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Chemistry

BINUCLEAR MALONOHYDRAZIDE DITHIOCARBAMATE COMPLEXES OF Ni (II), Pd (II) AND Pt (II): SYNTHESIS, CHARACTERIZATION, ANTIMICROBIAL ACTIVITY, AND SEM STUDIES

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Abstract. Binuclear complexes have been prepared with formula $[M_2L_2]$, where L is potassium 2,2'-malonylbis (hydrazine-1-carbodithioate), M is Ni (II), Pd (II), and Pt (II). Each complex has been characterized by elemental analysis (CHNS), UV-Visible, FT-IR spectra, proton and carbon nuclear magnetic resonance (^{1}H NMR and ^{13}C NMR) spectra, magnetic susceptibility, scanning electron microscopy (SEM), and molar conductivity.

Keywords: dithiocarbamate, metal complexes, biological activity, SEM study.

1. Introduction

The dithiocarbamate (DTC) ligand is one of commonly important dithiolite ligands similar to the xanthate. Dithiophosphate ligands are used for the preparation of coordination complexes[1] due to their high stability compared to other ligands[2]. Additionally, dithiocarbamate compounds are modifiable bonds forming stable complexes with most transition metals in a variety of oxidation states. The DTC group including bidentate sulfur atoms reduces the cytotoxicity of Pt compounds by selectively eliminating Pt-S thiol enzyme[3]. Electron density drifts towards the sulfur atoms of the dithiocarbamate ligands due to the mesmeric effect of the NR2 group[4]. Dithiocarbamate ligands and their complexes have attracted wide attention due to their diverse applications, biological, magnetic, electrical, and thermal properties[5]. They behave as non-trapping agents: lubricants[6, 7], vulcanizes, and solar controllers[8]. In addition, these complexes can be used in the field of healthcare and medicine, for example, to fight cancer[9]. The action of antimicrobial drugs can be described as the penetration of biologically active substances through the bacterial cell wall, which leads to a decrease in bacterial growth, causing an increase in the diameter of the inhibition zone and indicating an inhibitory effect against bacteria[10].

The aim of this research is to synthesize dithiocarbamate complexes derived from Ni (II), Pd (II), Pt (II) and malonyl dihydrazide dithiocarbamate ligand and evaluate the inhibitory effect of the prepared complexes against the three types of bacteria: gram-negative *Escherichia coli, Pseudomonas aeruginosa* and gram-positive *Staphylococcus aureus*.

2. Experimental

2.1. Materials and Methods

All used chemical reagents were of analytical grade and used as received without further purification. Malonic diethyl ester 99 %, carbon disulfide 99.5 %, and sodium hydroxide 99 % were obtained from Aldrich. Nickel (II) Chloride Hexahydrate 98 %, palladium (II) Chloride 99 %, platinum (II) chloride 98 % have been supplied by Merck. For identification purposes the following instruments were used: a Brucker Alpha-P FT-IR Spectrometer in the range of 500–4000 cm^{-1}, Vario ELIII Elemental Analyzer Device (for elemental analysis of CHNS), UV-visible spectrophotometer (PgT92+, DMSO as solvent at 25 $^{\circ}$C), (Varian 400 MHz Spectrometer by using DMSO-d6 solution (for ^{1}H-NMR and ^{13}CNMR). Additionally, magnetic susceptibility and conductivity were measured using Sherwood Scientific Instrument and MC-1 conductivity apparatus, respectively. Magnetic corrections were performed by pascal constants. For topography images, scanning electron microscopy (SEM, TESCAN-Vega3) was used.

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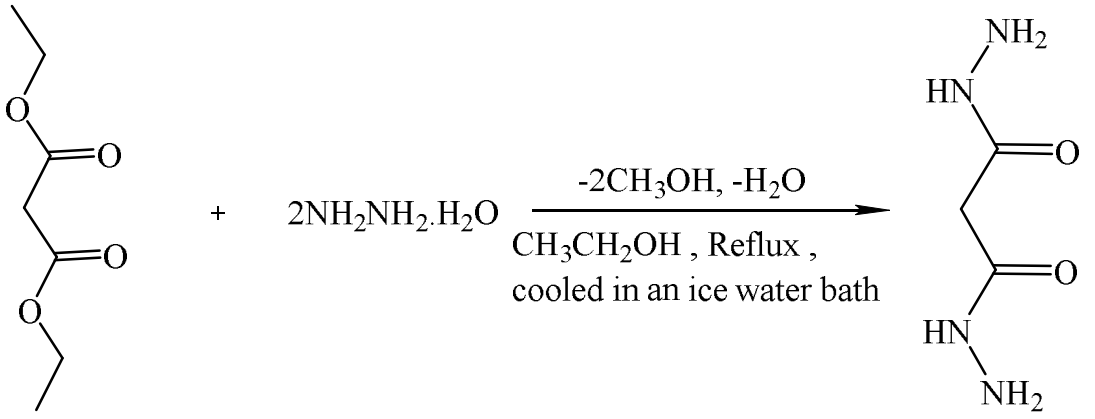
2.2. Synthesis of Malonyl Dihydrazide

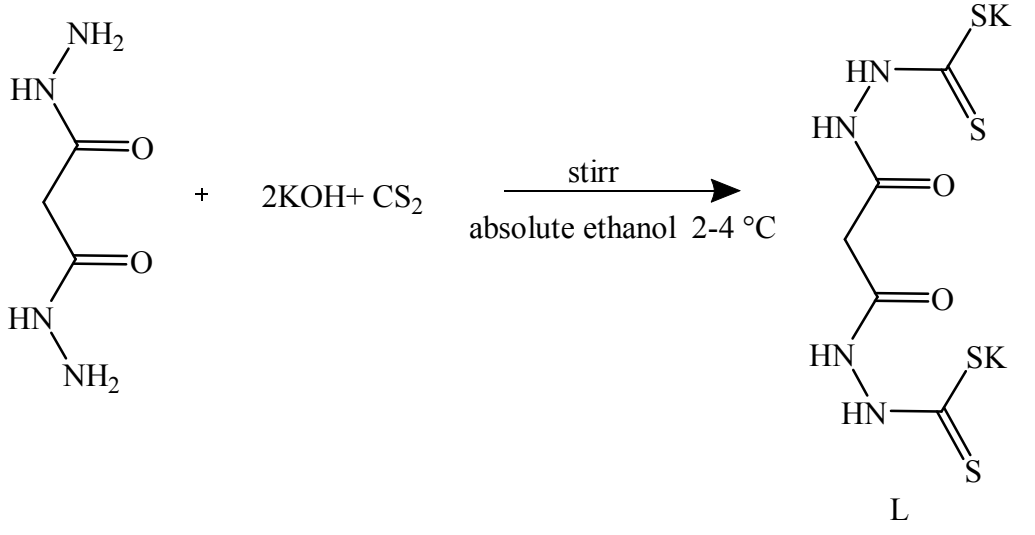
Diethylmalonic ester (9.5 mL, 10.00 g, 62.5 mmol) was dissolved in 25 mL of absolute ethanol and was slowly added for 2 h to a stirred excess of hydrazine hydrate hot solution (125 mmol, 80 % dissolved in 25 mL of absolute ethanol). The reaction mixture was then refluxed for 2 hrs and cooled in an ice water bath. The precipitated product was filtered and recrystallized from a mixture of ethanol and diethyl ether (1:1). Then white precipitate was dried in a desiccator over anhydrous $CaCl_2$. The yield was 90 %; m.p 154–155 °C.



Scheme 1. Synthesis reaction for malonohydrazide

2.3. Synthesis of Potassium 2,2′-Malonylbis(Hydrazine-1-Carbodithioate) (K_2L)

Malonyl dihydrazide (2 g, 0.015 mol) was dissolved in 25 mL of absolute ethanol. A solution of potassium hydroxide (1.68 g, 0.03 mol) in 25 mL of absolute ethanol was added to the malonyl solution. An excess of carbon disulfide (1.8 mL, 0.03 mol) was added to the mixed solutions and stirred for 2 hrs with keeping the temperature within a range of 2–4 °C. The precipitate was filtered and the yellow product was washed by ether. ^{1}H NMR [(DMSO-d^6, δ)]: δ 3.96 (2H, s, CH_2), δ 9.41 (2H, s, 2NH), δ 11.19 (2H, s, 2NH),[11] and ^{13}C-NMR (400 MHz); δ 23.65 (CH_2), 157.87 (C=O),[12] 180.14 (C=S). The spectral data agreed with the literature data[13–16].



Scheme 2. Synthesis reaction
for potassium 2,2'-malonylbis(hydrazine-1-carbodithioate)

2.4. Synthesis of $[Ni_2(L)_2]$ Complex

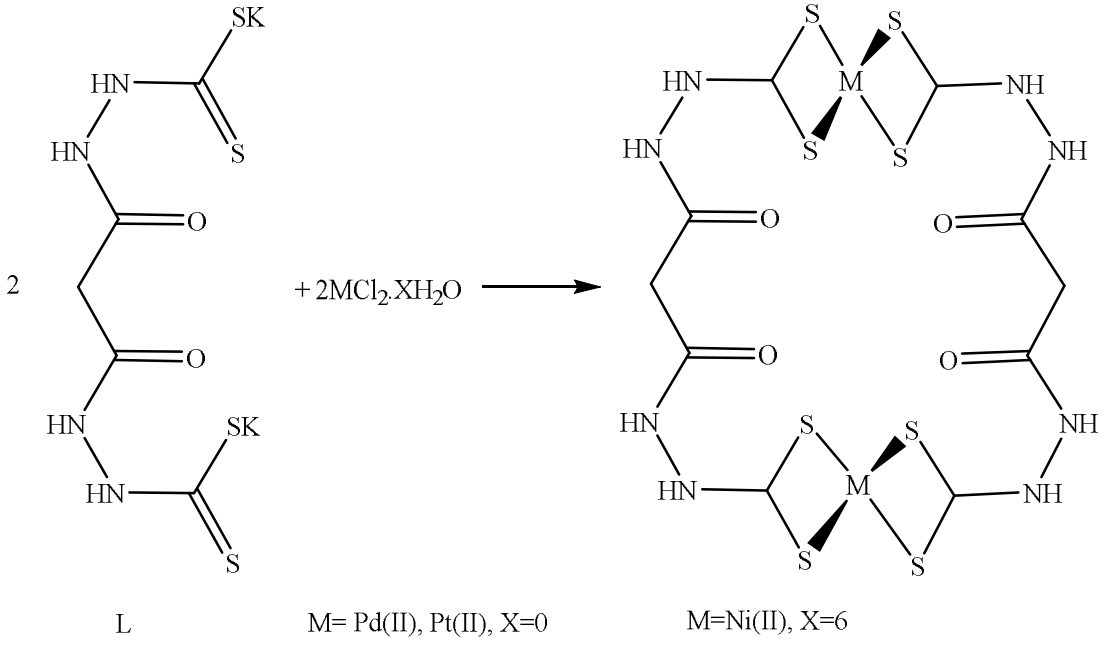
The metal salt (0.001 mol, 0.237 g $NiCl_2.6H_2O$,) was dissolved completely in 25 mL of ethanol to prepare K_2L (0.001 mol, 0.5 g). A light-green colored solid was precipitated and then left for 1 hour, filtered, and washed. The complex was recrystallized from ethanol and dried under vacuum.

2.5. Synthesis of $[M_2(L)_2]$ Complexes, M = Pd(II), Pt (II)

The complexes were prepared in the same manner as mentioned in paragraph 2.4 only changing the metal salts. 0.001 mol of $PdCl_2$ (0.177 g), $PtCl_2$ (0.266 g) was dissolved completely in 25 mL of cold water with few drops of HCl to prepare K_2L (0.001 mol, 0.5 g). A brown colored solid was precipitated and then allowed to stand for an hour. After this it was filtered and washed. The product was recrystallized from ethanol and dried under vacuum.

2.6. Antibacterial Activity

For biological activity evaluation, *Escherichia coli*, *Pseudomonas aeruginosa*, and *Staphylococcus aureus* have been isolated[17,18]. This part of the research was performed at the Biological Department, Pure Science Education College, Kirkuk University. The disks were prepared on filter paper (Whatman paper, number 1) and transferred into the used metal complexes (50, 100, 150) mg/mL in an incubator at 37 °C for 24 hrs[19]. Gentamycin and Chloramphenicol were used as control compounds (10 and 30 mg/mL, respectively). Agar-well diffusion method was used for biological activity tests (for additional information see the work[20]).



Scheme 3. Synthesis reaction for metal complexes

3. Results and Discussion

3.1. Molar Conductivity

Table 1 shows some physicochemical properties of dithiocarbamate ligands and their complexes along with molar conductivity for it (with concentration of 10^{-3}), DMF was used as solvent. The low values represented in Table 1, indicate that the complexes are non-electrolytic ones[21].

3.2. Electronic Spectra and Magnetic Susceptibility

Generally, the ligand cause the appearance of two strong adsorption bands at 236 and 328 nm due to n–π*, π-π* transitions. The intensity of these bands increases or decrees in complexes due to the coordination occurred. The tetra coordinated nickel II complexes, with a planar square shape, have diamagnetic moment resulting in two strong absorption bands, one of them at 400–600 nm and other at 300–343 nm. This relates to these reactions below:[22]

$^1A_{1g} \rightarrow {}^1A_{2g}$ (1)

$^1A_{1g} \rightarrow {}^1B_{1g}$ (2)

The complexes of Ni (II) exhibit adsorption bands at 329 and 411 nm due to ($^1A_{1g} \rightarrow {}^1B_{1g}$, $^1A_{1g} \rightarrow {}^1A_{2g}$), respectively. Other absorption bands are listed in Table 2 and depicted in Figs. 1–4. According to the literature data, Pd (II) and Pt (II) tetra coordinated complexes have a square plane form[13]. The prepared complexes of Pd (II) and Pt (II) were diamagnetic, and showed absorption bands at 655,718 nm and 496,647 nm referring to ($^1A_{1g} \rightarrow {}^1T_{2g}$, $^1A_{1g} \rightarrow {}^1T_{1g}$) and ($^1A_{1g} \rightarrow {}^1A_{2g}$, $^1A_{1g} \rightarrow {}^1B_{1g}$) respectively. The other bands are shown in Table 2.

3.3. FT- IR Spectral Studies

The infrared spectrum of dithiocarbamate compounds can be divided into three regions. The first region at 1400–1570 cm^{-1} belongs to the C-N group. The length of the C-N bond lies between that of a single and double bond. Also, the bond order lies between 1 and 2 because of the resonance state in dithiocarbamate. The second region is between 950–1050 cm^{-1} and 610–720 cm^{-1}, and this refers to the ν_{as} (C-S) and ν_s (C-S) wavelength of the asymmetric and symmetrical bands[14, 23–25]. The third region is within 325–519 cm^{-1} and belongs to ν (M-S)[3]. In the FT-IR spectra of the prepared complexes, adsorption bands at 3161–3331 cm^{-1}, 1418–1471 cm^{-1}, 995–1109 cm^{-1}, 630–697 cm^{-1}, and 425–431 cm^{-1} can be found due to ν(NH), ν(N-CS_2), ν_{as}(C-S), ν_s(C-S), and (M-S) stretching vibrations, respectively (Table 3). The FT-IR spectra of the ligands differ from those of their complexes either in the highest or lowest frequency, as shown in Figs. 5–8, and this is evidence of coordination.

Table 1. Physciochemical properties, molar conductivity values, and elemental analysis of the ligand and the prepared complexes

No.	Compounds	% yield	Color	M.P, ^0C	Molar conductivity Λ_m, S cm^2 mol^{-1}	C % cal	H % cal	N % cal	S % cal
						(C %) found	(H %) found	(N %) found	(S %) found
1.	L	90	a pale yellow	195–197	8.25	16.77 (15.68)	1.11 (1.83)	15.64 (16.32)	35.82 (34.79)
2.	$[Ni_2(L)_2]$	75	light green	278–280	10.16	17.73 (16.91)	1.18 (1.25)	16.53 (16.86)	37.87 (37.21)
3.	$[Pd_2(L)_2]$	80	brown	260–262	22.50	15.55 (16.23)	1.03 (1.11)	14.5 (14.11)	33.22 (32.71)
4.	$[Pt_2(L)_2]$	77	brown	273–275	19.42	12.64 (11.81)	0.84 (1.26)	11.79 (10.91)	26.99 (27.37)

Table 2. UV-Visible and magnetic moment data of the complexes

No.	Compounds	wave length, nm	Wave number, cm^{-1}	Transitions	μ_{eff} B.M	Geometric shape
1.	L	236	42372	$n \rightarrow \pi^*$	–	–
		328	30487	$\pi \rightarrow \pi^*$		
2.	$[Ni_2L_2]$	206	48544	$n \rightarrow \pi^*$	Dia	Square planer
		245	40816	$\pi \rightarrow \pi^*$		
		329	30395	$^1A_{1g} \rightarrow {}^1B_{1g}$		
		411	24331	$^1A_{1g} \rightarrow {}^1A_{2g}$		
3.	$[Pd_2L_2]$	254	39370	$n \rightarrow \pi^*$	Dia	Square planer
		400	25000	$\pi \rightarrow \pi^*$		
		496	20161	$^1A_{1g} \rightarrow {}^1A_{2g}$		
		647	15456	$^1A_{1g} \rightarrow {}^1B_{1g}$		
4.	$[Pt_2L_2]$	227	44053	$n \rightarrow \pi^*$	Dia	Square planer
		265	37736	$\pi \rightarrow \pi^*$		
		387	25840	CT		
		655	15267	$^1A_{1g} \rightarrow {}^1T_{2g}$		
		718	13928	$^1A_{1g} \rightarrow {}^1T_{1g}$		

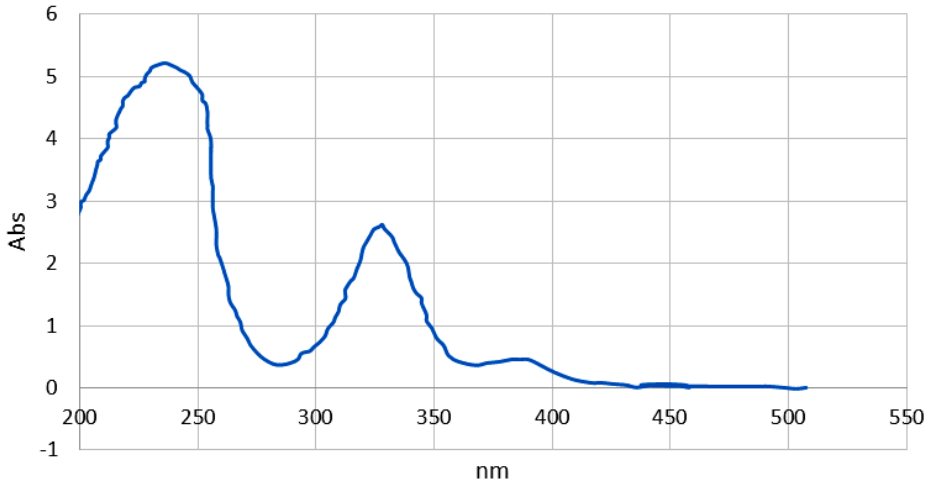


Fig. 1. UV-vis spectrum of the ligand K_2L

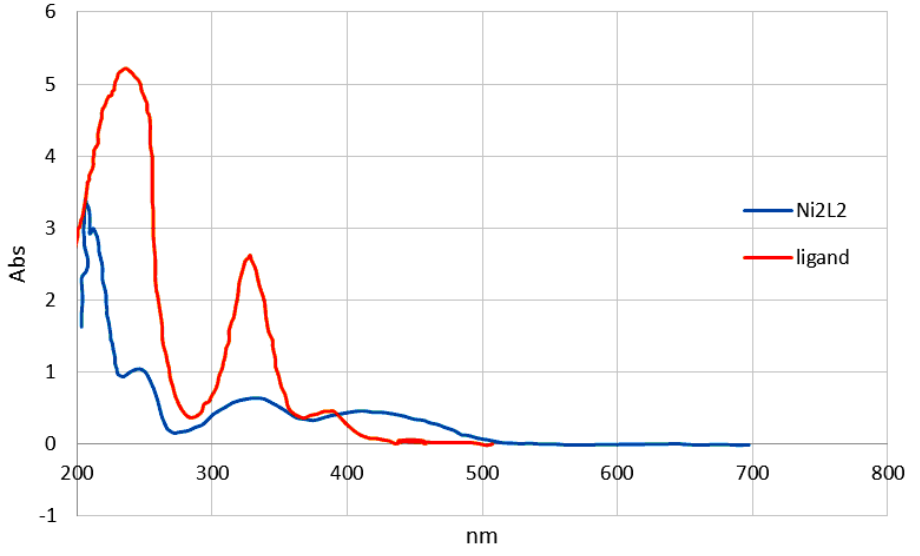


Fig. 2. UV-vis spectrum of the compound $[Ni_2 L_2]$

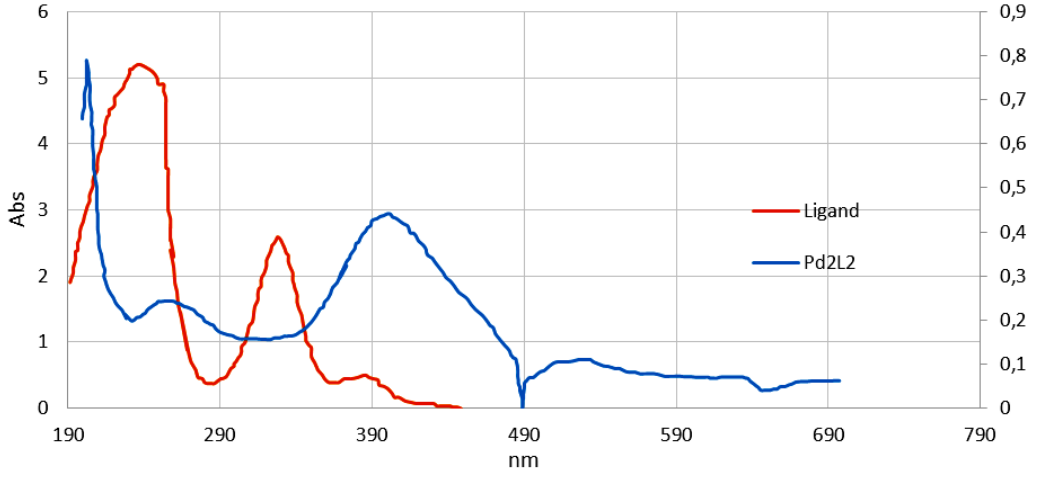


Fig. 3. UV-vis spectrum of the compound $[Pd_2 L_2]$

Table 3. IR data (400–4000 cm^{-1}) of ligands and their metal complexes

Compounds	ν (NH)	ν (N-CS_2)	ν_{as} (C-S)	ν_s (C-S)	ν (M-S)
L	3214	1418s	1040	645	–
[Ni_2 L_2]	3331	1458s	995	655	425
[Pd_2 L_2]	3161	1471s	1109	697	427
[Pt_2 L_2]	3199	1442s	1044	630	431

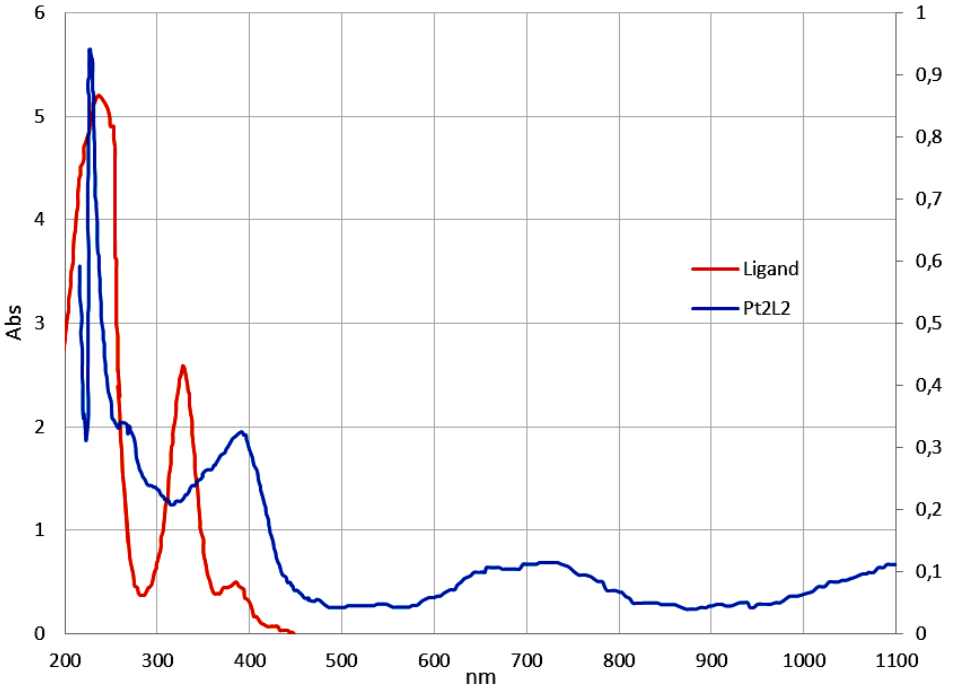


Fig. 4. UV-vis spectrum of the compound [Pt_2 L_2]

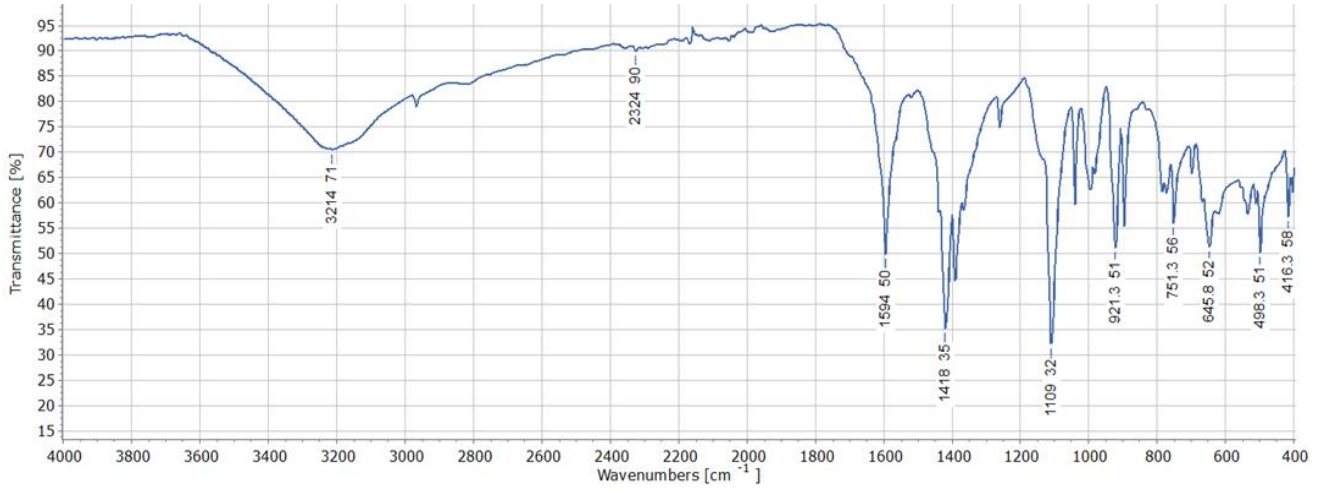


Fig 5. Infrared spectrum of the L ligand

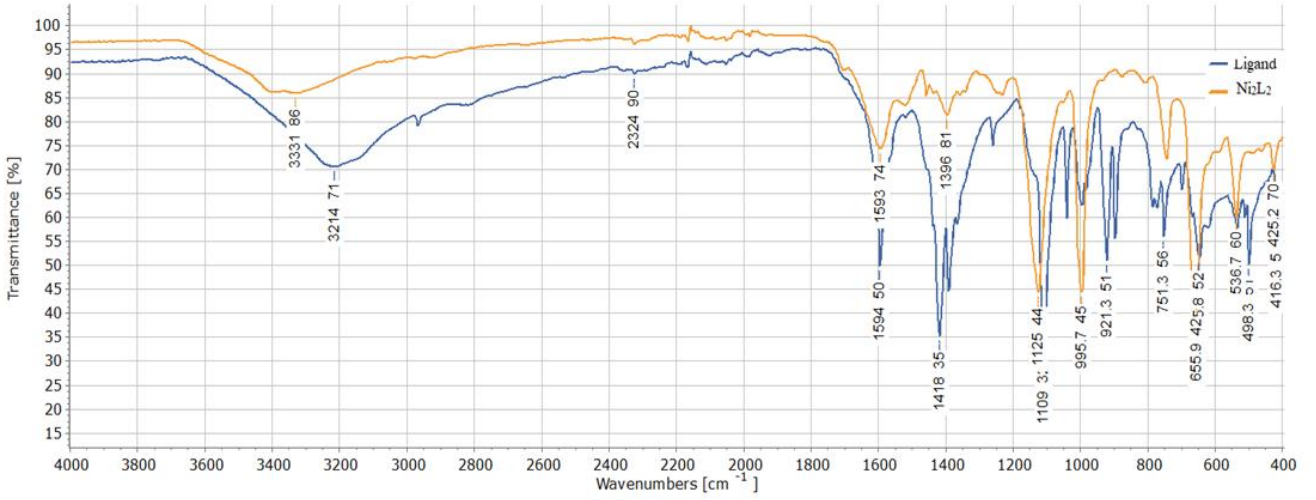


Fig. 6. Infrared spectrum of [Ni_2 L_2]

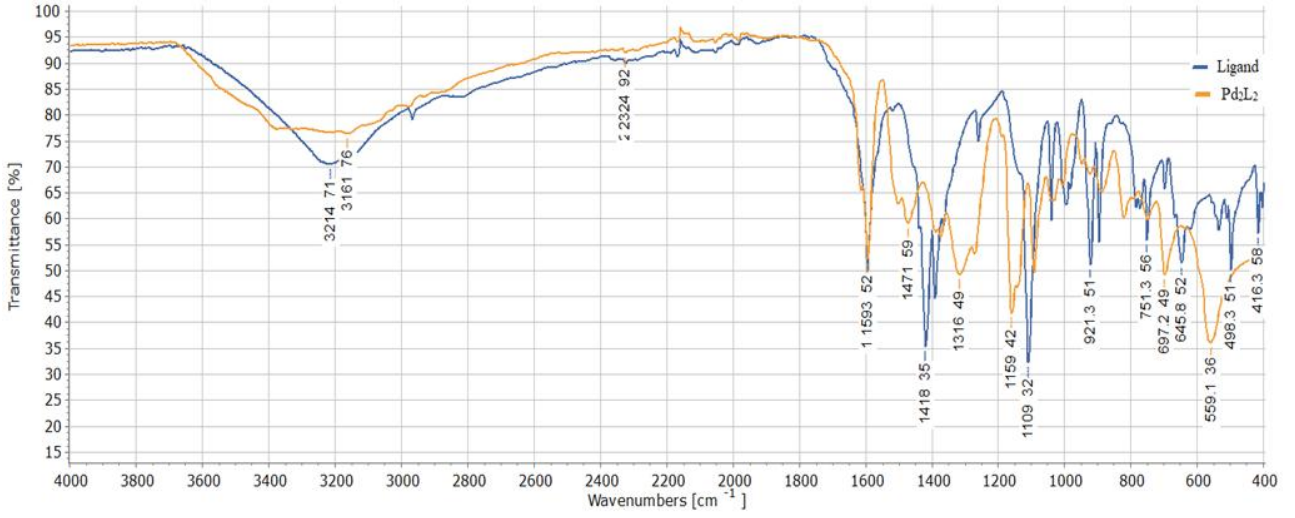


Fig. 7. Infrared spectrum of [Pd_2 L_2]

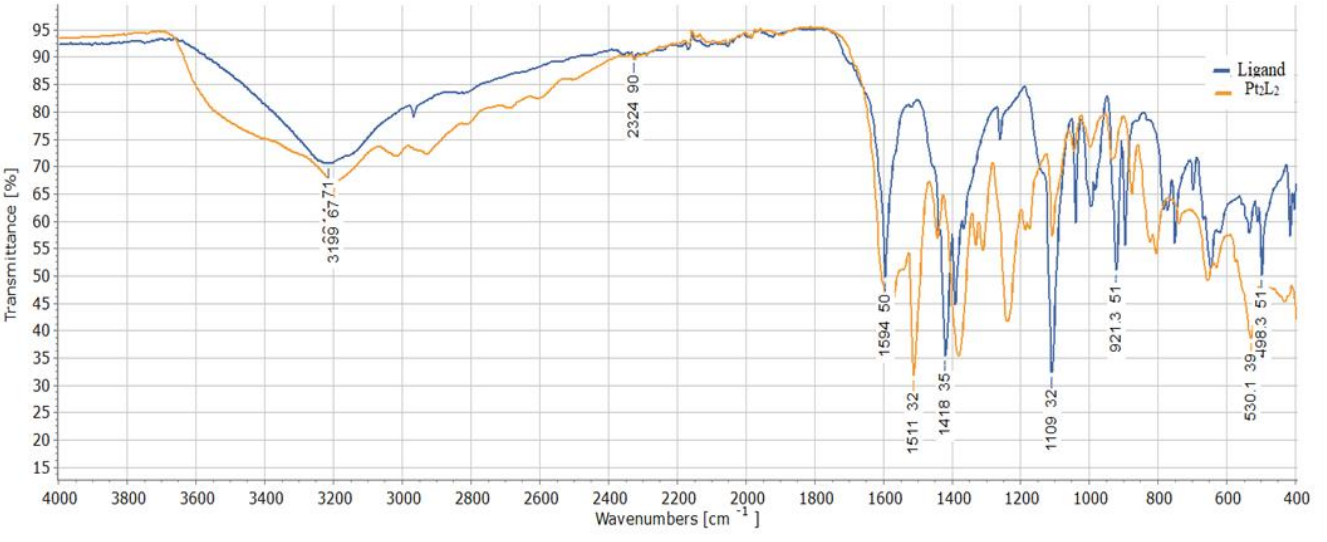


Fig. 8. Infrared spectrum of [Pt_2 L_2]

3.4. Determination of Antibacterial Activity

The inhibitory effect of the prepared complexes $[Ni_2L_2]$, $[Pd_2L_2]$, $[Pt_2L_2]$ was studied using three bacteria: *Escherichia coli, Pseudomonas aeruginosa, and Staphylococcus aureus*. The complexes showed different efficacy compared to control solutions containing antibiotics Chloramphenicol and Gentamycin[26, 27]. Inhibition measurements showed that the nickel complex is the most effective complex in relation to the three used bacteria, as shown in Table 4 and Fig. 9.

3.5. Scanning Electron Microscopy (SEM) Studies

The surface topography of the complexes was studied through electron microscopy. It can be concluded that the complexes have crystalline properties and the minimum particle size is less than 100 micrometer. It should be noted that the surface of the complexes differs from each other in terms of geometric shape due to the fact that some of them are in the form of layers[8, 27, 28].

Table 4. Inhibitory effect of prepared compounds against the used bacteria.

Comp.	Gram-negative bacteria						Gram-positive bacteria		
	Bacteria E.coli			*Bacteria Pseudomonas aeruginosa*			*Bacteria Staphylococcus aureus*		
	Zone of Inhibition, mm								
	Conc., mg/mL 50	Conc., mg/mL 100	Conc., mg/mL 150	Conc., mg/mL 50	Conc., mg/mL 100	Conc., mg/mL 150	Conc., mg/mL 50	Conc., mg/mL 100	Conc., mg/mL 150
$[Ni_2L_2]$	8	14	35	11	22	23	9	13	24
$[Pd_2L_2]$	6	13	21	16	17	22	11	14	18
$[Pt_2L_2]$	11	16	22	7	13	16	17	20	23
Chloramphenicol (10 mg/disc)	17	17	17	18	18	18	15	15	15
Gentamycin (10 mg/disc)	14	14	14	13	13	13	15	15	15

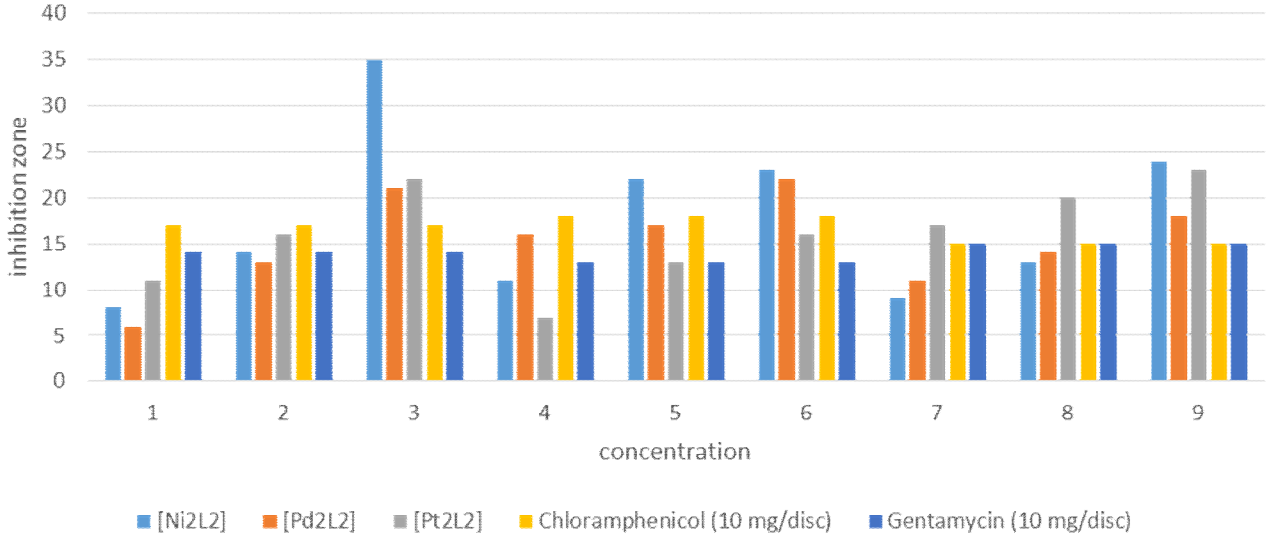


Fig. 9. Biological activities of the metal complexes against the three bacteria

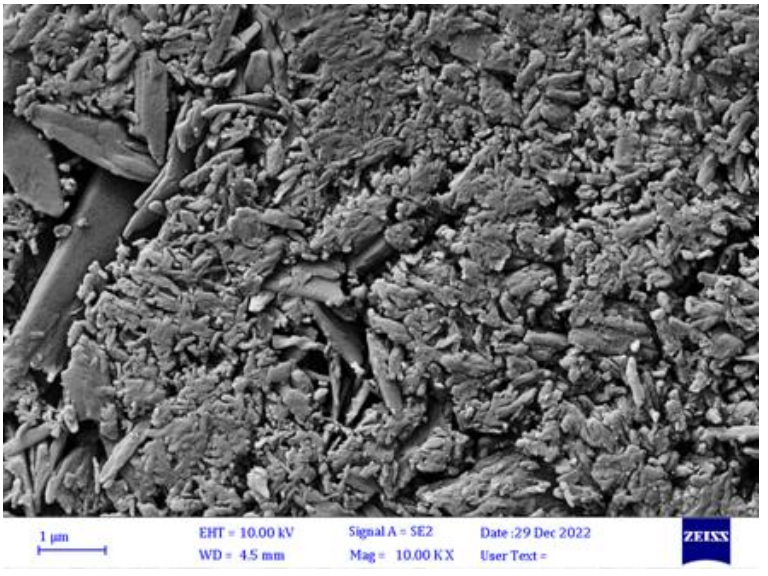


Fig. 10. SEM image of $[Ni_2L_2]$ complex

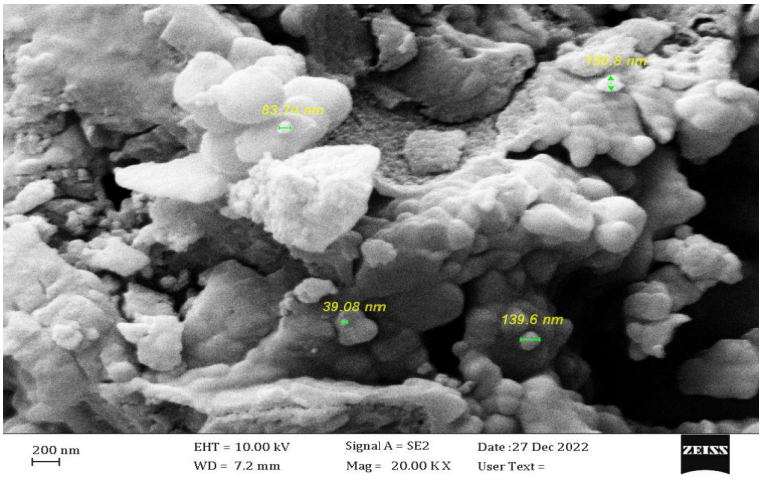


Fig. 11. SEM image of $[Pt_2L_2]$ complex

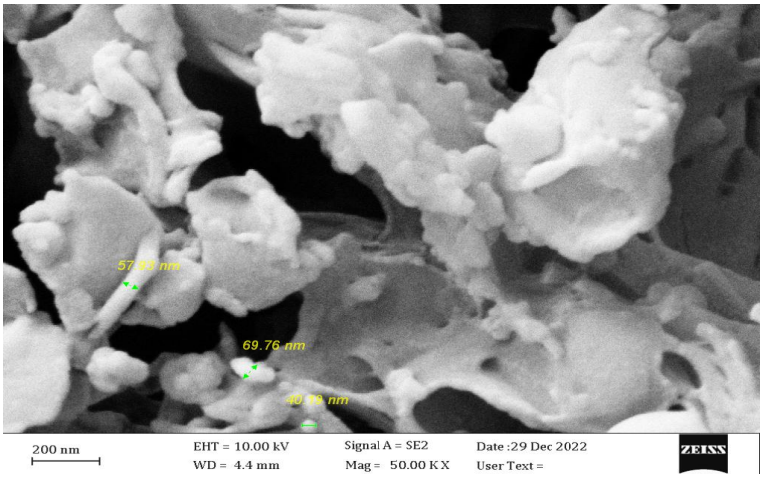


Fig. 12. SEM image of $[Pd_2L_2]$ complex

4. Conclusions

In this research, the potassium ligand 2,2'-malonylbis (hydrazine-1-carbodithioate) was synthesized starting from diethyl malonate and hydrazine to obtain malonyl hydrazine, and the last step was the addition of carbon disulfide. Dithiocarbamate ligand with bifunctional group was prepared. The diamagnetic and tetracoordinate complexes of Ni (II), Pd (II) and Pt (II) exhibit non-electrolytic behavior and have a planar square geometry. This is due to low values of molar conductivity indicating that the prepared complexes are non-electrolytes, and the dithiocarbamate ligand acts as bidentate through sulfur atoms. Results of spectral data analysis confirmed the ligand synthesis using H NMR and ^{13}C NMR technique. The results of scanning electron microscopy showed that the surface of the complexes is aggregated in crystalline form. Additionally, studying the inhibitory effect of the prepared complexes against the bacteria Escherichia coli, Pseudomonas aeruginosa, and Staphylococcus aureus, showed that the Ni (II) complex was more effective, especially against Pseudomonas aeruginosa.

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БІНУКЛЕАРНІ МАЛОНОГІДРАЗИДНІ ДИТІОКАРБАМАТНІ КОМПЛЕКСИ Ni (II), Pd (II) ТА Pt (II): СИНТЕЗ, ХАРАКТЕРИЗАЦІЯ, АНТИМІКРОБНА АКТИВНІСТЬ І СЕМ ДОСЛІДЖЕННЯ

Анотація. *Одержано бінуклеарні комплекси формули [M2L2], де L – калій 2,2'-малонілбіс(гідразин-1-карбодитіоат), M – Ni (II), Pd (II) і Pt (II). Кожен комплекс охарактеризовано за допомогою елементного аналізу (CHNS), УФ- та ІЧ-спектрів, спектрів протонного та вуглецевого ядерного магнітного резонансу (^{1}H ЯМР і ^{13}C ЯМР), магнітної чутливості, сканувальної електронної мікроскопії (СЕМ) і молярної провідності.*

Ключові слова: *дитіокарбамат, металокомплекси, біологічна активність, СЕМ дослідження.*