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SYNTHESIS AND CHARACTERIZATION OF A NUMBER OF COMPLEXES OF Pt(II), Pd(II), Cd(II) AND Zn(II) WITH NEW LIGANDS CONTAINING OXYGEN

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ABSTRACT. New mononuclear complexes of some transition metal ion with di-dentate ligand have been synthetized and characterized. The Pt(II), Pd(II), Cd(II) and Zn(II) complexes were synthesized by directly reaction of the above ligand with metal chlorides in (1:1) (ligand:metal) molar ratio in ethanolic medium. Formulated complexes classified by elementary analysis, spectral exploration (FTIR, UV/Vis), ^{1}H-NMR measure, conduction measure and electronic spectra rates suggest tetrahedral geometric for every metal complex, with the exception of Pt(II) complexes, which have octahedral geometry, and Pd(II) compound, which have square planar geometry and the study of inhibiting effect on the growth of different types of bacteria which are strains *staph. aurous* and *Klebsiella pneumoniae.* The results indicate that all complexes have strong effect on different types of bacteria.

KEY WORDS: Dicarboxlate, Direct reaction, Tetrahedral geometry, Ligand, Mononuclear, Complexes

INTRODUCTION

Studies on inorganic and organic structures are among the most common due to their importance in electrical conduction [1], magnetic conduction [2], photomechanics [3], chemical catalysis [4], guest and host chemistry [5], ion exchange [6], and some fibrous flame-resistant materials [7]. Monocarboxyl ligands are considered among the bonds of interest to chemists, as many metal atoms are linked in different ways, producing distinctive structures that have magnetic and electronic properties [8].

Significant number of research have been conducted on transition metal composite and their association with carboxylates, and metal carboxylates have been highlighted as being very important from a practical and biocatalytic standpoint [9]. Metal anions show distinct uses over metal cations. Metal carboxylates and dicarboxylates have previously been shown to have activity as antimicrobials [10]. Coumarins and coumarin complexes have also shown activity as antimicrobial and chemotherapeutics [11]. Combining coumarins and carboxylates or dicarboxylates allows for the synthsis of a novel series of complexes with interesting biological activity. According to chromatographic performance, electronic, and NMR techniques, the complex $[(NH_3)_5Co(dicarboxylateCo(NH_3)_5]X_4$ (dicarboxylate = oxalate, succinate, pimelate, fumarate, and maleate; X = ClO_4^- or Cl^-) is one of five recent complexes produced by the reaction of $[Co(hydrogen\ dicarboxylate)(NH_3)_5](ClO_4)_2$ with $[Co(NH_3)_5H_2O](ClO_4)_3$ [12].

As reported by Tabata baee *et al.* [13], μ-OCO-pydc)Cu(bipy)(H_2O) is the complex [(bipy)(pydc)Cu]. As novel copper complexes (**1**), 3.5H_2O is also produced as {[(μ2-C_2O_4)2,2'-bipy)Cu.2H_2O (**2**) has been hydrothermally produced (pydcH_2 = pyridine-2,6-dicarboxilic acid, bipy = 2,2'-bipyridine). Both complexes were categorized using singular crystal X-ray diffracting studies, elementary analysis, and infrared spectroscopy.

The first one carries a pair of neutral molecules. Each fragment distorts the octahedral geometry of the ion center to metal. The second coordinate polymer also distorted the octahedral geometry and has some H-bonds as well as π-π interaction of the 3D network.

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In this work, first of all, two types of ligands are prepared from the reaction succinic anhydride with sodium hydroxide for (L_1) and glutaric acid with sodium hydroxide for (L_2) and describes their coordination behavior towards Pt(II), Pd(II), Zn(II), and Cd(II) and screened of their antimicrobial action against *Staphs aurous* and *Klebsiella pneumonia* using paper disc diffusion method.

EXPERIMENTAL

Every chemical was of reagent grade and was acquired from a (FLUKA and BDH).

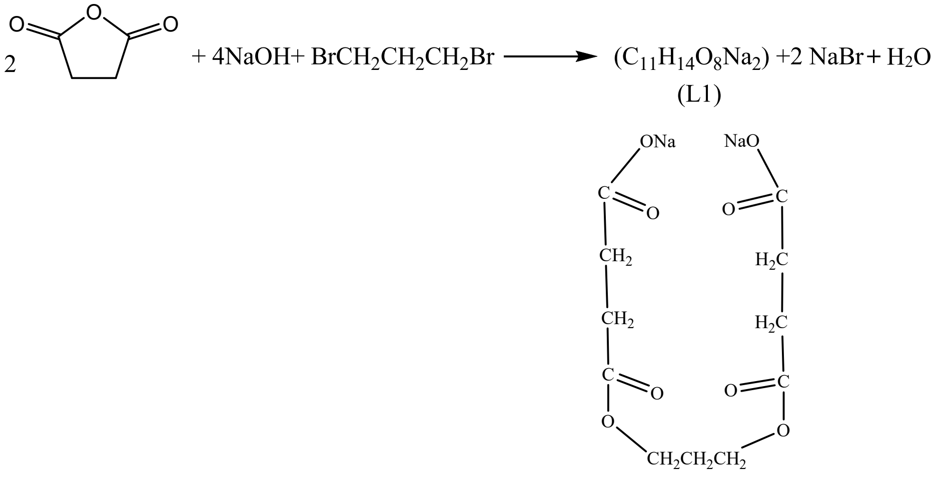
Physical characterization

Infrared spectra were recorded as CsI discs in the range 400-4000 cm^{-1}. Utilizing element (CHN) Analyzer LCHN-ALO Labtrone, elemental analyses (C and H) were carried out utilizing micro analytical techniques. Metal content were determined spectrophotometrically using AA670 atomic absorption. To measure the conductivity need to use 10^{-3} M of the DMSO complexes at temperature 25 °C solution utilization the conductivity meter PCM3 Jenway at the ambient temperature.

Preparation of the ligands

Preparation of 1,3-di(sodium succinate) propane (L_1)

Succinic anhydride (2.0 g, 0.02 mol) in 20 mL of ethanol was mixed with sodium hydroxide (1.6 g, 0.04 mol) in 15 mL ethanol, the mixture was added with moving to a solution of 1,3-dibromopropane (2.02 g, 0.01 mol) in 10 mL of ethanol. The resulting mixture stirred under reflux for 6 h. A white precipitation was made up. The NaBr was removed by filtering of the precipitate, and then the precipitate washing with ethanol several times then diethyl ether then dried under the vacuum for several hours as in equation shown in Scheme 1. Table 1 displays the color and yield of the ligand's elemental microanalysis data.

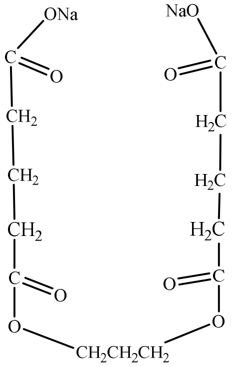


Scheme 1. Preparation of ligand 1,3-di(sodium succinate) propane (L_1).

Preparation of 1,3-di(sodium glutarate) propane (L_2)

Glutaric acid (2.6 g, 0.02 mol) in 10 mL ethanol was mixed with sodium hydroxide (1.6 g, 0.04 mol) in 15 mL ethanol, the mixture was added to a solution of 1,3-dibromopropane (2.02 g, 0.01 mol) in 10 mL of the ethanol. The resulting combination stirred upon reflux for 6 h. A white precipitate was formed and filtered off to remove NaBr. As shown in Scheme 2, the precipitation was repeatedly washed with ethanol, followed by diethyl ether, and then dry under vacuum for a number of hours. The elemental microanalysis data color and yield for the ligand are displayed in Table 1.

$$2HOOCCH_2CH_2CH_2COOH+2NaOH+BrCH_2CH_2CH_2Br \longrightarrow \underset{(L_2)}{C_{13}H_{18}O_8Na_2}+2NaBr+2H_2O$$



Scheme 2. Preparation of ligand 1,3-di(sodium glutarate) propane (L_2).

Preparation of complexes

Complexes of the formula [M(L_1)] and Na_2[Pt(L_1)Cl_2] (M = Pd(II), Zn(II), and Cd(II))

To prepare the $Na_2[Pt(L_1)Cl_2]$ and $[M(L_1)]$ complexes, (4.15 g, 0.01 mol) of K_2PtCl_4 or (1.77 g, 0.01 mol) of $PdCl_2$ or (1.36 g, 0.01 mol) of $ZnCl_2$ or (1.83 g, 0.01 mol) of $CdCl_2$ were disbanded in 10 mL of dilute hydrochloric acid, and the solving was then added to a solution containing (0.31 g, 0.01 mol) of L_1 dissolved in 10 mL of methanol. The resulting mixing was stirred under reflux for 2 h. Filter the precipitate and then wash with diethyl ether, and dry in vacuum for various hours. The data related to the colors and production of these composite are recorded in Table 1.

Complexes of the formula [M(L_2)] and Na_2[Pt(L_2)Cl_2] (M = Pd(II), Zn(II) and Cd(II))

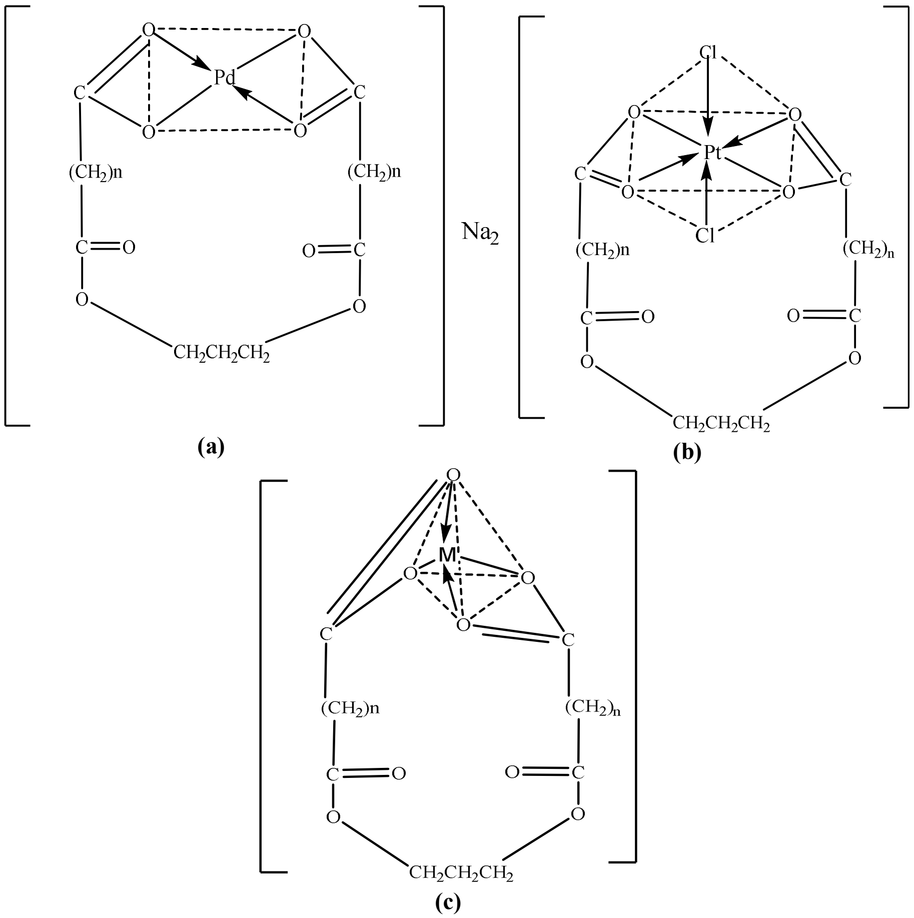
(4.15 g, 0.01 mol) of K_2PtCl_4 was dissolving in 10 mL dilute HCl, then added to a solution of (3.48 g, 0.01 mol) of L_2 dissolved in hot 10 mL of the ethanol, result combination stirred according to reflux for 2 h. Filter the formed precipitate, then it was washed by the diethyl ether then drying under vacuum for several hours. To prepare $[M(L_2)]$, we used (1.77 g, 0.01 mol) of $PdCl_2$ or (1.36 g, 0.01 mol) of $ZnCl_2$ or (1.83 g, 0.01 mol) of $CdCl_2$, then follow the same steps above.

RESULTS AND DISCUSSION

The precise analysis of metallic elements such as platinum was conducted using accurate and validated methods. Literature studies highlight effective microanalysis techniques for identifying metallic particles in environmental samples [15, 16]. This supports the analytical methods employed in this study to accurately characterize the prepared metal complexes.

The ligands (shown in Schemes 1 and 2) were synthesized and employed to create eight stable complexes containing Pt(II), Pd(II), Zn(II), and Cd(II) in a 1:1 (M:L) ratio. The synthesized compounds were soluble in most organic solvents, stable in dry air, and had melting points ranging from 210 to 300 $^{\circ}$C. Table 1 summarizes the complexes' analytic and physical features. The analytical data for the complexes were compatible with the proposed formulations: $[M(L_n)]$, where M = Pd(II), Zn(II), Cd(II)) and n = 1, 2. $Na_2[Pt(Ln)Cl_2]$ for platinum Pt(II).

The molar conduction measurements of the synthesized composite in DMSO showed that the complexes are non-electrolytes expect the two composite (**1** and **5**) were (2:1) electrolytes, Table 1. Figure 1 illustrates the proposed structure of synthesized complexes.



(a) (b)

(c)

Figure 1. Suggested structure of (a): $[Pd(L_n)]$, (b): $Na_2[Pt(L_n)Cl_2]$ and (c): $[M(L_n)]$ complexes. M = Zn(II) and Cd(II), n = 2, 3.

Elemental analysis

Elemental analysis recorded one of the particularly valuable procedures accessible to identify a compound [14]. Element investigation data about some prepared complexes are outlined in Table 1 which is persistent with indicated preparation.

Molar conductivity measurements

The molar conductivities of all formed composite were measurable in dimethyl sulfoxide (DMSO) at room temp. (25 °C) for 10^{-3} M solutions. Values of molar conductivity ($cm^2 \cdot ohm^{-1} \cdot mol^{-1}$) in this solvent are provided in Table 1. The observed values ranged between 12-18 $cm^2.ohm^{-1}.mol^{-1}$, suggest the non-electrolytic nature of composite [15, 16]. However, it is worth noting that the measured molar conductance values (12-19 $cm^2.ohm^{-1}.mol^{-1}$) deviate from those typically expected for 2:1 electrolytes, complexes (**1** and **5**) (76 and 73 $cm^2.ohm^{-1}.mol^{-1}$) [17]. This highlights the importance of further investigating the structural and solution behavior of complexes to confirm their electrolytic nature and alignment with the proposed structures.

Table 1. Physical characteristic of produced ligands and their composite.

Seq.	Compound	Color	m.p. (°C)	Yield %	Analysis, found (calc.)%			(Λ)(DMSO) $cm^2.ohm^{-1}.mol^{-1}$
					C	H	M	
L_1	$C_{11}H_{14}O_8Na_2$	White	>300	62	41.26 (41.04)	4.41 (4.35)	---	---
L_2	$C_{13}H_{18}O_8Na_2$	White	>300	66	44.84 (44.73)	5.21 (4.18)	---	---
1	$Na_2[Pt(L_1)Cl_2]$	Olive green	213^d	80	24.46 (24.08)	2.61 (2.45)	36.11 (36.08)	76
2	$[Pd(L_1)]$	Black	222^d	53	34.71 (34.66)	3.71 (3.65)	27.96 (27.26)	15
3	$[Zn(L_1)]$	Off white	245^d	82	38.90 (38.57)	4.16 (4.08)	19.25 (19.07)	17
4	$[Cd(L_1)]$	White	280^d	41	34.17 (34.12)	3.65 (3.60)	29.07 (29.09)	19
5	$Na_2[Pt(L_2)Cl_2]$	Green	235^d	80	27.48 (27.46)	3.19 (3.08)	34.33 (34.25)	73
6	$[Pd(L_2)]$	Black	220^d	49	38.20 (38.15)	4.44 (4.31)	26.04 (26.01)	12
7	$[Zn(L_2)]$	White	210^d	52	42.47 (42.53)	4.94 (4.91)	17.78 (17.48)	14
8	$[Cd(L_2)]$	White	270^d	70	37.65 (37.47)	4.38 (4.58)	27.11 (27.04)	18

d = decomposition temperature.

Electronic spectra

Electronic spectra of prepared ligands (L_1, L_2) consist of two absorption bands in the range (31657-36349 cm^{-1}) as shown in Table 2, assigned to (n-π^* and π-π^*) substantially [18].

Electronic spectra of prepared Pt(II) complexes (**1** and **5**) consist of two absorption band in the value (14657, 15642 cm^{-1}) corresponding to (ν_1) ($^1A_1g \rightarrow {}^1T_2g$) transition and (11546, 13645 cm^{-1}) corresponding to (ν_2) ($^1A_1g \rightarrow {}^1T_1g$) transition the position of (ν_1, ν_2) agree with the structure of octahedral complexes [19].

Electronic spectra of prepared Pd(II) complexes (**2** and **6**) consist of three absorption band in the value (15635, 14378 cm^{-1}) corresponding to (ν_1) ($^1A_1g \rightarrow {}^1A_2g$) transition and (14235, 21657 cm^{-1}) correspondent to (ν_2) ($^1A_1 \rightarrow {}^1B_1g$) transition and (21456, 30976 cm^{-1}) corresponding to (ν_3) ($^1A_1g \rightarrow {}^1Eg$) the position of ($\nu_1$, ν_2, ν_3) agree with the structure of square planer complexes [20].

Electronic spectra of prepared Cd(II) and Zn(II) complex (**3**, **4**, **7** and **8**) complexes showing no absorption peak at rang (33456-37658 cm^{-1}) that it show no d-d electronic transit induced d^{10} system in apparent area, which in a good consequence of Cd(II) and Zn(II) tetrahedral complexes [19, 21].

Table 2. Electronic spectrum of ligands and complexes.

No.	Complex	Transition		C.T.
L_1	$C_{11}H_{14}O_8Na_2$	32761 35643	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	31657
L_2	$C_{13}H_{18}O_8Na_2$	36349 34276	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	32743
1	$Na_2[Pt(L_1)Cl_2]$	14657 11546	$^1A_1g \rightarrow {}^1T_2g$ $^1A_1 \rightarrow {}^1T_1g$	32267
2	$[Pd(L_1)]$	15635 14235 21456	$^1A_1g \rightarrow {}^1A_2g$ $^1A_1 \rightarrow {}^1B_1g$ $^1A_1g \rightarrow {}^1Eg$	31098
3	$[Zn(L_1)]$	----	----	31850
4	$[Cd(L_1)]$	----	----	31658
5	$Na_2[Pt(L_2)Cl_2]$	15642 13645	$^1A_1g \rightarrow {}^1T_2g$ $^1A_1 \rightarrow {}^1T_1g$	----
6	$[Pd(L_2)]$	14378 21657 30976	$^1A_1g \rightarrow {}^1A_2g$ $^1A_1 \rightarrow {}^1B_1g$ $^1A_1g \rightarrow {}^1Eg$	----
7	$[Zn(L_2)]$	----	----	33456
8	$[Cd(L_2)]$	----	----	32578

The mean transition energy for $Na_2[Pt(L_n)Cl_2]$ is higher compared to $[Pd(L_n)]$, indicating a higher energy requirement for electronic transitions in the platinum complex. The standard deviation for $Na_2[Pt(L_n)Cl_2]$ is relatively high, suggesting variability in transition energies within this complex. The complexes $[Zn(L_n)]$ and $[Cd(L_n)]$, $n = 1, 2$ each have only one transition energy, which means they exhibit a single electronic transition. Comparing transition (C.T.) energies across different complexes can provide insights into the impact of metal ions and ligands on electronic transitions (Table 2).

^{1}H-NMR spectra

^{1}H-NMR assignments for the compounds and composite, along with the ^{1}H-NMR spectra of L_1, L_2, plus a few complexes measured in DMSO-d_6 solvent, are shown in (Table 3) [22, 23].

Table 3. The ^{1}H-NMR. data of ligand.

	δ (ppm)	Assignment
L_1	5.89-6.12	O-CH_2 (m, 4H)
	1.89	C-CH_2-C (s, 2H)
	1.77	CH_2 (s, 10H)
L_2	5.92-6.36	O-CH_2 (m, 4H)
	1.86-1.89	C-CH_2-C (s, 2H)
	2.31-2.40	CH_2 (m, 12H)

s = singlets, m = multiplet.

IR- spectra

Compounds with a certain frequency of electromagnetic radiation in the infrared spectrum are absorbed by any organic or inorganic compound with covalent bonds [24].

Used as a KBr disk with a range of 400-4000 cm^{-1} to recording infrared spectrum of prepared ligands as in Table 4. The infrared spectra of the ligand and their complexes are shown in Figure 2. All prepared two ligands had their infrared spectra studied, as the infrared spectrum is used to study the linking of ligands for metals. The band at (1227, 1534 cm^{-1}) and (1226,1586 cm^{-1}) in IR spectra of L_1 and L_2 supportive the forming of $\nu_{asym}(COO^-)$ and $\nu_{sym}(COO^-)$ as listed in Table 4 which agree with the reported values [25-27]. Stretching vibration of ν(C=O) in the ligands (L_1, L_2) has been prepared in the range (1627, 1724 cm^{-1}) which agree with the reported values [28]. In complexes the ν(C=O) shifted to lower frequency which it is coordinated to the metal ions [29, 30].

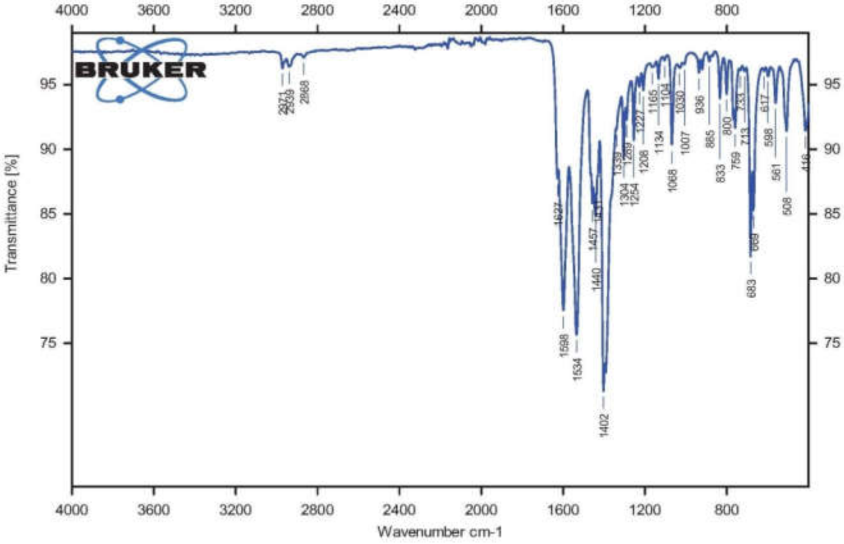
Aliphatic ν(C-H) stretching of ligands (L_1, L_2) shows band in the region (2971, 2932cm^{-1}) [31]. The prepared metal complexes displayed band in the range (2917-2951 cm^{-1}) as listed in Table 4. Showing that this band was shifted to different values [32].

Stretching vibration of ν(C-O) in ligands (L_1, L_2) has been prepared in the value (1208, 1226 cm^{-1}) which agree with the prepared values [33]. And the complexes have appeared in the range (1235-1286 cm^{-1}) as listed in Table 4 showing that this band was shifted to different values [34, 35].

The stretching vibration of ν(M-O) in metal complexes have appeared in range (431-492 cm^{-1}) are listed in Table 4 which is consistent with the reported values [36-38] such band did not appeared in the spectrum of the free ligands which indicated that the coordination between the metal and the ligands occur through the oxygen atoms [39-42].

Table 4. IR bands of L and their composite.

No.	Compounds	$\nu_{as}(COO^-)$	$\nu_s(COO^-)$	ν(C=O)	ν(C-H)	ν(C-O)	ν(M-O)
L_1	$C_{11}H_{14}O_8Na_2$	1534	1227	1627	2971	1208	---
L_2	$C_{13}H_{18}O_8Na_2$	1586	1226	1724	2932	1226	---
1	$Na_2[Pt(L_1)Cl_2]$	1556	1222	1723	2951	1255	434
2	$[Pd(L_1)]$	1574	1252	1696	2937	1254	467
3	$[Zn(L_1)]$	1527	1262	1622	2946	1246	433
4	$[Cd(L_1)]$	1549	1232	1644	2932	1286	431
5	$Na_2[Pt(L_2)Cl_2]$	1574	1237	1714	2927	1237	492
6	$[Pd(L_2)]$	1538	1253	1773	2917	1235	482
7	$[Zn(L_2)]$	1534	1257	1627	2939	1266	475
8	$[Cd(L_2)]$	1554	1254	1774	2927	1236	470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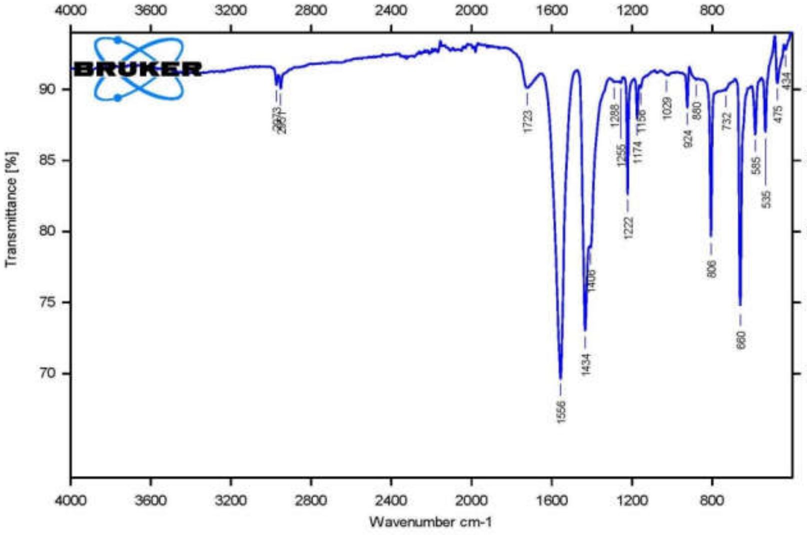
(a)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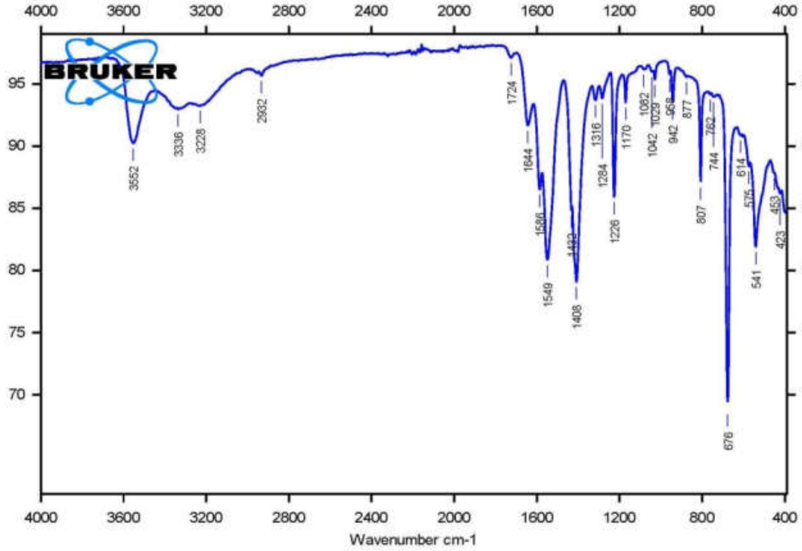
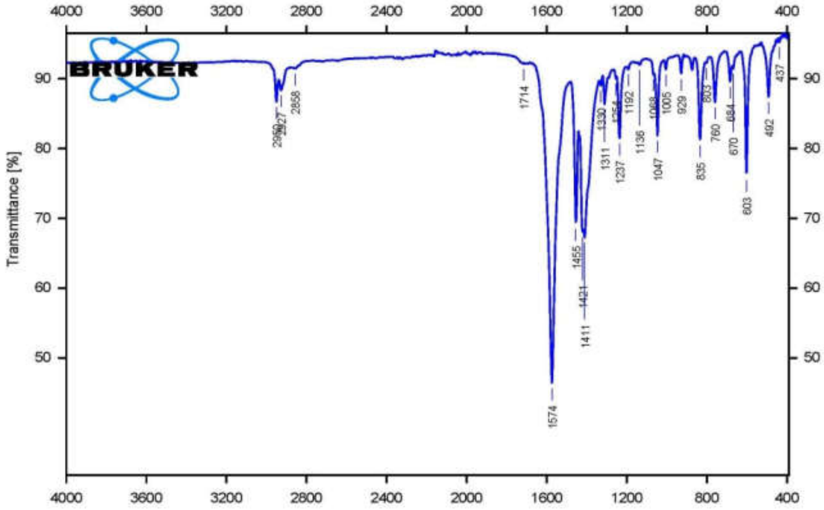
(b)



(c)



(d)

Figure 2. The IR spectrum of (a) L_1, (b) $Na_2[Pt(L_1)Cl_2]$, (c) L_2, and (d) $Na_2[Pt(L_2)Cl_2]$.

Antimicrobial activity

The electrical conductivity of the composite was calculated in DMSO as this solvent provides stable and suitable conditions for analyzing physical and chemical of produced complexes. Although solubility in water or lipids is a prerequisite for biological activity, many organic and metal complexes exhibit low solubility in water, which may limit their study in an aqueous medium.

Furthermore, studying electrical conductivity in an aqueous medium could complicate the results due to potential interactions between water and the complexes, such as hydration or dissociation, which could lead to inaccurate findings regarding the electroplated nature of the composite.

On the other hand, biological activity measurements (such as antimicrobial activity) are usually conducted in aqueous or aqueous-organic media, demonstrating that the complexes are somewhat soluble in these media and exhibit their biological effects. Nevertheless, conducting additional studies on electrical conductivity in an aqueous medium could be a future step to gain a more comprehensive understanding of the complexes' behavior under biological conditions.

In comparison to specific species of (G$^+$) *Staphylococcus aurous* and (G$^-$) *Klebsiella pneumoniae*, the estimated biologic action of ligands with complexes was examined at a concentration of 10 µg/1 mL, Table 5 and Figure 3, show the derived results, which are comparable to the control medication, Cipro. The assessment shows that ligands and their composite have an action on the bacteria used in this study that is described as less than effective of control.

Table 5. Antimicrobial action of the ligand with complexes.

No.	Symbol	compounds	*Staphylococcus aurous* (mm)	*Klebsiella pneumoniae* (mm)
1	L_1	L_1	16	25
2	L_2	L_2	15	22
3	M1	$[Pd(L_1)]$	17	27
4	M2	$Na_2[Pt(L_1)Cl_2]$	18	26
5	M5	$[Pd(L_2)]$	19	28
6	M6	$Na_2[Pt(L_2)Cl_2]$	18	27
Control			20	30

Figure 3. Compounds with antibacterial properties.

CONCLUSION

Several chemical compounds were synthesized in this research. Then studied to understand their properties chemically. These prepared compounds showed they could dissolve well in solvents which shed light on how they interact chemically. Elemental analysis was performed to verify that the structures created matched the expected outcomes of the synthesis process as planned. The molar conductivity tests indicated variations, from the values seen in 1 to 1 or 1 to 2 electrolytes due to the ionization of these compounds in solution. Moreover analyzing the transition energies within compounds demonstrated how metal ions and ligands affect transitions emphasizing how the chemical structure plays a significant role, in altering the optical characteristics of these substances. In terms of effects found in the study data demonstrated that the ligands and their combinations showed antibacterial properties, against the bacteria tested but were not as effective as ciprofloxacin which implies that making changes to their structure could improve their ability to fight microbes better. These results provide insights into how chemical structure influences the biological characteristics of these combinations which could open up opportunities for use, in healthcare and drug development fields.

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