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DIVALENT TRANSITION METAL COMPLEXES WITH MIXED OF β-ENAMINONE AND N,O-DONOR LIGANDS: SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL ASSESSMENT

Feryal Ali M. and Alyaa S.M.O. Al-barwari*

Department of Chemistry, College of Education for Pure Science, University of Mosul, Iraq

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ABSTRACT. This study extensively describes the synthesizing and characterizing of new β-enaminone complexes derived from dimedone and amines coordinated with transition metals Fe(II), Co(II), Ni(II), and Cu(II), also two ligand's metformin (Met) and 8-hydroxyquinoline (8-hq). These were determined via FT-IR, ^{1}H-NMR, electron and mass spectroscopy, molar electrical conductivity, thermal analysis (TGA, DTA and DSC) and characterized by metal content determination (%), magnetic susceptibility and elemental analysis (C.H.N.) and SEM technique. The measurements indicate that the complexes have six coordination numbers, where the β-enaminone is coordinated as tetradentate ligand through the two nitrogen atoms and the two oxygen atoms beside metformin is coordinated through the two nitrogen atoms and 8-hydroxyquinoline is coordinated by oxygen and nitrogen atoms as bidentate ligands. The conductance suggests that all complexes have electrolytic behavior, being conductive in a ratio of (1:2). SEM results revealed the presence of nanostructures on sample surfaces. The biological activity of the prepared ligands and complexes are studied in different concentrations for four types of bacteria *Staphylococcus aureus* has (+G) and *Escherichia coli*, *Pseudomonas aeruginosa*, *Klebsiella* have (-G), well as, the influence of the fungus. The ligands have disparate effectiveness while complexes have high effectiveness inhibiting the bacteria and fungus.

KEY WORDS: Mixed ligand systems, Dimedone, β-Enaminone ligands, Transition metals, Biological application

INTRODUCTION

The ligand β-enaminone is one of the most important compounds that plays a role in organic synthesis and is considered a raw material in the preparation of biologically active compounds that are used as anticonvulsants and inhibitors such as (oxytocin) and acetylcholine-steysane inhibitors [1]. The β-enaminone derivatives are used to synthesize various biologically active antibacterial, anti-epileptic compounds, inflammatory and tumor [2]. Many catalysts have been used in the direct condensation of amines with β-dicarbonyl compounds as a suitable synthesis method for producing β-enaminone. It is also utilized in the preparation of complexes of metallic elements Mn(II), Co(II), Ni(II), Cu(II), Cd(II) and others. These complexes are used in many fields. Cobalt complexes are used in many industries, including electronics, dyes, mining, and paint. However, these materials and their residues in river water increase the concentration of cobalt in water, and this leads to pollution and the incidence of some diseases, including paralysis, diarrhea, decreased blood pressure, and others [3].

β-enaminone is an important organic intermediate. Enaminones are used in the preparation of heterocyclic, amino alcohols, azo compounds, amino acids, and quinolones [4]. Biological properties of 5,5-dimethyl-1,3-cyclohexanedione (dimedone) and its derivatives include inorganic compound anti-cancers, antihistamines, antioxidants, and anticoagulants [5]. One of its main uses is in green operations [6, 7]. These enaminones are usually prepared from the reaction of aromatic and aliphatic amines with β-dicarbonyl compounds using a catalyst, as well as without the use of a catalyst [8].

In 2020, the researcher Waheed *et al.* prepared the compound β-enaminone from the reaction of dimedone with 4-aminoacetophenone. To prepare the complexes, the ligand reacts with

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*Corresponding authors. E-mail: solve415@uomosul.edu.iq divalent metals Mn(II), Co(II), Ni(II). Cu(II), Zn(II) and Cd(II) with another ligand, 3-aminophenol. The ligand and the prepared complexes have been identified by FT-IR, ^{1}H, ^{13}C-NMR, UV-Visible spectrum, mass spectrometry and thermal analysis [9]. During in 2021, the researcher Kadhom *et al.* prepared β-enaminone ligands from the reaction of trimethoprim with dimedone, and their complexes interacting with a group of ions of transitional and non-transitional elements Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) characterization of the ligand and the prepared complexes by (^{1}H, ^{13}C-NMR), FT-IR, UV, mass spectrum, molar conductivity, C.H.N., and atomic absorption and magnetic susceptibility. It showed that the resulting shape is octahedral. It also showed that some complexes have a paramagnetic property and some of them are diamagnetic. It showed the biological effectiveness of the prepared ligands complexes and demonstrated antibacterial and antifungal activities [10].

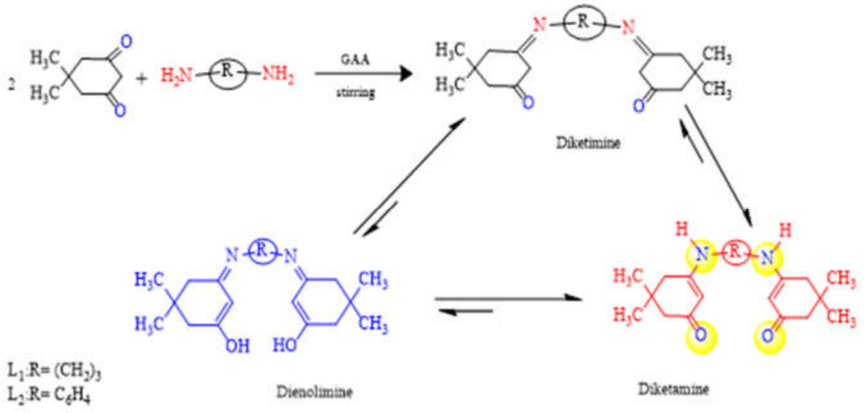
Later, in 2022, researcher Abdulsalam *et al.* prepared compounds from the reaction of dimedone with furosemide, producing beta-enaminone compounds, and their interaction with divalent metal elements Co(II), Ni(II), Cd(II), Pd(II) and Pt(II) moreover 8-hydroxyquinoline was added to it. The complexes are prepared in a ratio of (1:1:1), coordination was achieved through nitrogen and oxygen atoms. The proposed shape was octahedral due to the presence of two chlorine atoms inside the coordination sphere. The biological effectiveness of these complexes against bacteria has been studied and it has been varied [11].

In this research, we aim to investigate the possibility of synthesizing metal-β-enaminone complexes with mixed ligands, a series of novel divalent metals ion complexes have been synthesized and characterized, besides, using SEM techniques. We have studied the properties of nanoparticles of some of the above complexes.

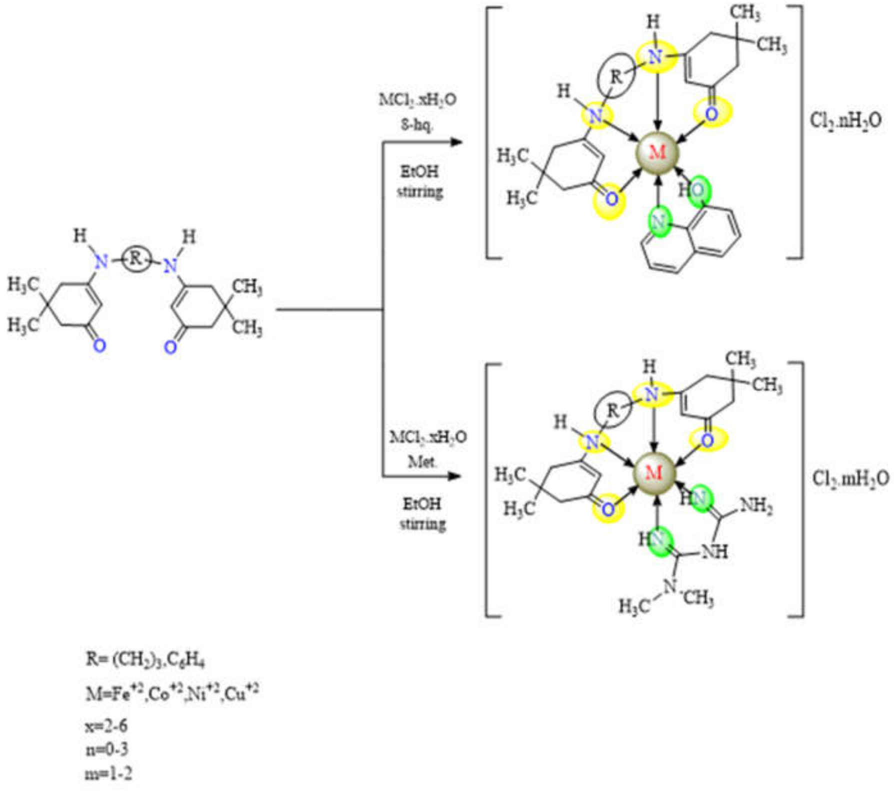
EXPERMENTAL

Devices and materials

The materials used were prepared by Fluka, Aldrich and BDH companies. The ligand and there complexes were detected through various measurements: An UV-Visible spectrophotometer (UV Spectrophometr PG instruments) was used to measure the ultraviolet spectra in a binary solvent such as formamide, with the measurements conducted at the University of Mosul, College of Education for Pure Science. FTIR measurement was performed using two devices - one at the University of Mosul, College of Science, covering the range 400-4000 cm^{-1}, and another at Tikrit University, College of Education for Pure Science, a Shimadzu device in the range 400 – 4000 cm^{-1}, using KBr disks. The electrical conductivity was measured on a Conductive Meter-Model (Eutech pc700) device, with the samples melted in DMF at a concentration of 10^{-3} M and a temperature of 25 °C, and the melting point determined using the Aparatues-stuart-smp Melting Point device at Mosul University, College of Education for Pure Science. Magnetic susceptibility was measured using the Gouy method at a temperature of 25 °C, utilizing the Magnetic Susceptibility Balance device at Tikrit University, College of Education for Pure Science. The elements (C.H.N.) of the ligands and complexes were analyzed using a device (Eleminter German) at the University of Tehran in Iran, and the percentage of metals was determined by atomic absorption at the University of Mosul, College of Agriculture and Forestry, using a device (Analytik JenaNovaA350). Finally, H^1-NMR measurement was performed at the University of Tehran in Iran using a Varian Agilent USA type device, with the samples dissolved in deuterium DMSOd$_6$ at room temperature, and mass spectra measurement was also carried out at the University of Tehran in Iran using a USA-Agilent device.



Scheme 1. Preparation of ligands.



Scheme 2. Preparation of complexes.

Preparation of ligands L_1, L_2

Dimedone (2.8 g, 0.02 mol) was placed in a 250 mL round flask and dissolved with 30 mL absolute ethanol, and 0.74 g, 0.01 mol of 1,3-propanediamine/or 1.08 g, 0.01 mol of *p*-phenylenediamine was and dissolved in 25 mL of absolute ethanol, it was added to the reaction slowly, then we added a few drops of glacial acetic acid, and left to stir and heat for four hours. The reaction was followed up with TLC to ensure the reaction was complete. The precipitate was left to settle, filtered, washed with methanol and ether, left to dry, and recrystallized with ethanol [12].

Preparation of the complexes

(0.318 g, 1 mmol) of L_1 and (0.352 g, 1 mmol) of L_2 was dissolved in 20 mL absolute ethanol and placed in a 250 mL round-bottom flask, then added (0.19 g, 1 mmol) of $FeCl_2.4H_2O$ dissolved in 15 mL absolute ethanol and left to sublimate and stir for half an hour, then weigh (0.16 g, 1 mmol) of metformin/or (0.145 g, 1 mmol.) of 8-hydroxyquinoline dissolved in 15 mL absolute ethanol and add it to the mixture then leave to complete the interaction for 8 hours and followed up with TLC. The precipitate was left to settle, after which it was filtered and washed with methanol and ether and left for 24 hours to dry, and recrystallized with ethanol. The rest of the complexes were prepared in the same way by using the special weights of each the metal salts: (0.23 g, 1 mmol $CoCl_2.6H_2O$), (0.23 g, 1 mmol $NiCl_2.6H_2O$), and (0.17 g, 1 mmol $CuCl_2.2H_2O$).

RESULTS AND DISCUSSION

The prepared compounds were characterized using conductance measurements, magnetic susceptibility, elemental analysis (%), metal percentage (%), (C.H.N.), FT-IR, UV-Vis, ^{1}H-NMR and mass spectroscopy.

Atomic absorption

The amount of divalent iron, cobalt, nickel, and copper in the complexes was analyzed spectrophotometrically. The process was carried out by digesting the complexes using concentrated nitric acid, then the solution was supplemented to the specified volume with distilled water. It was shown that the concentrations of the prepared complexes fell within the range of concentrations consistent with the linear range of the standard curve, and the values were very close to the theoretically calculated value [13]. The measurement is shown in Table 1.

Analysis of elements (C.H.N.)

The ligands and complexes were analyzed to confirm their proposed formulas, and it was found that there is a great agreement between the practical and theoretical ratios. The values are shown in Table 1

Molar electrical conductivity

The molar conductivity of the complexes was measured using a binary solvent such as Dimethyl formamide with a concentration of 10^{-3} M at 25 ^{o}C. The conductivity values howed that they are consistent with the proposed formulas for the complexes, as it was shown that all complexes are conductive in a ratio of (1:2). The values are shown in Table 1 [15].

Table 1. Chemical formulas and some physical properties of the ligands and complexes.

No.	Molecular formula	M. wt. g/mol	M.p. °C	Color	Yield %	C% Cal. (Exp.)	H% Cal. (Exp.)	N% Cal. (Exp.)	M% Cal. (Exp.)	Molar cond. $\Omega^{-1}cm^{2}\,mol^{-1}$
L_1	$C_{19}H_{30}O_2N_2$	318	280 -282	Light yellow	74	71. 69 (71.52)	9.43 (9.25)	8.80 (8.50)	--	--
L_2	$C_{22}H_{28}O_2N_2$	352	350D*	Yellow	80	75.00 (75.15)	7.95 (7.72)	7.95 (7.64)	--	--
1	$[Fe(L_1)(Met)]Cl_2.2H_2O$	610	330D*	Yiddish brown	72	45.24 (45.09)	7.27 (7.15)	16.06 (15.99)	9.15 (9.08)	115
2	$[Fe(L_1)(8\text{-}hq)]Cl_2.2H_2O$	625.85	300D*	Brown	80	49.85 (49.72)	6.55 (6.34)	6.71 (6.55)	8.92 (8.73)	162
3	$[Co(L_1)(Met)]Cl_2.2H_2O$	613.09	350D*	Dark olive	74	45.01 (44.90)	7.33 (7.12)	15.98 (15.80)	9.61 (9.42)	147
4	$[Co(L_1)(8\text{-}hq)]Cl_2.2H_2O$	628.93	300D*	Yellowish green	62	49.60 (49.45)	6.51 (6.34)	6.67 (6.48)	9.36 (9.19)	167
5	$[Ni(L_1)(Met)]Cl_2.2H_2O$	612.85	320D*	Yiddish green	55	45.93 (45.78)	7.34 (7.12)	15.99 (15.80)	9.57 (9.35)	139
6	$[Ni(L_1)(8\text{-}hq)]Cl_2.3H_2O$	646.69	290	Light green	87	48.24 (48.15)	6.33 (6.21)	6.49 (6.38)	9.07 (8.99)	179
7	$[Cu(L_1)(Met)]Cl_2.2H_2O$	617.71	340	Dark brown	69	44.68 (44.42)	7.28 (7.12)	15.86 (15.67)	10.28 (10.13)	148
8	$[Cu(L_1)(8\text{-}hq)]Cl_2$	597.55	330	Light brawn	76	52.22 (52.09)	6.86 (6.57)	7.02 (6.94)	10.63 (10.44)	165
9	$[Fe(L_2)(Met)]Cl_2.2H_2O$	644.00	250	Earthy yellow	88	48.44 (48.25)	6.67 (6.47)	15.21 (15.10)	8.67 (8.42)	158
10	$[Fe(L_2)(8\text{-}hq)]Cl_2.2H_2O$	659.74	360D*	Light brawn	70	56.38 (56.18)	5.91 (5.72)	6.36 (6.24)	8.46 (8.36)	167
11	$[Co(L_2)(Met)]Cl_2.2H_2O$	647.09	300D*	Light green	80	48.21 (48.13)	6.64 (6.48)	15.14 (14.99)	9.10 (8.92)	125
12	$[Co(L_2)(8\text{-}hq)]Cl_2.2H_2O$	662.93	360D*	Olive	64	56.11 (55.98)	5.88 (5.68)	6.33 (6.23)	8.88 (8.69)	134
13	$[Ni(L_2)(Met)]Cl_2.2H_2O$	646.85	330D*	Light yellow	68	48.23 (48.12)	6.64 (6.46)	15.15 (15.09)	9.07 (8.98)	173
14	$[Ni(L_2)(8\text{-}hq)]Cl_2.2H_2O$	662.69	300D*	Greenish yellow	85	56.13 (55.97)	5.88 (5.68)	6.33 (6.16)	8.85 (8.55)	165
15	$[Cu(L_2)(Met)]Cl_2.H_2O$	633.71	280	Bluish-green	71	49.23 (49.10)	6.46 (6.23)	15.46 (15.28)	10.02 (9.90)	118
16	$[Cu(L_2)(8\text{-}hq)]Cl_2$	631.55	320D*	Cumin yellow	88	58.90 (58.65)	5.54 (5.30)	6.65 (6.48)	10.06 (9.96)	154

Thin layer chromatography

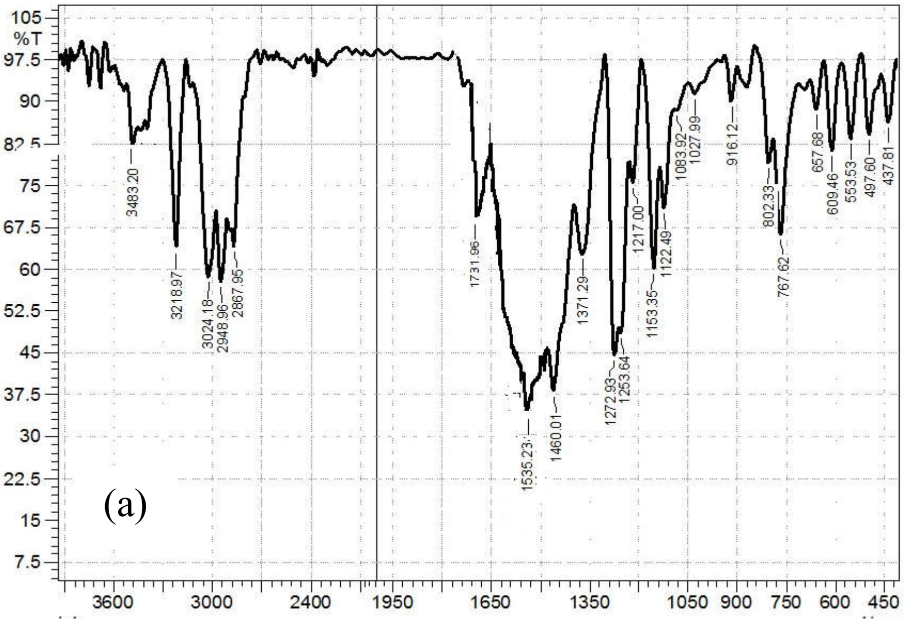
This technique was used to ensure the completeness of the reaction during the preparation of coordination complexes. The use of TLC strip, potassium permanganate solution, and lancet aldehyde solution helps to verify the completion of the reaction between the reactants when preparing the coordination complex. This is crucial to ensure the desired product is obtained with high purity and assist in determining the optimal conditions (such as concentration, temperature, and reaction time) for the efficient preparation of the complex. In addition, this technique is rapid and easy to perform, making it suitable for both laboratory and industrial applications in the field of coordination complex synthesis [16].

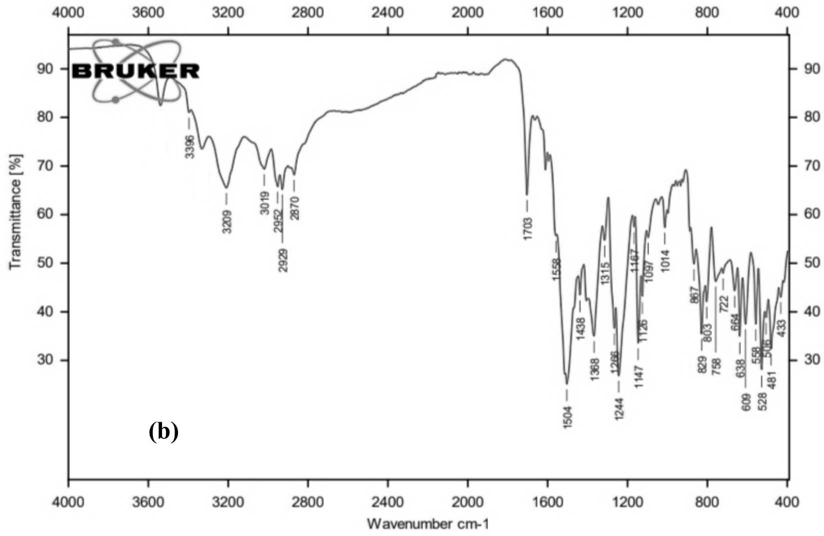
Infrared spectra

The infrared spectrum showed appropriate shifts for the ligand due to the formation of the complex, as it showed a C=O band at 1731 and 1703 cm^{-1} in the ligands shifted towards lower values with a range of (1608-1690) cm^{-1}. This confirms the coordination of the ligand with the metal via the oxygen atom [17] and the appearance of a band at 3218 and 3209 cm^{-1} in the ligands it comes back to NH, as this value was shifted to a range of 3148-3205 cm^{-1}. In the complexes, there is evidence of the coordination of the nitrogen atom with the metal. Likewise, in the C=C bands, values appeared at 1445-1559 cm^{-1} and (C-N) at 1209-1278 cm^{-1} [18, 19]. The appearance of new M-O and M-N bands in the complexes is evidence of consistency with the metallic atom [20]. The values are shown in Table 2.

Table 2. Infrared spectra of the prepared ligands and complexes.

No.	Molecular Formula	N-H	C=O	C=C	C-N	M-O	M-N	H_2O
L_1	$C_{19}H_{30}O_2N_2$	3218	1731	1535	1272	--	--	--
L_2	$C_{22}H_{28}O_2N_2$	3209	1703	1504	1242	--	--	--
1	$[Fe(L_1)(Met)]Cl_2.2H_2O$	3202	1657	1505	1260	550	450	3530
2	$[Fe(L_1)(8\text{-}hq)]Cl_2.2H_2O$	3188	1637	1559	1239	528	486	3416
3	$[Co(L_1)(Met)Cl_2.2H_2O$	3190	1650	1515	1240	535	420	3540
4	$[Co\ (L_1)(8\text{-}hq)Cl_2.2H_2O$	3164	1639	1498	1240	503	447	3556
5	$[Ni(L_1)(Met)]Cl_2.2H_2O$	3203	1652	1525	1209	518	447	3458
6	$[Ni\ (L_1)(8\text{-}hq)]Cl_2.3H_2O$	3184	1670	1490	1230	570	435	3580
7	$[Cu(L_1)(Met)]Cl_2.2H_2O$	3175	1685	1510	1260	545	428	3612
8	$[Cu(L_1)(8\text{-}hq)]Cl_2$	3195	1630	1503	1210	580	445	--
9	$[Fe(L_2)(Met)]Cl_2.2H_2O$	3205	1670	1491	1242	565	418	3490
10	$[Fe(L_2)(8\text{-}hq)]Cl_2.2H_2O$	3185	1645	1515	1255	575	427	3490
11	$[Co(L_2)(Met)]Cl_2.2H_2O$	3184	1608	1529	1278	592	437	3546
12	$[Co(L_2)(8\text{-}hq)Cl_2.2H_2O$	3148	1652	1558	1272	513	442	3431
13	$[Ni(L_2)(Met)]Cl_2.2H_2O$	3155	1658	1529	1244	514	453	3627
14	$[Ni(L_2)(8\text{-}hq)]Cl_2.2H_2O$	3150	1690	1480	1255	534	490	3460
15	$[Cu(L_2)(Met)]Cl_2.H_2O$	3190	1675	1466	1230	580	432	3522
16	$[Cu(L_2)(8\text{-}hq)]Cl_2$	3176	1680	1445	1215	525	445	--

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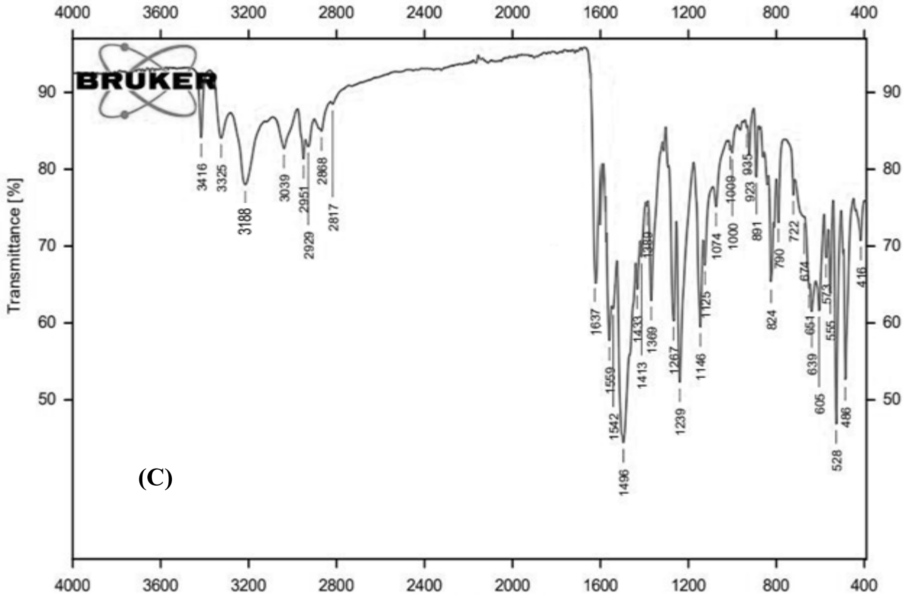
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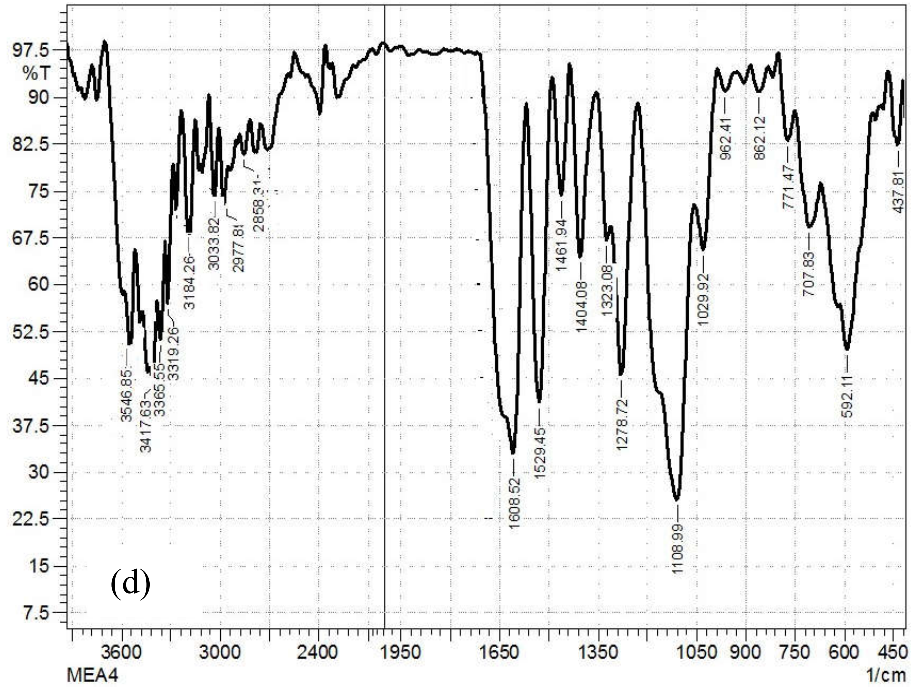


Figure 1. Infrared spectra of (a) = L_1, (b) = L_2, (c) = complex **2** and (d) complex **11**.

H^1-NMR measurement

The ligand (L_1) was measured using a nuclear magnetic resonance spectrometer, where the solvent DMSO-d_6 was used, and the measurement reference was tetramethylsilane (TMS) [21, 22]. The ligand spectrum is presented in Figure 2 and the values are shown in Table 3.

Table 3. ^{1}H-NMR data for ligand L_1 evaluated in DMSO–d^6 and chemical shift in ppm.

No.	Active group	ppm	No.	Active group	ppm
1	C_1 (CH_3)	0.98 (6H,s)	5	(NH)	8.7 (1H,s)
2	C_2 (CH_2)	2.03 (2H,s)	6	C_6(CH_2)	3.6 (2H,t)
3	C_3 (CH_2)	2.35 (2H,s)	7	C_7(CH_2)	4.3 (2H,m)
4	C_4 (CH)	5.2 (1H,s)	-	-	-

Mass spectrum

It is used to determine the molecular formula of the prepared ligands and their dissociation methods. This process is affected by the type of solvent, the type of detector used, the ionization energy, and the partial weight. The mass spectrum of the ligand (L_2) was measured, showing a peak at 352, and this corresponds to the partial weight of the compound and the proposed formula [23].

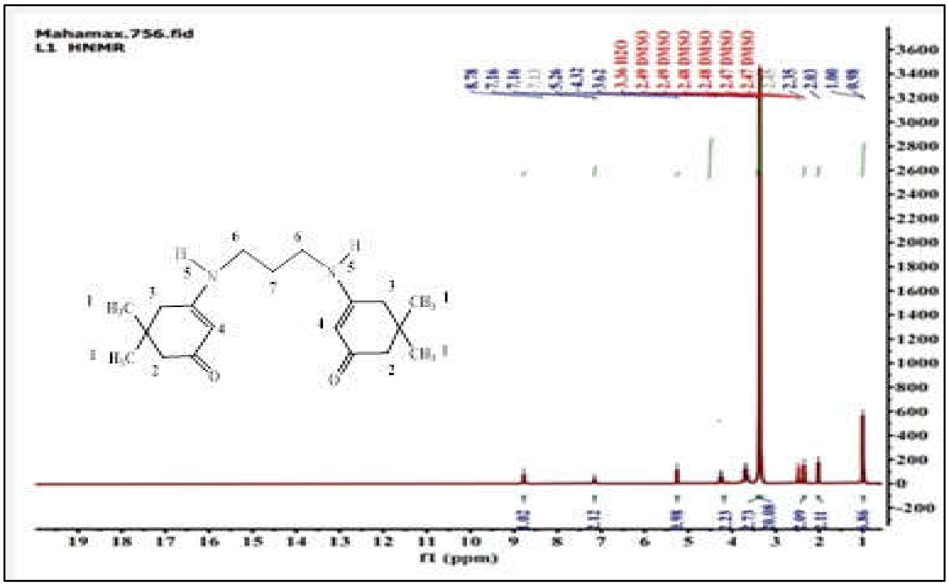


Figure 2. The ^{1}H-NMR spectra for the ligand L_1.

Table 4. Mass spectra data for ligand L_2.

No.	Fragment	Mass/charge (m/z)	Relative intensity (%)	No.	Fragment	Mass/charge (m/z)	Relative intensity (%)
1	$[C_{22}H_{28}N_2O]^+$	352.4	2.6	6	$[C_9H_9]^+$	117.1	5.2
2	$[C_{11}H_{12}NO]^+$	217.4	6	7	$[C_8H_{11}]^+$	107.2	21
3	$[C_{11}H_9N]^+$	155	5	8	$[C_6H_{11}]^+$	83.0	42
4	$[C_{10}H_{11}N]^+$	145.1	3.5	9	$[C_5H_7]^+$	67.0	100
5	$[C_{10}H_{10}]^+$	130.1	10.5	10	$[C_4H_7]^+$	55.1	4

Magnetic measurements

The effective magnetic moments of the complexes were measured at (25 °C) and the magnetism measurements confirmed that all the complexes with high paramagnetic properties took the octahedral shape, in contrast, the iron(II) complexes showed hexagonal symmetry (**1**, **2**, **9**, **10**). The magnetism values ranged (4.86 - 5.34) BM, confirming the presence of four individual electrons in the (d^6) system, with an increase in the value of the measured magnetic moment over the theoretically calculated value due to the orbital contribution. This is consistent with the eight bright, high-spin iron complexes [24]. The blocked cobalt complexes (**3**, **4**, **11**, **12**) had magnetic moment values between (3.75 - 4.06) BM. This indicates the presence of three single electrons in (d^7). The prepared nickel complexes (**5**, **6**, **13**, **14**) had magnetic moment values between (2.89 - 3.17) BM. These values confirm the presence of two lone electrons in (d^8) [25]. Copper complexes (**7**, **8**, **15**, **16**) have magnetic moment values ranging between (1.86 - 2.37) BM, and this indicates that they have an octahedral shape [26]. The values are shown in Table 5

Electronic spectra

Ultraviolet spectra were measured at a concentration of 10^{-3} M in DMF for the ligand and the complexes, where the ligand showed values in the range 29585-34246 cm^{-1} belongs to $n\rightarrow\pi^*$ and $\pi\rightarrow\pi^*$ transitions. The electronic spectrum of the octahedral iron complex (d^6) showed an absorption band in the range 11363-11792 cm^{-1}, it belongs to the $^{5}T_{2}g\rightarrow{}^{5}Eg$ transition with a charge transfer band in the range 34246-38167 cm^{-1}. Highly twisted octahedral cobalt complexes show absorption bands.

ν1 $^{4}T_{1}g(F) \rightarrow {}^{4}T_{2}g$ (11248-12345) cm^{-1}

ν2 $^{4}T_{1}g(F) \rightarrow {}^{4}A_{2}g$ (14705-16103) cm^{-1}

ν3 $^{4}T_{1}g(F) \rightarrow {}^{4}T_{1}g(P)$ (20533-25641) cm^{-1}

C.T. 33557-37678 cm^{-1}

Highly octahedral nickel complexes showed three bands [27]:

ν1 $^{3}A_{2}g \rightarrow {}^{3}T_{2}g(P)$ (11210-11792) cm^{-1}

ν2 $^{3}A_{2}g \rightarrow {}^{3}T_{1}g(F)$ (13020-19193) cm^{-1}

ν3 $^{3}A_{2}g \rightarrow {}^{3}T_{1}g(P)$ (21267-25773) cm^{-1}

C.T. 34129-40160 cm^{-1}

Highly twisted octahedral copper complexes showed a broad absorption band [28]:

ν1 $^{2}Eg \rightarrow {}^{2}T_{2}g$ 13812-16806 cm^{-1}

C.T. 33783-38022 cm^{-1}

Table 5. Electronic and magnetic spectra of the prepared ligands and complexes.

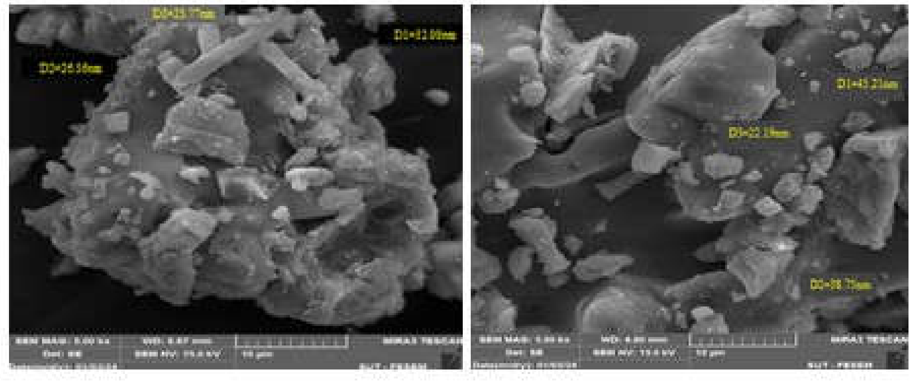
No.	ν1 cm^{-1}	ν2 cm^{-1}	ν3 cm^{-1}	C.T. cm^{-1}	μeff $B.M.$
L_1	29585	34246	--	--	--
L_2	29940	34013	--	--	--
1	11363	--	--	34246	4.86
2	11494	--	--	37735	5.09
3	11248	14880	21008	37678	4.06
4	12150	16103	20533	34722	3.75
5	11261	14705	23809	34482	3.08
6	11363	13020	24271	34246	2.97
7	15151	--	--	33783	1.99
8	16000	--	--	35971	2.00
9	11560	--	--	35335	5.34
10	11792	--	--	38167	4.97
11	12345	14814	20618	33557	3.95
12	11904	14705	25641	34246	3.84
13	11210	19193	25773	34129	3.17
14	11792	16180	21267	40160	2.89
15	13812	--	--	38022	1.86
16	16806	--	--	34843	2.37

Thermal analyses

The complexes prepared using the TGA, DTA, and DSC techniques were studied at a heating rate of 25 °C per min in the temperature range 0-600 °C. In the first heating stage (below 150 °C), there was no weight loss, indicating the absence of coordinated water molecules in the complex. In the later heating stages, a significant weight loss was observed accompanied by an increase in temperature, suggesting the decomposition of the organic ligands and the formation of the metal oxide [29, 30].

Study of the nanoscale properties of complexes using SEM

The scanning electron microscope relies on the use of a beam of high-energy electrons that interact with the sample to produce topographical images to generate a variety of signals on the surface of solid samples and can provide more information about the surface of the sample [31]. Figure 3 shows the SEM of nanoparticales for the complex $[Ni(L_1)(Met)]Cl_2.2H_2O$ with average particle size of 26 nm was aprovided. As well as seen nanoparticales 38 nm for the cobalt complex $[Co(L_2)(8\text{-}hq)]Cl_2.2H_2O$.



(a) (b)

Figure 3. The SEM images of the complexes (a) = $[Ni(L_1)(Met)]Cl_2.2H_2O$ and (b) = $[Co(L_2)(8\text{-}hq)]Cl_2.2H_2O$.

Biological efficacy

A biological test was conducted for the prepared ligands and their complexes on four types of bacteria, *Escherichia coli*, *Klebsiella*, *Stapyloccus aureus*, *Pseudomonas aeruginosa* and a type of fungus *Aspergillus niger*. The compounds were dissolved in DMSO, and solutions were prepared at a concentration of (10^{-2}, 10^{-3}) M. The bacteria were placed on the agar medium. Holes are made in which samples are placed. The dish is placed in an incubator at a temperature of 37 °C for 24 hours for bacteria and 28 °C for 48-72 hours for fungi [32, 33]. Section of complexes (**7**, **8**, **13**, **15**, **16**) had strong effectiveness against bacteria and fungi.

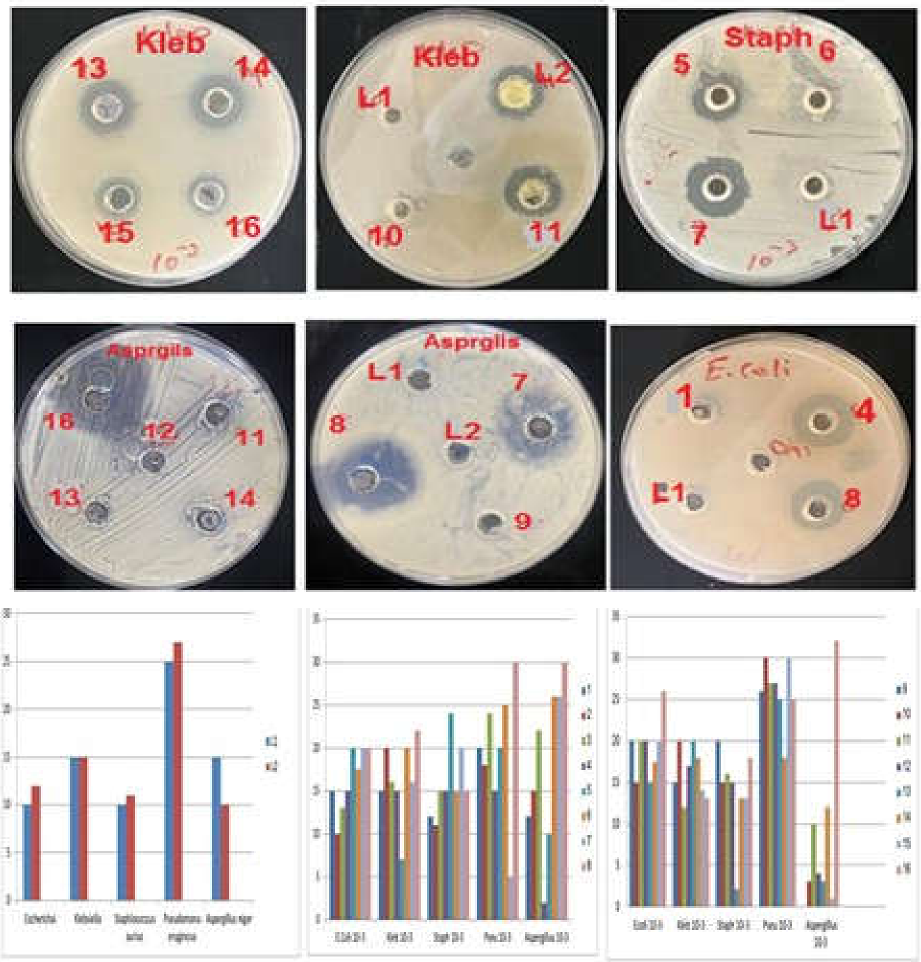


Figure 4. Effect of different concentration of materials on the growth of bacteria and fungi.

CONCLUSION

The ligands were prepared of new β-enamines derived from dimedone and amines and their complexes with transition metals Fe(II), Co(II), Ni(II), and Cu(II), in addition to two ligands (Met) and (8-hq) have been synthesized and characterized by physical-chemical methods and techniques, In molar conductance all the complexes being conductive in a ratio of (1:2). All complexes were octahedral in shape the complexes have six coordination numbers, according to spectroscopic and molar conductivity studies. The SEM shows nanoparticales for some complexs, furthermore, the ligands and complexes had varying effectiveness against certain types of bacteria and fungi.

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