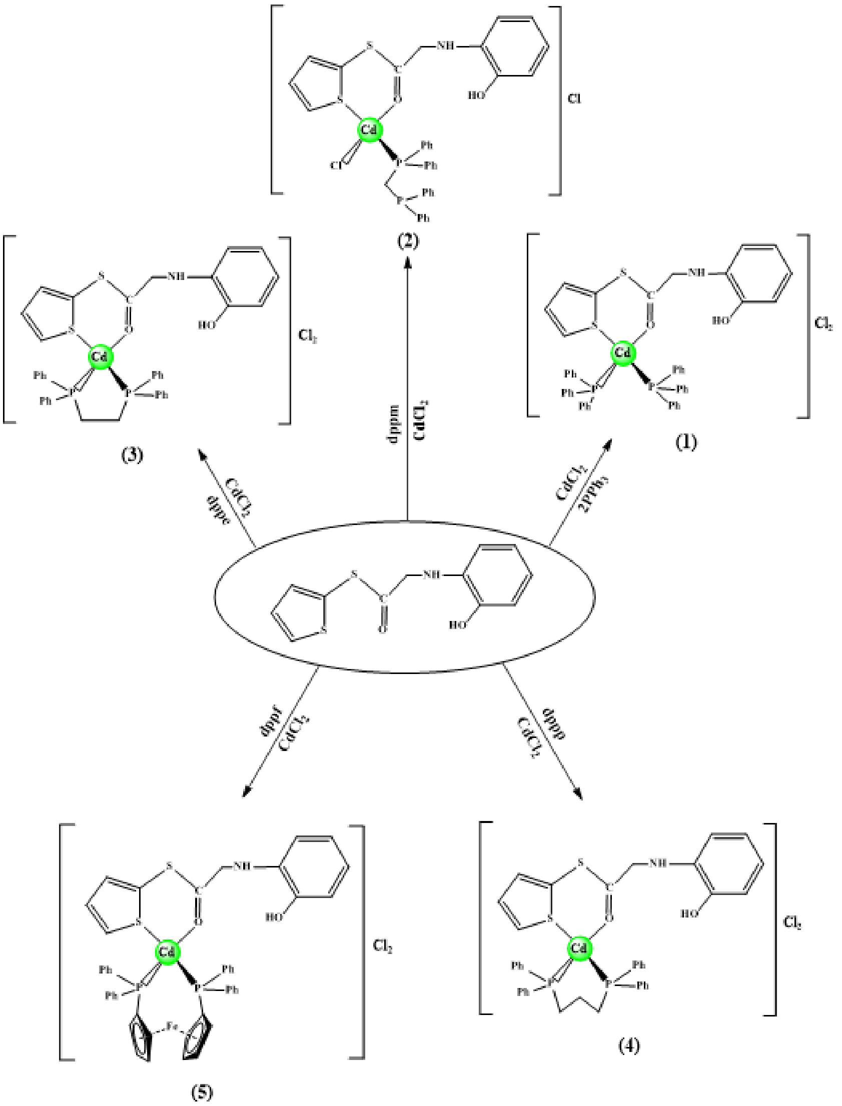
Preparing the Ligand

(10 ml) of THF was added to (2.32 g) of 2-thiothiophenone directly, then (2 ml) of ethylenetriamine (Et3N) was added to the mixture with (2.0 g) of Cl- gradually added, stirring continued in a water bath for 2 hours, then the reaction was elevated for 1 hour and a solution of sodium bicarbonate dissolved in 9 ml of ethanol and 10 ml of distilled water was added to it to transform the solution into two oil layers and an aqueous layer that was separated by a separating funnel, then (2 g) of 2-aminophenol was added to the oily solution and the mixture was elevated for 6 hours giving a dark yellow compound that was filtered and recrystallized (3.429 g, 82%).



Preparation of Complexes

The complexes were prepared by adding one mole of the prepared ligand with an equivalent mole of each of cadmium chloride CdCl2 and the phosphines PPh3, dppm, dppe, dppp, dppf, where it was possible to obtain complexes with tetrahedral coordination shapes in which the ligand behaves in a dihedral manner through the oxygen atom and the sulfur atom, as shown in the complex diagram (1-1).



Scheme 1. Complex structures [Cd(L)(PPh_3)$_2$]Cl_2 (1), [Cd(L)(◎^{1}dppm)Cl]Cl (2), [Cd(L)(dppe)]Cl_2 (3), [Cd(L)(dppp)]Cl_2 (4), [Cd(L)(dppf)]Cl_2 (5)

L Brown powder (0.682 g) yields 79%, m.p : 138-140 ^{o}C . FTIR(KBr): 3411s v(O-H),3180m v(N-H), 3029w v(C-H)Arom, 2923w v(C-H)aliph, 1685s v(C=O), 1562s v(C=C), 1330s v(C-O), 1166m v(C-N), 771s v(C-S) . ^{1}H NMR (DMSO-d^6) δ(PPm) : 12.12 (s, 1H, OH), 7.48 (d, ^3J = 7.72 Hz, 1H, H$_g$), 7.42 (d, ^3J = 7.78 Hz, 1H, H$_d$), 7.15 (t, ^3J = 8.45 Hz, 1H, H$_e$), 7.01 (t, ^3J = 7.50 Hz, 1H,H$_f$), 6.96(d, ^3J = 7.68 Hz, 1H, H$_a$), 6.88 (d, ^3J = 7.84 Hz, 1H, H$_c$), 6.74 (t, ^3J = 7.46 Hz, 1H,H$_b$), 5.73 (s, 1H, NH), 4.12 (s, 2H, CH$_2$). The elemental analysis is calculated for $C_{12}H_{11}NO_2S_2$: C,54.32 ; H,4.18 ; N,5.28 . It is found that: C,54.04 ; H,3.96 ; N,5.13. ʌ (Ω^{-1}.cm^2.mol^{-1}) : 12.4

[Cd(L)(PPh3)$_2$]Cl$_2$ (1) Brown powder (0.682 g) yields 79%, m.p : 184-186 ^{o}C . FTIR(KBr): 3413s v(O-H),3191m v(N-H), 3035w v(C-H)Arom, 2891w v(C-H)aliph, 1664s v(C=O), 1569s v(C=C),1431s v(P-Ph), 1330s v(C-O), 1163m v(C-N), 1099m v(P-C), 746s v(C-S), 694s δ(P-C), 516m v(Cd-S), 443m v(Cd-O) . ^{1}H NMR (DMSO-d^6) δ(PPm) : 10.55 (s, 1H, OH), 7.53 (d, ^3J = 7.80 Hz, 1H, H$_g$), 7.42 (d, ^3J = 7.78 Hz, ^4J = 1.23 Hz, 1H, H$_d$),7.23(m, 12H, H$_{ph}$), 7.18 (t, ^3J = 7.54 Hz, 1H, H$_e$), 7.01(m, 18H, H$_{ph}$), 6.95 (t, ^3J = 7.48 Hz, 1H,H$_f$), 6.84 (d, ^3J = 7.69 Hz, 1H, H$_a$), 6.74 (d, ^3J = 7.71 Hz, 1H, H$_c$), 6.64 (t, ^3J = 7.41 Hz, 1H,H$_b$), 5.83 (s, 1H, NH), 4.25 (s, 2H, CH$_2$). ^{13}P{^{1}H}NMR(DMSO-d^6): δ30.40 ppm The elemental analysis is calculated for $C_{48}H_{41}CdNO_2P_2S_2Cl_2$: C,63.89 ; H,4.58 ; N,1.55 . It is found that: C,63.24 ; H,3.96 ; N,1.13. ʌ (Ω^{-1}.cm^2.mol^{-1}) : 74.9

[Cd(L)(⚜^{1}-dppm)Cl]Cl (2) Brown powder (0.682 g) yields 79%, m.p : 184-186 ^{o}C . FTIR(KBr): 3303s v(O-H),3197m v(N-H), 3053m v(C-H)Arom, 2914m v(C-H)aliph, 1674s v(C=O), 1544s v(C=C),1433s v(P-Ph), 1332s v(C-O), 1182m v(C-N), 1095m v(P-C), 748s v(C-S), 694s δ(P-C), 526m v(Cd-S), 460m v(Cd-O) . ^{1}H NMR (DMSO-d^6) δ(PPm) : 10.45 (s, 1H, OH), 7.74 (d, ^3J = 7.70 Hz, 1H, H$_g$), 7.67 (d, ^3J = 7.68 Hz, 1H, H$_d$), 7.55(m, 8H, H$_{ph}$), 7.48 (t, ^3J = 7.40 Hz, 1H, H$_e$), 7.30(m, 12H, H$_{ph}$), 7.22 (t, ^3J = 7.53 Hz, 1H,H$_f$), 7.13 (d, ^3J = 7.88 Hz, 1H, H$_a$), 7.01 (d, ^3J = 7.74 Hz, 1H, H$_c$), 6.87 (t, 1H,H$_b$), 5.66 (s, 1H, NH), 4.25 (s, 2H, CH$_2$), 3.04 (t, 2H, CH$_2$ $_{dppm}$). ^{13}P{^{1}H}NMR(DMSO-d^6): δ(ppm) : 27.58 d ($^2J_{P-P}$= 48.63 Hz), , -28.16 d ($^2J_{P-P}$= 48.59 Hz) . The elemental analysis is calculated for $C_{37}H_{33}CdCl_2NO_2P_2S_2$: C,53.35 ; H,3.99 ; N,1.68 . It is found that: C,52.98 ; H,3.56 ; N,1.10. ʌ (Ω^{-1}.cm^2.mol^{-1}) : 32.1

[Cd(L)(dppe)]Cl$_2$ (3) Brown powder (0.682 g) yields 79%, m.p : 184-186 ^{o}C . FTIR(KBr): 3315s v(O-H),3209m v(N-H), 3047m v(C-H)Arom, 2952m v(C-H)aliph, 1670s v(C=O), 1539s v(C=C),1438s v(P-Ph), 1336s v(C-O), 1188m v(C-N), 1099m v(P-C), 750s v(C-S), 688s δ(P-C), 522m v(Cd-S), 462m v(Cd-O) . ^{1}H NMR (DMSO-d^6) δ(PPm) : 10.12 (s, 1H, OH), 7.55 (d, ^3J = 7.86 Hz, 1H, H$_g$), 7.45 (d, ^3J = 7.77 Hz, 1H, H$_d$), 7.41(m, 8H, H$_{ph}$), 7.25 (t, ^3J = 7.44 Hz, 1H, H$_e$), 7.20(m, 12H, H$_{ph}$), 7.15 (t, ^3J = 7.36 Hz, 1H,H$_f$), 7.08 (d, ^3J = 7.80 Hz, 1H, H$_a$), 6.98 (d, ^3J = 7.64 Hz, 1H, H$_c$), 6.89 (t, 1H,H$_b$), 5.85 (s, 1H, NH), 3.94 (s, 2H, CH$_2$), 2.88 (t, 4H, CH$_2$ $_{dppe}$). ^{13}P{^{1}H}NMR(DMSO-d^6): δ21.86 ppm. The elemental analysis is calculated for $C_{38}H_{35}CdCl_2NO_2P_2S_2$: C,53.88 ; H,4.16 ; N,1.65 . It is found that: C,53.62 ; H,3.99 ; N,1.37. ʌ (Ω^{-1}.cm^2.mol^{-1}) : 71.7

[Cd(L)(dppp)]Cl$_2$ (4) Brown powder (0.682 g) yields 79%, m.p : 184-186 ^{o}C . FTIR(KBr): 3271s v(O-H), 3191m v(N-H), 3035m v(C-H)Arom, 2923m v(C-H)aliph, 1679s v(C=O), 1553s v(C=C),1423s v(P-Ph), 1332s v(C-O), 1180m v(C-N), 1095m v(P-C), 750s v(C-S), 692s δ(P-C), 518m v(Cd-S), 437m v(Cd-O) . ^{1}H NMR (DMSO-d^6) δ(PPm) : 9.97 (s, 1H, OH), 7.61 (d, ^3J = 7.78 Hz, 1H, H$_g$), 7.53 (d, ^3J = 7.80 Hz, 1H, H$_d$), 7.36(m, 8H, H$_{ph}$), 7.28 (t, ^3J = 7.54 Hz, 1H, H$_e$), 7.22(m, 12H, H$_{ph}$), 7.15 (t, ^3J = 7.41 Hz, 1H,H$_f$), 7.05 (d, ^3J = 7.74 Hz, 1H, H$_a$), 6.93 (d, ^3J = 7.80 Hz, 1H, H$_c$), 6.78 (t, 1H,H$_b$), 6.02 (s, 1H, NH), 4.10 (s, 2H, CH$_2$), 1.94 (t, 4H, CH$_2$ $_{dppp}$), 1.73 (q, 2H, CH$_2$ $_{dppp}$) . ^{13}P{^{1}H}NMR(DMSO-d^6): 12.65 ppm . The elemental analysis is calculated for $C_{39}H_{37}CdCl_2NO_2P_2S_2$: C,54.40 ; H,4.33 ; N,1.63 . It is found that: C,54.14 ; H,4.02 ; N,1.33. ʌ (Ω^{-1}.cm^2.mol^{-1}) : 80

[Cd(L)(dppf)]Cl$_2$ (5) Red powder (0.682 g) yields 79%, m.p : 184-186 ^{o}C . FTIR(KBr): 3419s v(O-H), 3197m v(N-H), 3053m v(C-H)Arom, 2918m v(C-H)aliph, 1674s v(C=O), 1568s v(C=C),1433s v(P-Ph), 1325s v(C-O), 1168m v(C-N), 1093m v(P-C), 738s v(C-S), 694s δ(P-C), 528m v(Cd-S), 430m v(Cd-O) . ^{1}H NMR (DMSO-d^6) δ(PPm) : 9.40 (s, 1H, OH), 7.45 (d, ^3J = 7.70 Hz, 1H, H$_g$), 7.37 (d, ^3J = 7.64 Hz, 1H, H$_d$), 7.29(m, 9H, H$_{ph+e}$), 7.18(m, 13H, H$_{ph+f}$), 7.12 (d, ^3J = 7.66 Hz, 1H, H$_a$), 7.04 (d, ^3J = 7.78 Hz, 1H, H$_c$), 6.98 (t, 1H,H$_b$), 6.01 (s, 1H, NH), 4.35 (s, 2H, CH$_2$), 3.99 (m, 4H, Cp), 3.88 (m, 4H, Cp) . ^{13}P{^{1}H}NMR(DMSO-d^6): 30.07 ppm. The elemental analysis is calculated for $C_{46}H_{39}CdCl_2FeNO_2P_2S_2$: C,55.08 ; H,3.92 ; N,1.40 . It is found that: C,54.85 ; H,3.71 ; N,1.33. ʌ (Ω^{-1}.cm^2.mol^{-1}) : 69.8

Results and Discussion

Molar Conductivity

The data showed high conductivity values which means the presence of free ions outside the coordination sphere if the values are consistent with the formulas of the prepared complexes [5]. The data showed that the ligand solution (L) is non-electrolytic as its electrical conductivity was (12.4 ohm-1.cm-2.mol-1) while the cadmium complex solutions reached the electrical conductivity value of the complex [Cd(L)(1-dppm)Cl]Cl (32.1 ohm-1.cm-2.mol-1) which is within the ratio of 1:1 i.e. positive ion to negative ion, while the rest of the complexes are within the ratio of 1:2 as they gave conductivity values ranging between (80.0-69.8 ohm-1.cm-2.mol-1).

The current values of the conductivity measurements show that complex (2) has one out-of-coordination Cl ion, on the other hand complexes (3,1-5) have two out-of-coordination Cl ions, which would most likely lead to the formation of a tetrahedral arrangement of the central cadmium ion of the complexes and exclude an octahedral arrangement.

Infrared spectrum IR

The FTIR spectrum of ligand L showed a strong band at (3411cm-1) due to OH)) υ, [6] and a signal at frequency (3180cm-1) due to NH)) υ [7], in addition to a signal at (1685cm-1) due to the (υ(C=O) group [8], and a band at frequency (1552cm-1) due to the aromatic v(C=C) [9] stretch , in addition to a signal at (1330cm-1) due to the (C-O) υ group [7], and the appearance of a band at frequency (1166cm-1) due to the v(C-N) stretch [10], and the appearance of a band also at (771cm-1) attributed to v(C-S) [9], in addition to two other bands at frequency (3029cm-1) and (2923cm-1) due to The aromatic and aliphatic (C-H) bonds are shown in the spectrum of ligand (1-1), respectively [9].

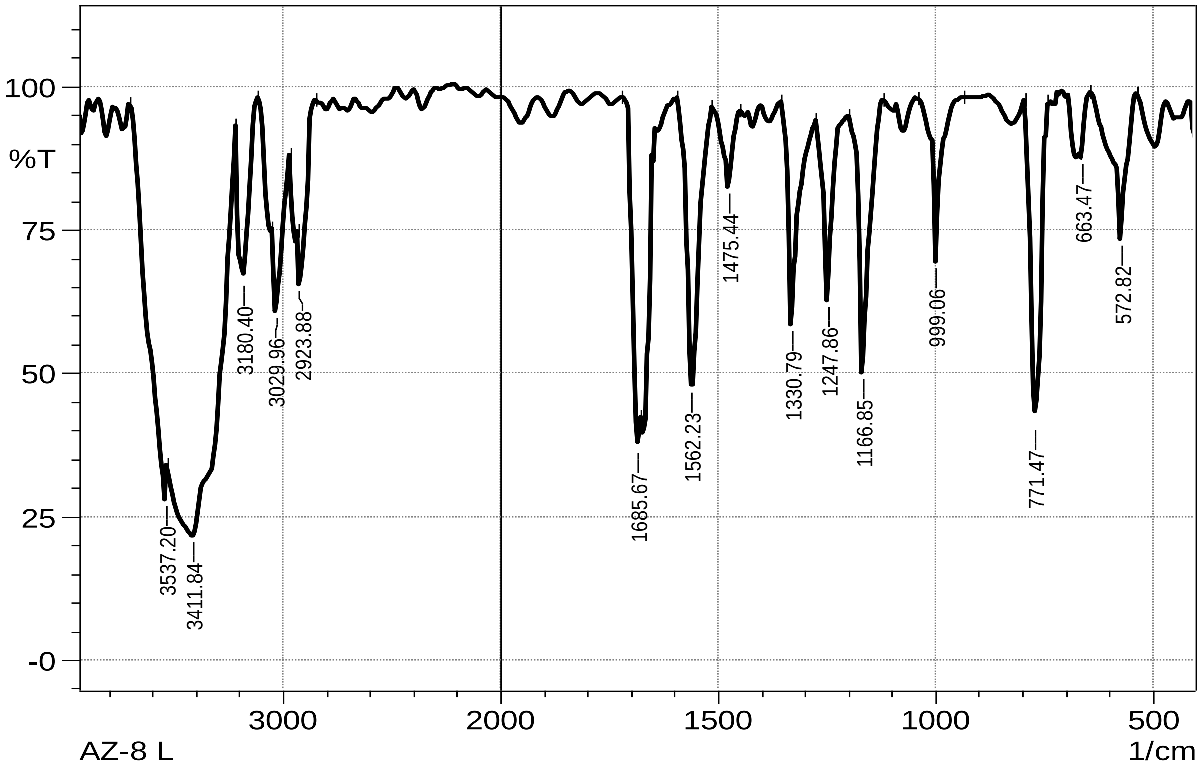


Figure 1. Infrared spectrum of the ligand used (L)

While the infrared spectrum of the prepared complexes showed a new multiple signal between the range (1423s-1438s) and (1093s-1099s) attributed to (υ(P-Ph) and (υ(P-C) respectively [11], and a signal between the range (688s-694s) attributed to the (δ(P-C) bend, and a signal between the range (1664s-1674s) attributed to the (υ(C=O) group, and a signal between the range (738s-750s) attributed to (υ(C-S). These signals prove that the bonds are successfully linked to the metal.

1H {31P} NMR spectrum of ligands and complexes

The 1H NMR spectrum of the ligand measured in DMSO-d6 solvent showed a single signal at the shift (δH = 10.12 ppm) and with one proton integration attributed to the proton of the ((OH group. The spectrum also showed double, double, triple, triple, double, double and triple signals at the shifts 7.48, 7.42, 7.15, 7.01, 6.96, 6.88 and 6.74 ppm due to the protons Hg, Hd, He, Hf, Ha, Hc and Hb respectively. In addition, a single signal at the shift (δH = 5.73 ppm) and with one proton integration attributed to ((NH, and a signal at (δH = 4.12 ppm) and two proton integration attributed to (CH2) of the ligand. Shown in Figure 2.

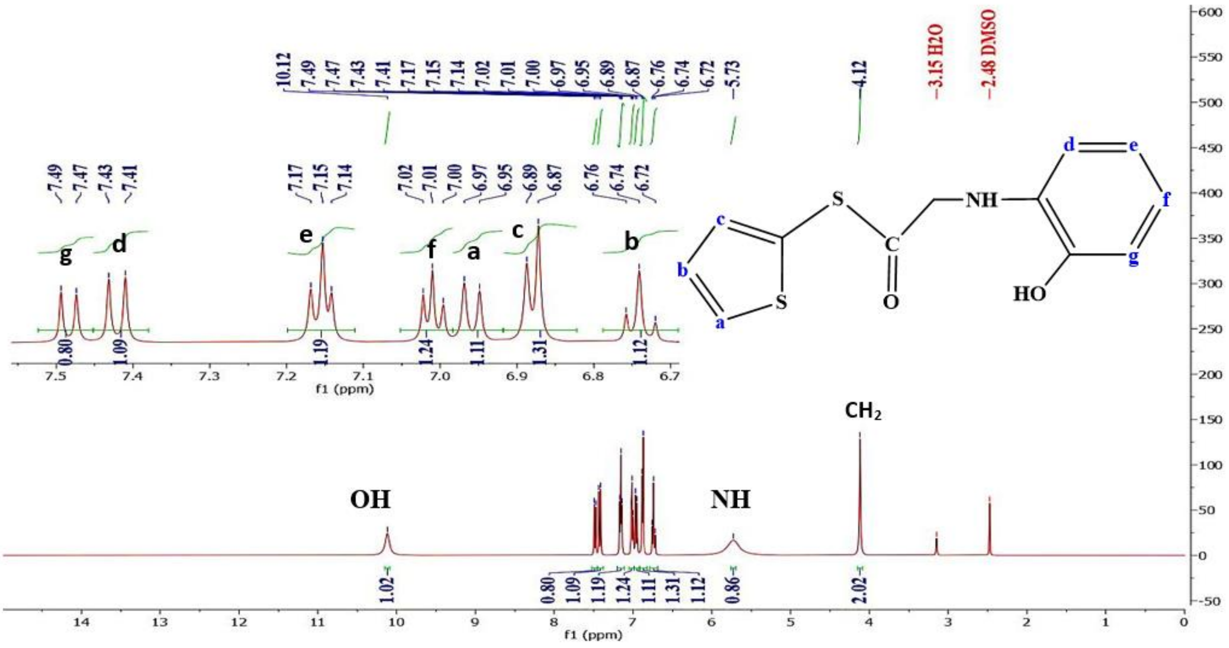


Figure 2. 1H NMR spectrum of ligand (L)

The proton nuclear resonance spectra of the prepared complexes (1-6) showed multiple signals at the shift (7.23, 7.01), (7.55, 7.30), (7.20, 7.41), (7.36, 7.22) and (7.29, 7.18) respectively, due to the protons of the phosphine rings. The phosphorus NMR {1H} 31P- spectrum of complexes (3,1-5) showed a positive single signal at 30.40, 21.86, 12.65 and 30.07 ppm, respectively. The signal indicates the valence of the two phosphorus atoms and the complex possessing one isomer. The phosphorus spectrum of complex (2) showed the presence of two double signals, the first at (27.58) on the positive side and the other at (-28.16) on the negative side. The positive double signal indicates the association of one of the two phosphorus atoms with the metal, and the other negative signal indicates the presence of the other phosphorus atom in a free form.

SEM and Edx results

Recent studies on the geometric structure of coordination complexes have shown that it is possible to produce most of them in regular geometric structures at nanoscale sizes [12]. The morphological appearance of the microstructure of some of the prepared complexes was studied by SEM technique.

SEM image of [Cd(L)(dppe)]Cl2 complex showed irregular cubes with nano-sized 31-188 nm as in (Fig. 1b), and EDX of the complex showed characteristic peaks representing the atoms found in the complex (Fig. 1b).

SEM image of [Cd(L)(dppp)]Cl2 complex showed irregular protrusions, some of them merged with nano-sized 31-50 nm as in (Fig. 2a), and EDX of the complex showed characteristic peaks representing the atoms found in the complex (Fig. 2b).

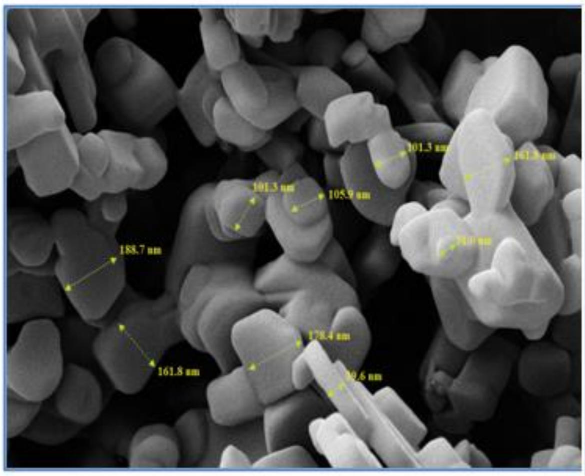


Fig1a : SEM measurement of $[Cd(L)_2(dppe)]Cl_2$

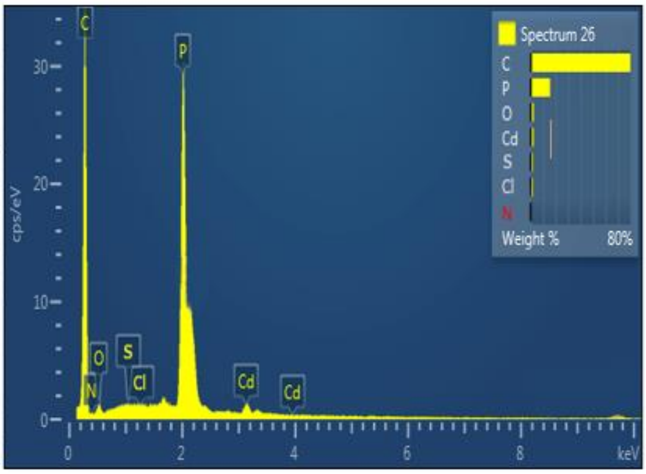


Fig1b : EDX measurement of $[Cd(L)_2(dppe)]Cl_2$

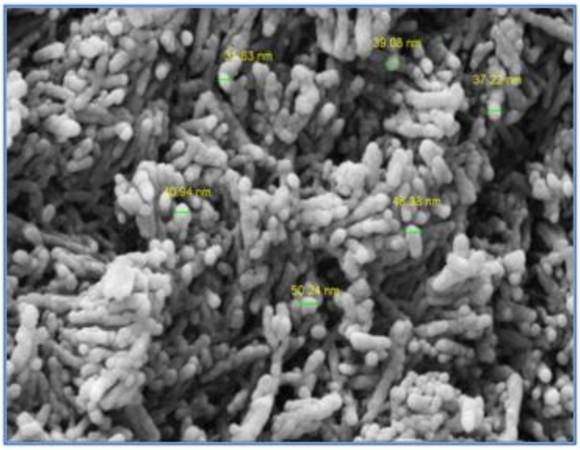


Fig2a : SEM measurement of $[Cd(L)_2(dppp)]Cl_2$

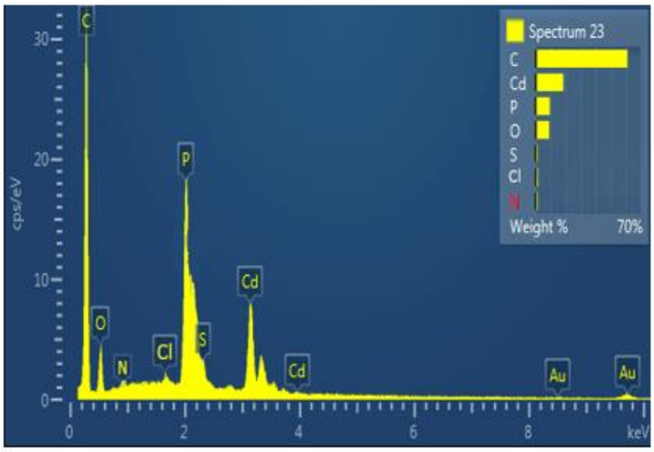


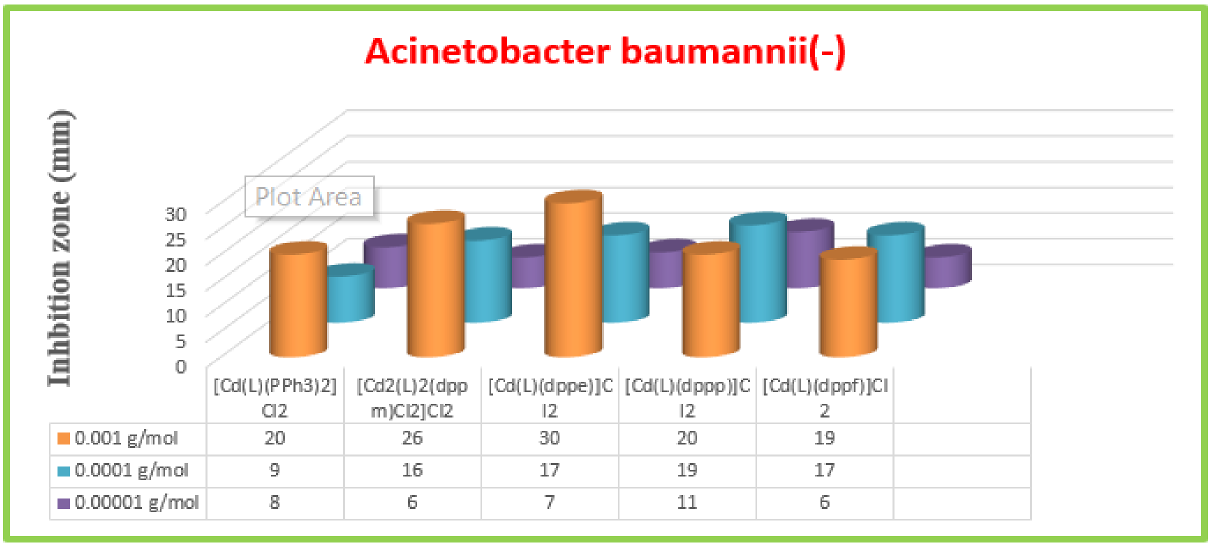
Fig2b : EDX measurement of $[Cd(L)_2(dppp)]Cl_2$

Because they create opportunities for the complexes to be useful in a variety of applications, these findings are regarded as positive findings in the field of coordination chemistry. According to the measurements, regular nanostructures with high porosity were present in the results obtained here, indicating that these complexes can be used in applications that call for high porosity surface area, like those found in hydrogen storage applications [14,13]. The SEM results in this inquiry are consistent with previously published results [15,16], demonstrating that complexes containing nanostructures can be produced, despite the fact that the formulations of nanocomplexes have not been extensively studied [15,16]. The results also showed that the study's particle sizes were somewhat greater than originally reported.

Biological Activity of the Prepared Compounds

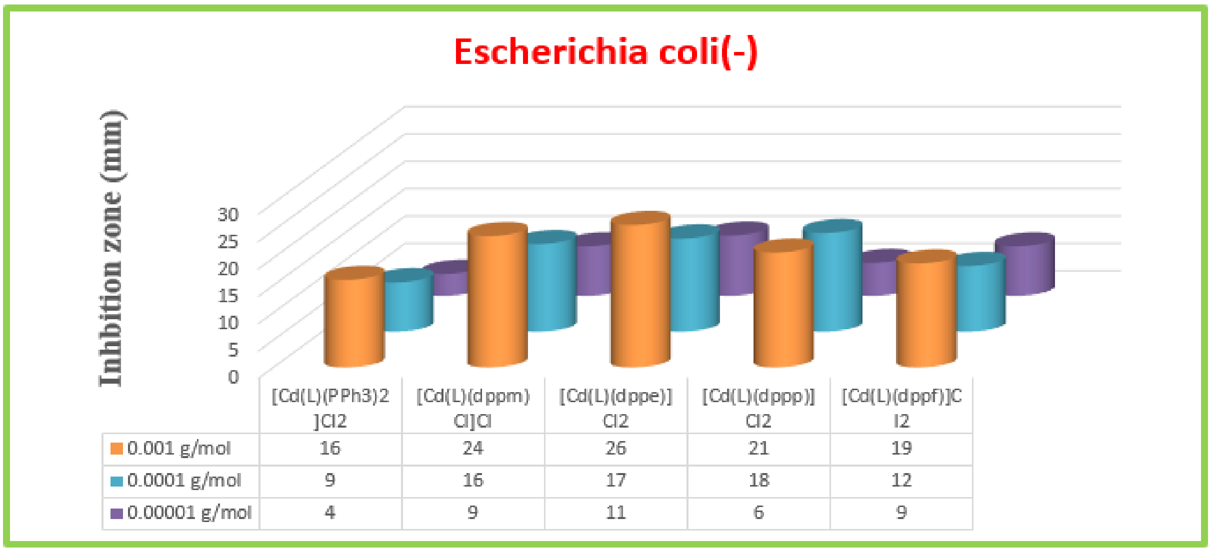
The biological activity of the complexes (0.01, 0.001, 0.0001 mg/ml) was tested against four bacterial species: Acinetobacter baumannii(-), Escherichia coli(-), Enterococcus faecalis(+), Staphylococcus aureus(+) [17-21].

Using the antibiotic amikacin as a standard. All complexes showed activity against the bacterial species as shown in Figure (1-3). Where the concentration 10-4 gave weak effectiveness towards bacterial species, while complex (4) showed the highest inhibition rate at a concentration of 10-3 for Acinetobacter baumannii (-) and Escherichia coli (-) [22-26], and complex (5) showed the highest inhibition rate at a concentration of 10-3 for Staphylococcus aureus (+) and Enterococcus faecalis (+), and complex (3) showed the highest effectiveness at a concentration of 10-2 for Escherichia coli (-), while complex (3) showed the highest effectiveness at the same concentration for the other species [27-30] .



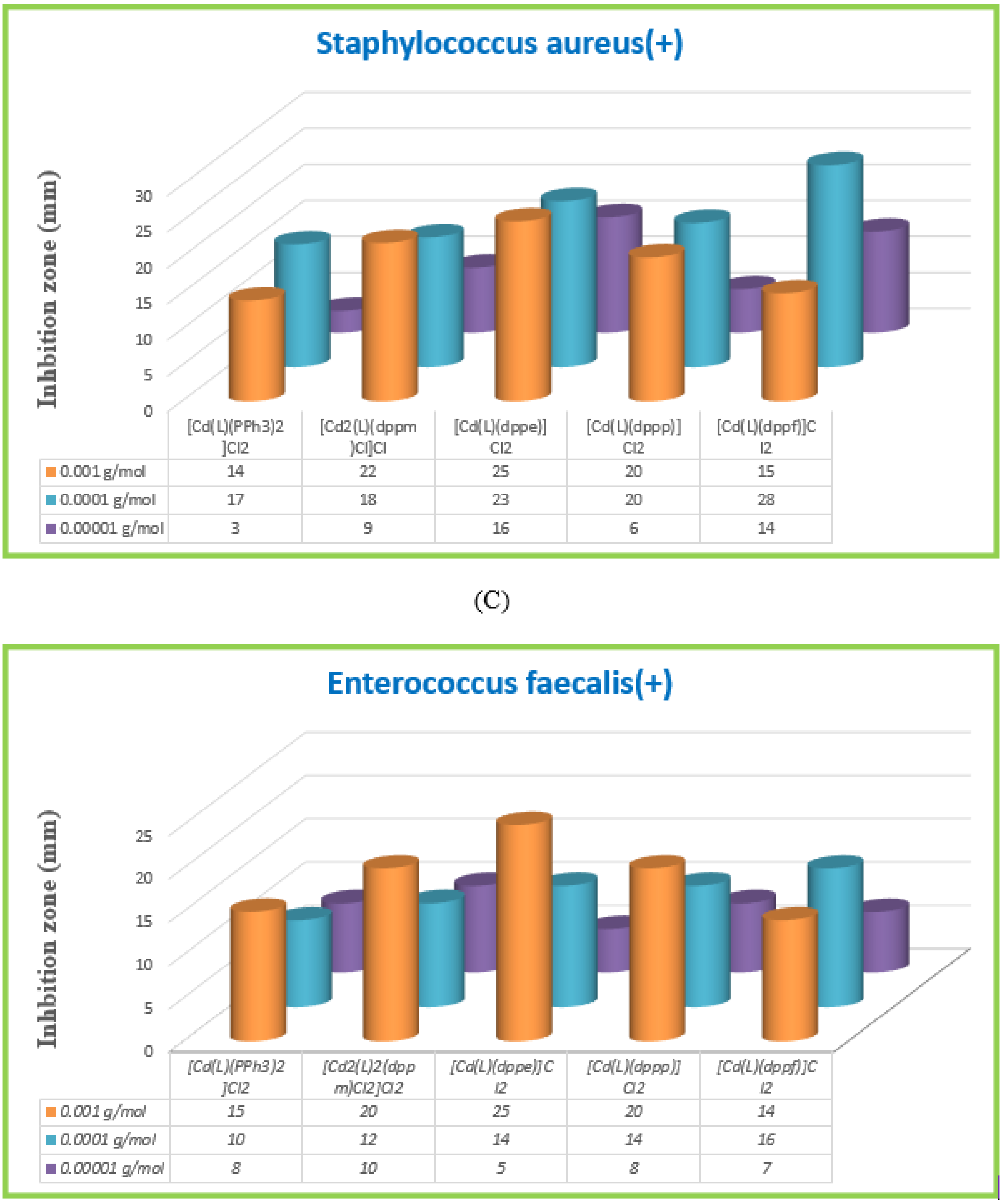
	[Cd(L)(PPh3)2]Cl2	[Cd2(L)2(dppm)Cl2]Cl2	[Cd(L)(dppe)]Cl2	[Cd(L)(dppp)]Cl2	[Cd(L)(dppf)]Cl2	
🟧 0.001 g/mol	20	26	30	20	19	
🟦 0.0001 g/mol	9	16	17	19	17	
🟪 0.00001 g/mol	8	6	7	11	6	

(A)



	[Cd(L)(PPh3)2]Cl2	[Cd(L)(dppm)Cl]Cl	[Cd(L)(dppe)]Cl2	[Cd(L)(dppp)]Cl2	[Cd(L)(dppf)]Cl2
🟧 0.001 g/mol	16	24	26	21	19
🟦 0.0001 g/mol	9	16	17	18	12
🟪 0.00001 g/mol	4	9	11	6	9

(B)



	[Cd(L)(PPh3)2]Cl2	[Cd2(L)(dppm)Cl]Cl	[Cd(L)(dppe)]Cl2	[Cd(L)(dppp)]Cl2	[Cd(L)(dppf)]Cl2
🟧 0.001 g/mol	14	22	25	20	15
🟦 0.0001 g/mol	17	18	23	20	28
🟪 0.00001 g/mol	3	9	16	6	14

(C)

	[Cd(L)(PPh3)2]Cl2	*[Cd2(L)2(dppm)Cl2]Cl2*	*[Cd(L)(dppe)]Cl2*	*[Cd(L)(dppp)]Cl2*	*[Cd(L)(dppf)]Cl2*
🟧 *0.001 g/mol*	*15*	*20*	*25*	*20*	*14*
🟦 *0.0001 g/mol*	*10*	*12*	*14*	*14*	*16*
🟪 *0.00001 g/mol*	*8*	*10*	*5*	*8*	*7*

Figure 3. Inhibitory activity of the prepared compounds on the growth of a number of negative bacteria (A), (B) and positive bacteria (C), (D)

Conclusion

The prepared ligand harmonizes with the dioxygenated metals and phosphines in a two-age form, this is what the applications used in the diagnosis mentioned above through the oxygen and sulfur atoms of the pentagonal ring, the complexes also showed tetrahedral symmetry forms, which gave the prepared complexes more stability and stability.

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