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Synthesis and Antimicrobial Activity of Zinc (II) Complexes of Schiff Bases Derived from 2-Aminobenzoic Acid

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Abstract

Research in Schiff base complexes has been among the mostintriguing areas in coordination chemistry. Mononuclear zinc Schiffbase complexes of the type [Zn(L1) (H2O)2] and [Zn(HL2)2] anddinuclear complexes of the type [Zn2(L1) Q(OAc) (H2O)] and [Zn2(L2) Q(OAc) (H2O)] (where H2L1= 2-2((2hydroxybenzylidene) amino) benzoic acid, H2L2 = 2 -((4-hydroxy-3methoxybenzylidene) amino) benzoic acid and HQ= 8hydroxyquinoline) are described. The complexes were investigatedbyusing various spectroscopic methods including elemental analysis, mass spectra, 1H-NMR spectra, Fourier transform infrared (FT-IR) and UV−visible spectra. Using the diffusion method, the Schiff bases and Schiff base zinc complexes were screened in vitro against four bacteria (S. aureus, K. pneumonia, and Acinetobacter spp. and E. coli).

Keywords: 2-aminobenzoic acid; Zinc (II) complexes; 8-hydroxyquinoline Ligand, Spectrophotometry; Antibacterial

INTRODUCTION

Schiff base metal complexes have been playing an important role in coordination chemistry development due to the simple and direct synthesis of quite different ligands, reactivity, versatility and richness of the chemistry, stability of the complexes and their fascinating potential applications (Abu-Dief & Mohamed, 2015; Gupta & Sutar, 2008). Schiff base complexes play vital roles in modern coordination chemistry in addition to the improvement of bioinorganic chemistry (Deghadi *et al.*, 2022), catalysis (Dalia *et al.*, 2018; Cozzi, 2004) and magnetism (Rani *et al.*, 2018). Schiff base type ligands in coordination chemistry are flexible ligands that can confer better coordination tendency to a metal center in different coordination modes. They commonly coordinate to metal centers through azomethine nitrogen with other donor atoms from carbonyl moiety yielding mono or polynuclear complexes. However, the chelation capacity of the Schiff base ligand can be increased by introducing additional donor atoms ((Rani *et al.*, 2018; Abdel-Rahman *et al.*, 2019)). Among Schiff bases ligands, N, O type ligands have attracted considerable attention because of the stability that they give to their complexes by chelation (El-t Ashoor & Shawish, 2015; Lazzarini *et al.*, 2016).

Schiff bases that are derived from 2-aminobenzoic acid have gained significant attention.These ligands coordinate with the metal ions as anionic NO and NOX (whereX = O, N, or S) chelating ligands. In the NO mode, the coordination occursthrough the imine nitrogen and oxygen atomsof the carboxylic group to form mononuclear complexes with the ligand binding to the metal center forming one six-membered ring (Abdel-Rahman *et al*., 2019; Jirjees *et al*., 2021). Whereas, the NOX donor ligands coordinate to the metal center through the imine nitrogen, oxygen of the carboxylic group and the X atom attached to the aldehyde/ketone skeletal forming mononuclear or dinuclear complexes (Wu *et al*., 2020).

Interest has centered on zinc(II) complexes with N, O bidentatedonor ligands due to the ability of zinc to bind tohard donors. Generally, Zinc in biological systems is mainly coordinated with nitrogen and oxygen of amino acid residues (Pellei *et al*., 2021). Moreover, certain zinc complexes with N, O type ligands exhibit considerable biological activities. Dasgupta *et al*., It has been reported that Zn (II) complexes with acyl hydrazone Schiff base ligands exhibit anti-cancer activity towards (human colorectal carcinoma) HCT116, (human hepatocellular carcinoma) HepG2, and (human non-small lung carcinoma) cell lines A549. (Dasgupta *et al*., 2020).

In this study, the aim was to present the synthesis and structural characterization of zinc (II) complexes with Schiff base derivatives of 2-aminobenzoic acid.The Schiff bases (H_2L^1 and H_2L^2) react with zinc(II) acetate dihydrate to afforda mononuclear zinc complex. However, the reaction of these Schiff bases with $Zn(OAc)_2.2H_2O$ in the presence of 8-hydroxyquinoline afforded binuclear complexes and the antibacterial activity of the complexes has been evaluated.

MATERIALS AND METHODS

Reagents and solvents

All reagents and solvents were obtained from commercial sources and used without further purification. 2-aminobenzoic acid, salicylaldehyde and 3-methoxy 4-hydroxybenzaldehyde have been acquired from Alfa Aesar, 8-hydroxyquinoline was supplied from Riedel-De Haen, zinc (II) acetate dihydrate was supplied from Merck. The ligands H_2L^1 and H_2L^2have beensynthesized according to thepreviously published procedure (El-Ajaily *et al*., 2016)

Physical measurements

The equipment used for elemental analysis was the Perkin Elmer Model 2400. PerkinElmer FT-IR Spectrometer (Frontier) where used for infrared spectra. The UV-Vis spectra for the metal complexes in the DMSO solution were recorded in the 200–800 nm range on an Agilent Technologies Cary 60 UV-Vis spectrophotometer. Mass spectra (EI) were recorded using a micromassautospec spectrometer. JEOL JNM-ECA Series FT NMR was used to document 1H-NMR spectra.

Synthesis of the complexes

Synthesis of the complexes $[ZnL^1(H_2O)_2]$ (C1) and $[Zn(HL^2)_2]$ (C2)

A solution of $Zn(CH_3COO)_2$. H_2O (0.5 mmol, 0.1097 g) in 20 mL of methanol was added to a solution of the ligand H_2L^1 (1 mmol, 0.241 g) or H_2L_2 (1 mmol, 0.271 g) dissolved in 20 mL of methanol. The mixture was heated to reflux for 4 hours. The resulting precipitates were filtered then washed with water, methanol and then dried at room temperature.

Synthesis of the complexes$[ZnL^1Q(OAc)(H_2O)]$ (C3) and $[Zn(L^2)Q(OAc)(H_2O)]$ (C4)

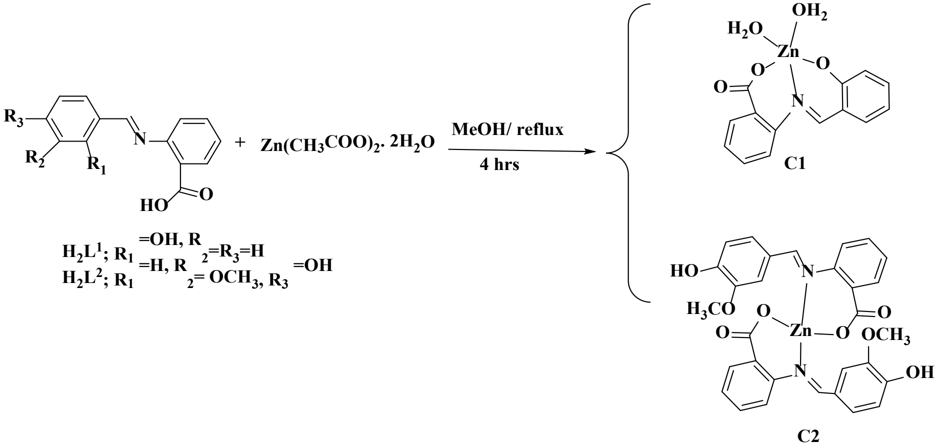
In a 5 mL methanol solution, 1 mmol (1.45g) of 8-hydroxyquinoline was mixed with either 1 mmol (0.241g) of Schiff base ligand H_2L^1 or 1 mmol (0.271g) of H_2L^2Schiff base ligand, dissolved in 20 mL methanol. Next, a solution of $Zn(CH_3COO)_2$. H_2O (1mmol, 0.219g) in 20 mL methanol was added. The mixture was heated and stirred for 5 hours. The resulting suspension was filtered washed with methanol then dried in air.

Antibacterial study

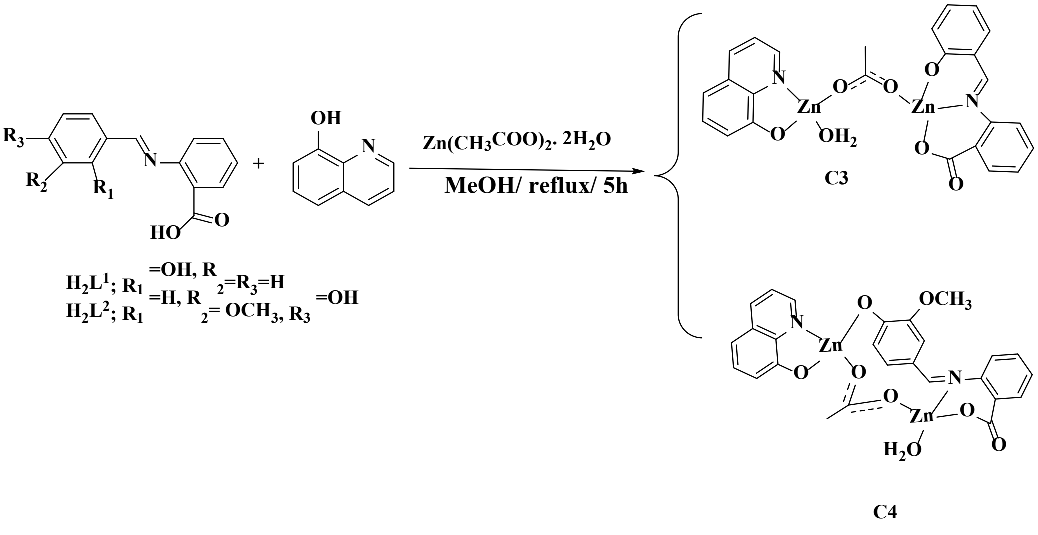
The Schiff bases H_2L^1 and H_2L^2, along with zinc (II) complexes, were tested against bacterial strains: Acinetobacter spp., Staphylococcus aureus, Streptococcus, Klebsiella pneumonia, and Escherichia coli by using the agar-well diffusion method (Bauer, 1996). The antibacterial efficiencywas tested using the DMSO as solvent. The tested compounds were added to agar plates, which were then incubated at 37 °C for 24 hours. The inhibition zones were measured in millimeters, and the experiment was repeated twice to ensure accuracy.

RESULTS

The mononuclear zinc (II) complexes C1 and C2 are prepared from a typical synthetic procedure, in which $Zn(OAc)_2.2H_2O$ is reacted with Schiff bases H_2L^1 and H_2L^2 in methanol aqueous (Scheme 1), while the dinuclear mixed ligand complexes C3 and C4 are prepared with the Schiff bases in the presence of 8-hydroxyquinoline as a co-ligand (Scheme 2). The prepared complexes are air stable, soluble in DMSO, DMFand almost insoluble in water and other common organic solvents as well. The physical properties and analytical data of the prepared complexes are summarized in Table 1. The elemental analysis results of the prepared complexes are in good agreement with the results required by the proposed formula.



Scheme (1). Schematic routes of the synthesis of complexes C1 and C2



Scheme (2). Schematic routes of the synthesis of complexes C3 and C4

Table (1). Analytical data of the ligand–metal complexes

Compound	Molecular Formula	Colour	Melting Point °C	Yield (%)	Anal.Calc. (Found)%		
					N	H	C
[ZnL^1(H_2O)$_2$](C1)	$C_{14}H_{14}NO_5Zn$	Pale yellow	280	68	4.11 (4.01)	4.40 (4.02)	49.36 (49.10)
[Zn(HL^2)$_2$](C2)	$C_{30}H_{40}N_2O_8Zn$	Pale yellow	285	72	4.62 (4.60)	3.99 (4.06)	59.47 (59.44)
[ZnL^{1}Q(OAc)(H_2O)](C3)	$C_{25}H_{20}N_2O_7Zn_2$	Yellow	Up to 300	44	4.74 (4.78)	3.41 (3.80)	50.79 (51.02)
[ZnL^{2}Q(OAc)(H_2O)](C4)	$C_{26}H_{22}N_2O_8Zn_2$	Pale yellow	Up to 300	48	4.51 (4.35)	3.57 (3.43)	50.27 (50.12)

IR spectra of the prepared complexes were compared with those of their free Schiff bases H_2L^1 and H_2L^2(Table 2). The bands at 1688 and 1690 cm^{-1} in the spectra of free H_2L^1 and H_2L^2 ligands can be attributed to the C=O stretching of the carboxylic group. However, these bands disappeared in the spectra of the zinc complexes and new bands at around 1540 cm^{-1} and 1370-1407 cm^{-1} which attributed to $v_{as}(COO^-)$ and $v_s(COO^-)$, respectively (Refat *et al.*, 2013; Dubey & Mishra, 2011). Also, the IR spectra of the free ligands presented distinguishing bands at 1615 and 1593 cm^{-1} for the CH=N, whichwere moved downfield to 1593 and 1589 cm^{-1} upon complexation of ligands H_2L^1 and H_2L^2, respectively,hence elucidating the coordination of N atom ofthe CH=N group to the central zinc (II) ion (Aldawood, 2013) . coordination of the phenolic oxygen to zinc (II) ion in C1, C3, and C4 complexes is confirmed by the shift of the strong band at about 1244 cm^{-1} in the H_2L^1 and at 1279 cm^{-1} in H_2L^2, which is ascribed to C-O stretching vibrations, to lower frequency at 1232 cm^{-1} (Uba, 2023).

Table (2). IR spectra of the ligands and theirzinc complexes

compound	Stretching frequency (cm^{-1})							
	$\upsilon_{C=O}$	$\upsilon_{C=N}$	$\upsilon_{asyCOO-}$	υ_{syCOO-}	υ_{C-O}	$\upsilon_{C=N}$ Quinoline	υ_{C-O} Quinoline	υ_{asyCOO^-} acetate	υ_{syCOO^-} acetate
H_2L^1	1688	1615	-	-	1244	-	-	-	-
H_2L^2	1690	1589	-	-	1279	-	-	-	-
C1	-	1593	1542	1406	1232	-	-	-	-
C2	-	1591	1540	1407	1272	-	-	-	-
C3	-	1593	1541	1372	1231	1467	1109	1499	1390
C4	-	1593	1543	1387	1235	1467	1110	1499	1387

This band did not shift in the IR spectrum of complex C2, which appeared at 1275 cm^{-1}. This confirms that the phenolic OH group is not coordinating with Zn. (II) (Fugu *et al.*, 2013) Moreover, the IR spectra of the mixed complexes C3 and C4 displayed new bands at 1109 and 1467 cm^{-1} assignable to v(C-O) and v(C=N), respectively in the co-ligand 8-hydroxyquinoline. (Shivankar *et al.*, 2003; Bufarwa & Abdel-Latif, 2022) Another new band appeared only in the IR spectra of C3 and C4 at 1499 and 1390 cm^{-1}assigned for $v_{as}(COO^-)$ and $v_s(COO^-)$ in the acetate group, indicating the involvement of acetate as a bridging ligand (Nakamoto, 2009). When used in conjunction with other spectroscopic information, ^{1}H NMR is a valuable tool for identifying coordination sites in zinc (II) complexes. The chemical shifts of the Schiff base ligands and their zinc complexes

$[ZnL^1(H_2O)_2]$ (C1), $[Zn (HL^2)_2]$ (C_2), $[ZnL^1Q(OAc)(H_2O)]$ (C_3) and $[Zn(L^2)Q(OAc)(H_2O)]$ (C_4)are presented in Table 3. Figure 1 shows the ^{1}H NMR spectra for the ligands and their prepared zinc complexes. The singlet band appeared at 10.25 and 9.77 ppm for the free Schiff bases H_2L^1 and H_2L^2 respectively, and is assigned to the phenolic proton.

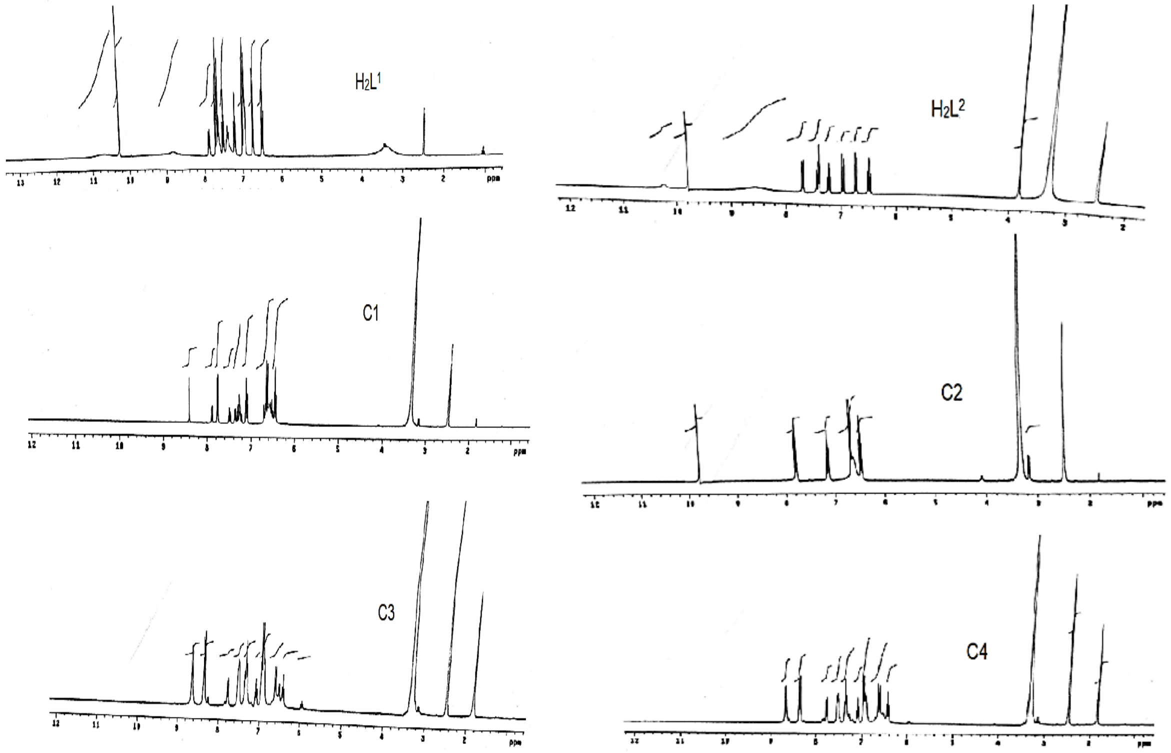


Figure (1). ^{1}H NMR spectra for the ligands and their prepared zinc complexes

Table (3). ^{1}H NMR spectral data (δ, ppm) of H_2L^1, H_2L^2, and their Zn (II) complexes

compound	Chemical shifts, δ (ppm)					
	COOH	OH	C=N	Aromatic	CH_3 (acetate)	OCH_3
H_2L^1	10.90	10.25	8.86	7.71- 6.49	-	-
H_2L^2	10.30	9.77	8.84	6.49–7.69	-	3.84
C1	-	-	8.47	7.90- 6.42	-	-
C2	-	9.85	7.89	6.44 –7.77	-	3.18
C3	-	-	8.64	6.43-7.19	1.85	-
C4	-	-	8.66	6.41-7.78	1.85	

This signal is absent from the spectra of complexes C1, C3, and C4, showing that coordination of the ligands H_2L_1 and H_2L_2 to Zn (II) was through the oxygen atom of these ligands with deprotonation (Nishal *et al.*, 2014). Also, the singlet at 10.90 and 10.30 ppm in the free ligands H_2L^1 and H_2L^2 respectively, which is attributed to the proton of the carboxylic group, disappeared in the ^{1}H NMR spectra of all complexes. The coordination of the carboxylic oxygen with the zinc center is likely confirmed. In all complexes, the azomethine proton signal in the ^{1}H NMR spectra is shifted upfield compared to the free ligands, indicating coordination through the azomethine nitrogen. Additionally, the singlet at 1.85 ppm corresponds to the CH_3 of the acetate moiety in the C3 and C4 complexes, integrating as three hydrogens (Abu-Dief *et al*., 2019).

Electronic spectra of the zinc(II) complexes and the free Schiff bases were recorded in DMSO (1×10^{-5} M) at room temperature.The study results are depicted in Figure 2 and Table 4.The Schiff base H_2L^1 exhibits two absorption bands at 261 and 315 nm, respectively attributed to ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$) transitions of the azomethine and carboxylic chromophore.Similarly, the ligand H_2L^2 exhibits two bands that appeared at 280 and 313 nm which are attributed to the ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$), respectively. On complexation, this band was shifted to a higher wavelength region, proposing the coordination of azomethine nitrogen and carboxylic oxygen with Zn (II) ion (Belal *et al*., 2015). Furthermore, complexes C3 and C4 exhibit a broadband at 398 nm, which is assigned to metal-ligand charge transfer (MLCT) transitions. This is expected for zinc complexes (Abdel-Rahman *et al*., 2017). The mass spectra of the complexes showed different patterns of fragmentation, as expected. The results were consistent with the molecular formulas of the compounds. Figure 3 displays the mass spectra of the C1, C2, C3, and C4 complexes. These spectra showed molecular ion peaks at m/z = 341, 607, 593, and 622, respectively. These peaks matched the calculated weights of 340.64, 592.21, 605.91, and 621.23.

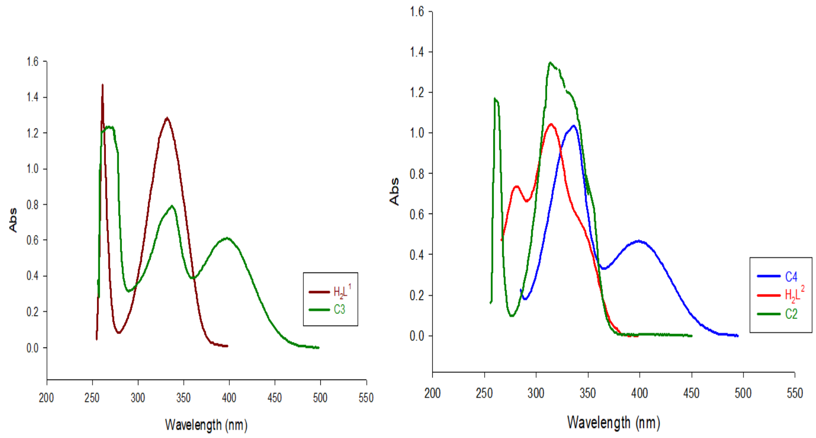


Figure (2). Electronic spectra of the ligands andtheir zinc (II) complexes

Table (4). Electronic spectra data of the ligands and their zinc (II) complexes

Compound	Absorbance (nm)	Assignment
H_2L^1	259	$\pi \rightarrow \pi^*$
	331	$n \rightarrow \pi^*$
H_2L^2	260, 281	$\pi \rightarrow \pi^*$
	315	$n \rightarrow \pi^*$
C1	266	$\pi \rightarrow \pi^*$
	338	$n \rightarrow \pi^*$
	396	M→ L
C2	272	$\pi \rightarrow \pi^*$
	336	$n \rightarrow \pi^*$
C3	268	$\pi \rightarrow \pi^*$
	336	$n \rightarrow \pi^*$
	403	M→ L
C4	336	$n \rightarrow \pi^*$
	405	M→ L

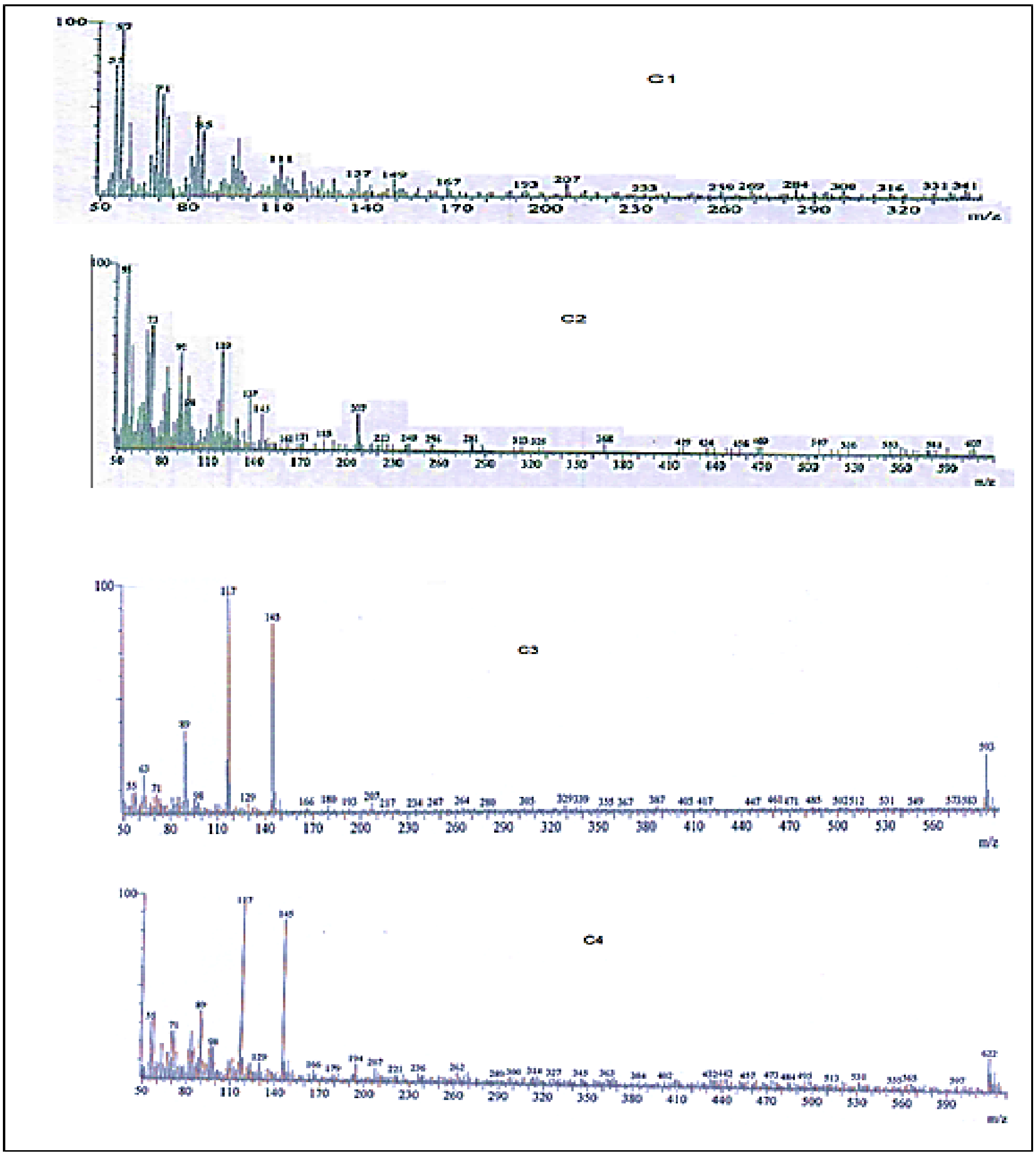


Figure (3). Mass spectra of zinc complexes

The antibacterial potential of the ligands and their zinc (II) complexes is investigated. For antibacterial testing, Gram-negative strains (Escherichia coli, Streptococcus Klebsiella pneumonia, and Staphylococcus aureus) and Gram-positive strains (Acinetobacter Spp.) were used. Table 5 summarizes the results of the antibacterial activity. The ligands had low activity against each bacterial species, however, the zinc complexes performed better than the original ligands. All prepared zinc complexes showed notable antibacterial activity against the *Streptococcusaureus* bacterial strain with an inhibition zone range of 14-20. However, the zinc complex $[ZnL^1(H_2O)_2]$ (C1) With an inhibition zone diameter of 20mm, showed demonstrated activity against the Staphylococcus aureus strain. This result can be comparable with results reported in the literature (Joseyphus & Nair, 2008; Zabin *et al*., 2018).

Table (5). shows the antibacterial activity of ligands and their zinc complexes.

Compound	Bacterial Zone of Inhibition (mm)			
	Gram Positive		Gram Negative	
	Staphylococcus aureus	*Escherichia coli*	*Acinetobacter Spp.*	*Klebsiella pneumonia*
DMSO(control)	6	6	6	6
H_2L^1	6	11	7	7
H_2L^2	6	11	10	7
C1	20	17	19	15
C2	14	6	11	10
C3	16	14	14	10
C4	19	14	19	14

CONCLUSION

Schiff bases derived from 2-aminobenzoic acid were synthesized and characterized as zinc (II) complexes. Schiff base ligands coordinate with the Zn (II) ion in various ways, including as a dibasic tridentate ONO chelating ligand and as a negative ON bidentate ligand. According to the antibacterial study, Zinc (II) complexes have better antibacterial properties than free Schiff base ligands.

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