

SYNTHESIS AND CHARACTERIZATION OF NOVEL CADMIUM COMPLEXES FROM ORGANIC LIGANDS

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ABSTRACT : Cd (II) complexes derived from azomethine ligands were synthesized, obtained by condensing different aldehydes with the primary amine. The complexes were described by the analysis result (C.H.N), molar conduction, magnetic susceptibility, FTIR, UV-Visible, ¹H-NMR and GC. Mass spectra. The complexes were found to be non-conductive and some relatively conductive depending on the molar conduction value. From spectral data, octahedral geometry was suggested for all complexes. Furthermore, metal complexes were examined for their antifungal activity.

Key words : Azomethine, molar conductivity, cadmium complexes.

INTRODUCTION

Azomethine compounds and their mineral complexes have received much attention and include a wide area of organic compounds and various parts of biochemistry (Sumrra *et al*, 2014). In 1864, synthesis of azomethine compounds was first introduced by the German scientist Höckel Schiff (Qin *et al*, 2013) derived from the condensation of aldehydes (aromatic or aliphatic) and amines, carbonyl group is exchanged by the azomethine group (HC=N), which occurs in the presence of an acid medium as a catalyst, or upon heating (Abu-Diefa and Mohamed, 2015). The azomethine group has a double bond of carbon nitrogen as a functional group, in which the nitrogen atom is coupled to an aryl group or an alkyl group, but not hydrogen (Abid *et al*, 2017; Muslim and Eaid, 2019).

Azomethine compounds implicate aryl groups are more stable, while compounds with alkyl groups are comparatively unstable and readily disintegrate (Dalia *et al*, 2018). They are characterized by aromatic aldehydes are more stable and effective than aliphatic aldehydes (Neelakantana *et al*, 2008).

Azomethine compounds form stabilized complexes with various transition metal ions are capable of proving many metals in various oxidation states (Chaudhary and Singh, 2017). Azomethine compounds are more stable and include NO or N₂O₂ donor atoms, but the oxygen atoms can be exchanged by sulphur, nitrogen (Thombal *et al*, 2017). The chelating capacity for metal complexes has

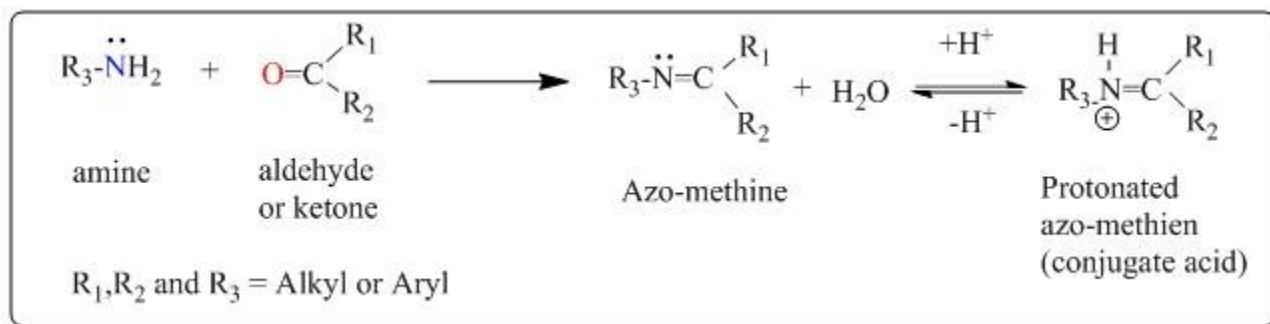
played a large part in biological applications that act as a pattern of interactions biologically (Abdel-Rahman *et al*, 2016; Gonul *et al*, 2016). A number of azomethine compounds contain a group (HC = N) a large area of studies biological efficacy and include antibacterials, antifungals, antidiabetic, anti-cancer, anti-inflammatory and anti-corrosion (Gomathi and Selvameena, 2013; Yusof *et al*, 2015).

Biological activity is thought to be linked to the hydrogen bonding of the group (HC = N) with the effective centers of cell components (Gomathi and Selvameena, 2013; Dalia *et al*, 2018; Abdel-Rahman *et al*, 2016). The fusion of transition metals with azomethine compounds enhances biological activity and reduces the cellular toxicity of both metals and ligands (Panneerselvam *et al*, 2005).

MATERIALS AND METHODS

Chemicals : Chemicals, transition elements salts and solvents were used according to their producing companies. All materials were of high purity. 4-nitrobenzaldehyde (Aldrich), 4-chlorobenzaldehyde (C.D.H), isatin (C.D.H), 1H-1,2,4-triazole-3,5-diamine (Aldrich), 5-(3,4,5-trimethoxybenzyl) pyrimidine 2,4-diamine (Aldrich), Glacial acetic acid, Cd(CH₃COO)₂·2H₂O (B.D.H), methanol, ethanol (Scharlur), chloroform, diethyl ether and dimethyl sulphoxide (C.D.H).

Instruments : Proton Nuclear Magnetic Resonance



Scheme 1 : Scheme for formation of Azomethine.

Table 1 : Physical property and analyzed for Azomethine compounds and its Metal complexes.

Comp code	Molecular formula	M.wt	M.P. (°C)	Colour	Yield (%)	C%	H%	N%	M%
L ₁	C ₁₆ H ₁₁ Cl ₂ N ₅	343.20	264-266	Yellowish white	70%				--
L ₂	C ₂₈ H ₂₄ N ₆ O ₇	556.54	175-177	Pale yellow	73%	60.37 (60.41)	4.35 (4.31)	15.09 (15.10)	--
L ₃	C ₁₆ H ₁₁ N ₇ O ₄	365.31	263-265	Yellow	71%	52.56 (52.59)	3.01 (3.04)	26.81 (26.85)	--
L ₄	C ₁₈ H ₁₁ N ₇ O ₂	357.33	200-202	Bright reddish	68%				--
C ₁	C ₃₈ H ₃₇ BrCdCl ₂ N ₈ O ₆	964	271 dec	Pale Green	78%	47.30 (47.80)	3.73 (3.86)	11.61 (11.30)	11.65 (12.19)
C ₂	C ₄₆ H ₄₄ BrCdN ₉ O ₁₀	1193	219-221	Grayish	67%				9.43 (9.72)
C ₃	C ₃₂ H ₂₆ CdN ₁₄ O ₁₀	998	268 dec	Mustard	85%	43.72 (42.07)	2.98 (3.15)	22.39 (121.2)	11.27 (11.49)
C ₄	C ₁₈ H ₁₅ CdN ₇ O ₄	589	205-207	Orange	55%				19.18 (18.79)

dec = Decomposition

Spectrum (¹H - NMR) using DMSO-d₆, CDCl₃ and TMS solvents was recorded as an internal plane at 300 MHz, Infrared spectra were obtained using FTIR spectrometer in the range (4000-400) cm⁻¹ Shimadzu (KBr Disc). The visible UV spectra were recorded and GC-mass spectra. Melting points were gained on the electric thermometer.

Synthesis of azomethine ligands (L₁-L₄) and metal complexes (C₁, C₂, C₃, C₄): 1H-1,2,3-Triazole 2,5 diamine (1 mmol, 0.4 g) dissolve in 15 ml pure ethanol was slowly added to a mimetic solution of various aldehydes (2 mmol), in the presence of a 3 drops of glacialacetic acid. mixture was returned for (4hr). The solution was then concentrated on half its amount and cooling by ice. start product at deposition. The product was filtered and recrystallized at ethanol (Al-Janabi and Al-Dulaimi, 2016; Muslim *et al*, 2019).

Various complexes were synthesized by dissolve 1 mmol of Cd

(acetate)₂.2H₂O in 10 ml of ethanol, to a ligand solution (2 mmol in 15 ml of ethanol) was re-mixed at (6hr), the concentrated solution was filtered and washed in distilled water and ethanol (Rosua *et al*, 2011; Adly *et al*, 2017).

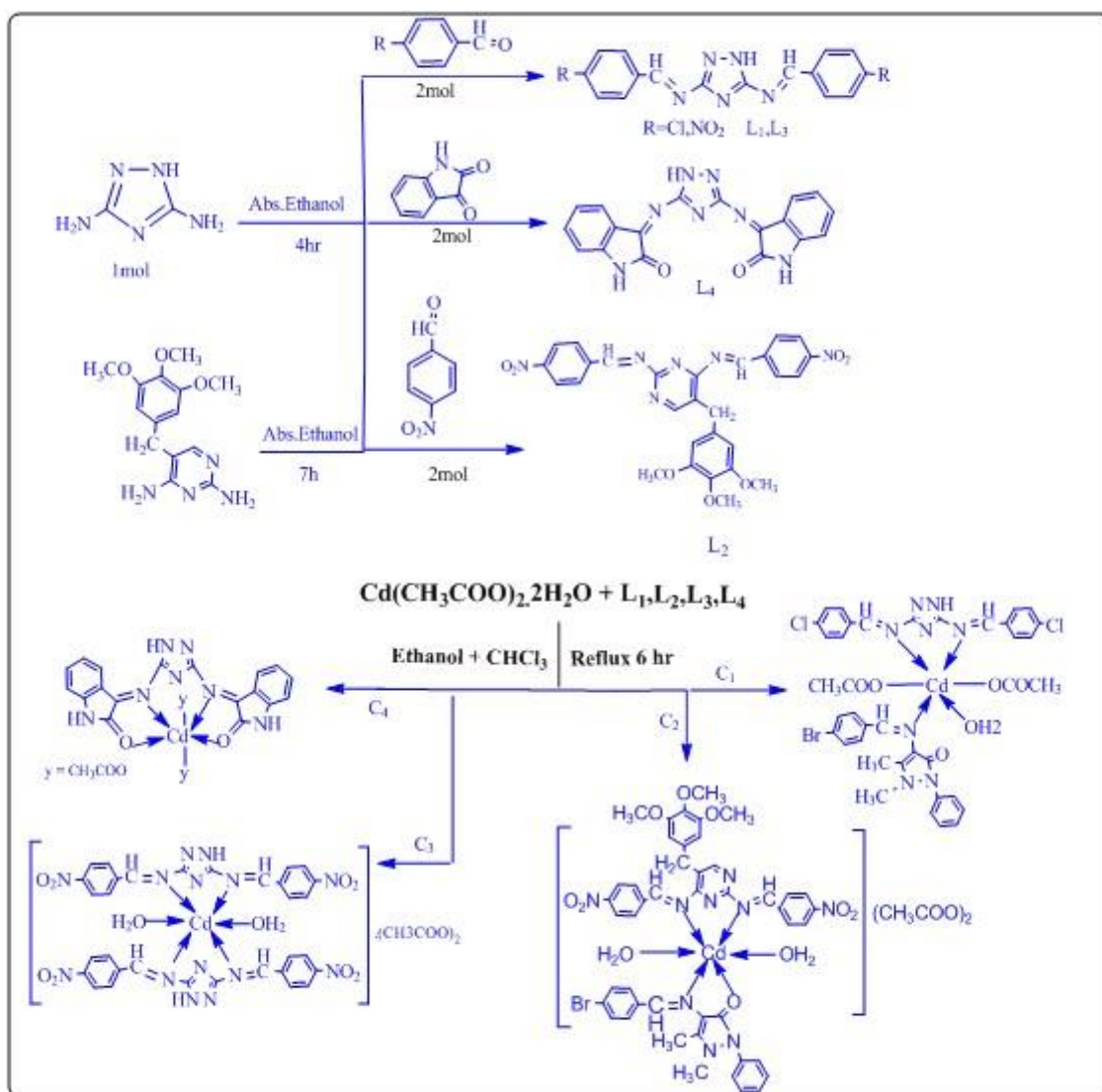
Antifungal activity : The antifungal activities of Complexes synthesized were examined *in vitro* for growth inhibitory against *C. albicans* and *A. niger* by agar well spread method at several concentrations compared with Anti fungal (Fluconazole, Itraconazole) and DMSO as (control) (Patil *et al*, 2017).

RESULTS AND DISCUSSION

Characterization azomethine compounds and Cadmium complexes : Azomethine compounds were prepared by interaction between aromatic aldehydes and primary amines used in ethanol. According to the condensation method, the (CdL) metal complexes were prepared by a direct reaction between azomithene compounds and cadmium acetate salt in the same solvent in a ratio of [1:1,1:2]. The complexes prepared in coordination between the nitrogen atom in group (HC = N) and oxygen atom for Isatine with cadmium. All compounds are soluble in some common solvents such as DMSO, DMF, Chloroform and Ethanol. Analytical data for the compounds are presented in Table 1. The prepared compounds were diagnosed with

spectroscopic measurements were identical to the proposed formulas for the complexes.

Infrared Spectrum : The infrared spectra of the cadmium complexes were charactrazed and compared with the results of azomethene compounds and the finding of difference between ligands and complexes at indicating the occurrence of complexity, the ligands spectra showed strong absorption bands at the range $1612-1662\text{ cm}^{-1}$, due to the formation of the azomethine group (C = N) (Muslim *et al*, 2018), while the group (C = N) of the triazole of the ring appeared at lower frequencies, the triazole group (NH) at $3278-3406\text{ cm}^{-1}$ (Silverstein *et al*, 2005) during the complexity process, the location of the absorption bands of the azomethine group (C=N) in ligands is



Scheme 2 : Synthesized azomethine Compounds and Cadmium Complexes.

FTIR, UV-Visible, $^1\text{H NMR}$, C.H.N, GC. Mass spectrometry and visible UV radiation, the results of the

transferred to the low or high frequency as in the complexity. The displacement in the bands is an indication of the nitrogen coordination of the

Table 2 : FT-IR spectrum for azomethine ligands and Cadmium Complexes (KBr Disc).

Comp Code	(C=N)	(C=C) Ar-ring	(C-N)	(N-H)	M-O	M-N	(H ₂ O)	(COO)	Others
L ₁	1616	1489	1219	3317	--	--	--	--	C-Cl 825
L ₂	1631	1458	1195	--	--	--	--	--	NO ₂ 1539,1346 CH ₂ 2846 C-O 1234
L ₃	1662	1415	1215	3329	--	--	--	--	NO ₂ 1523,1346
L ₄	1612	1459	1200	3186	--	--	--	--	C=O 1726 N-H(amide)3448
C ₁	1589	1489	1215	3348	505	470	3236	1531Asy. 1350Sy.	C-Cl 825 C=O 1647
C ₂	1620	1458	1126	--	582	451	3371	--	C=O 1708, C-B 624 NO ₂ 1523,1342
C ₃	1666	1427	1215	3433	--	415	3317	--	NO ₂ 1523,1346
C ₄	1618	1459	1201	3187	543	478	--	1440Asy. 1330Sy.	N-H 3448 C=O 1723

Table 3 : ¹H-NMR spectra of prepared azomethine Compounds L₁-L₄.

No.	Molecular Formula	Chemical Shift (δ ppm)	Type of Single	No. Protons	Functional groups
L ₁	C ₁₆ H ₁₁ Cl ₂ N ₅	9.29	S	2	HC=N
		8.51	S	1	-NH
		7.01-8.19	m,d	8	Ar-H
L ₂	C ₂₈ H ₂₄ N ₆ O ₇	10.17	S	2	HC=N
		6.37-8.41	m,dd	10	Ar-H
		4.62-4.84	S	2	Pyr-CH ₂ -Ar
		3.84-3.62	S	3	OCH ₃
L ₃	C ₁₆ H ₁₁ N ₇ O ₄	10.17	S	2	HC=N
		9.11	S	1	-NH
		8.19-8.44	m	8	Ar-H
L ₄	C ₁₈ H ₁₁ N ₇ O ₂	8.02	S	2	-NH
		6.92-7.65	m,t	8	Ar-H

azomethane group with the cadmium ion in (complexes) (Abd-Elzaher *et al*, 2016). In addition, the location of the group (C=N) and (NH) of the triazole ring (ligand) did not change in all complexes, indicating that the nitrogen atom was not associated with the formation of the complexes (Amer *et al*, 2013) two absorption bands appeared at the range (455-482) and (509-563) cm⁻¹ attributed to the groups of M-N and M-O in the complexes, respectively. This indicates the correlation of the metal with the ligand from the side of the nitrogen atom of the group (C=N) and the oxygen atom of (C=O) (Abd-Elzaher *et al*, 2016; Kuddushi *et al*, 2018). Cd(II) complexes showed bands that are somewhat broad in the 3200-3417 cm⁻¹ region, indicating the presence of water molecules inside the composition of these complexes (Manjula and Antony, 2013; Koushik *et al*, 2007). The apparent bands are

attributed at the frequencies at 1427-1546cm⁻¹ and 13301354cm⁻¹ belong to the group of symmetrical and asymmetric acetate in the complexes (Adly *et al*, 2017;

¹H-NMR Spectra : ¹H-NMR Spectrum for azomethine ligands were listed in DMSO-d₆, CDCl₃ and shown in Figs. (9-12). The nuclear magnetic resonance spectrum (1H-NMR) of the azomethine ligands display a single singlet at 9.25 = 10.25 ppm (S, 1H) due to existence proton the group (HC=N), Signal at 8.1 8.159.15 ppm (S, 1H) due to the existence proton the group (-NH). Signal at 6.9- 8.19 ppm (S, 8H,12H) due to the presence of aromatic protons (Silverstein *et al*, 2005), signal at δ = 2.50,7.72 ppm (S) due to solvent.

Where, s = singlet, d = doublet, t = triplet, m = multiplet.

Sebastian *et al*, 2011).

recorded in Table 4.

Magnetic studies : The magnetic sensitivity of Cd (II) complexes is characterized by di-magnetic properties and is consistent with the composition d^{10} ($t_2g^6 e_g^4$) which indicates octahedral geometry and sp^3d_2 hybridization (AlAdilee and Atyha, 2018).

Electronic spectral studies : The Uv-Vis spectra of the ligands and cadmium complexes prepared in the DMSO solution were recorded in the range of 190 to 800 nm and the values are listed in Table 4.

The electronic absorption spectra of azomethine compounds showed two peaks, the first transition can be set in bond, (390-450)nm to $\pi-\pi^*$ due to aromatic rings, which have not already changed at the complexity, while

Molar conductivity : The Molar conductivity for complexes prepared at laboratory temperature was measured using DMSO (10^{-3}) as a solvent. The values

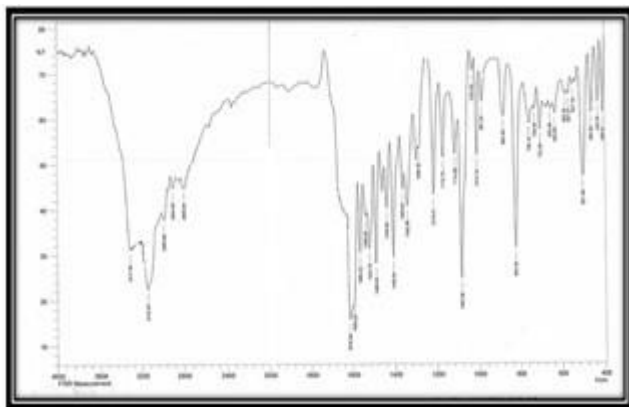


Fig. 1 : [FT-IR Spectrum for ligand L₁].

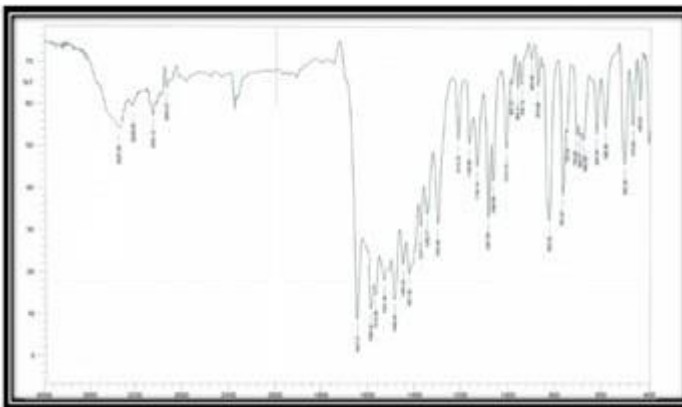


Fig. 2 : [FTIR Spectrum for Complex C₁].

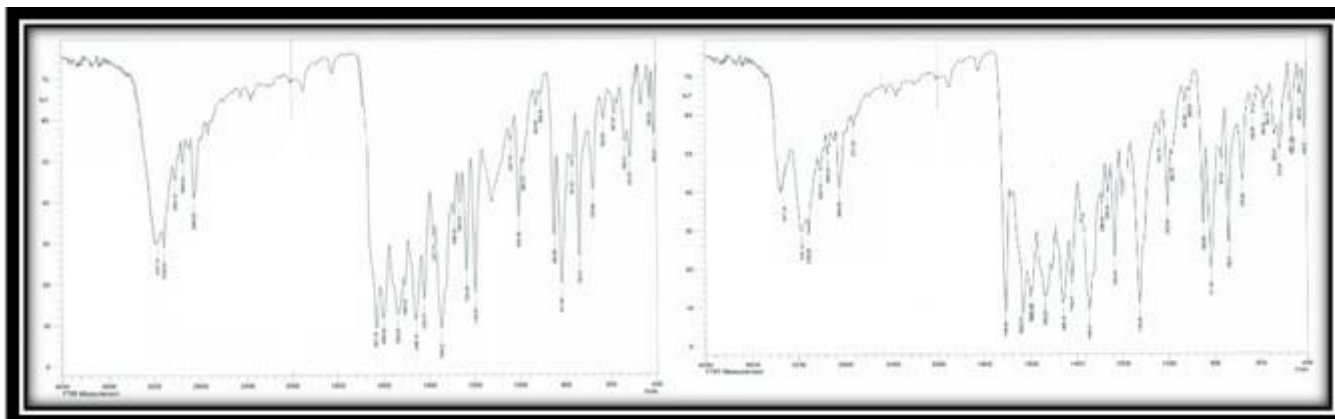


Fig. 3 : [FTIR Spectrum for ligand L₂].

Fig. 4 : [FTIR Spectrum for Complex C₂].

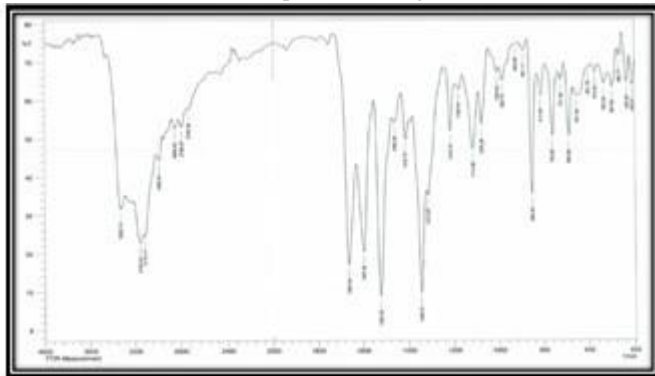


Fig. 5 : [FTIR Spectrum for ligand L₃].

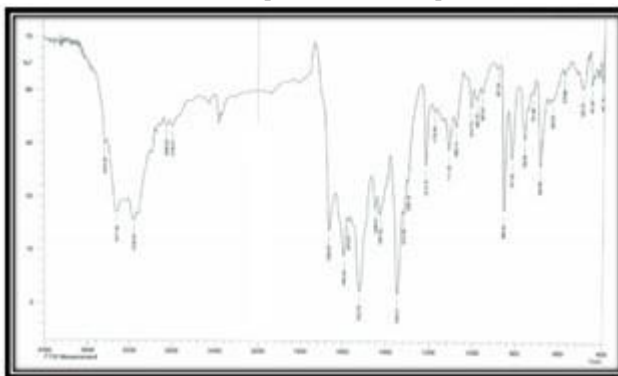


Fig. 6 : [FTIR Spectrum for Complex C₃].

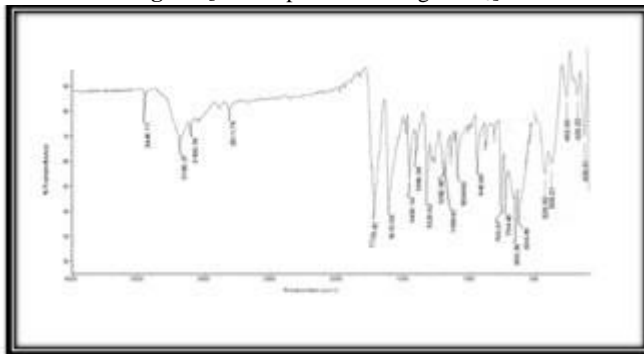


Fig. 7 : [FTIR Spectrum for ligand L₄].

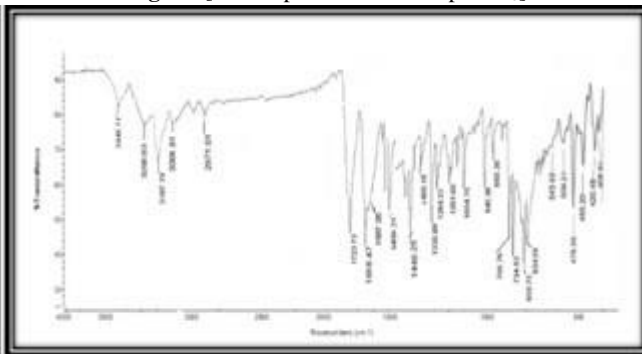


Fig. 8 : [FTIR Spectrum for Complex C₄].

Fig. 9 : [¹H-NMR Spectrum for The ligand L₁].

Fig. 10 : [¹H-NMR Spectrum for The ligand L₂].

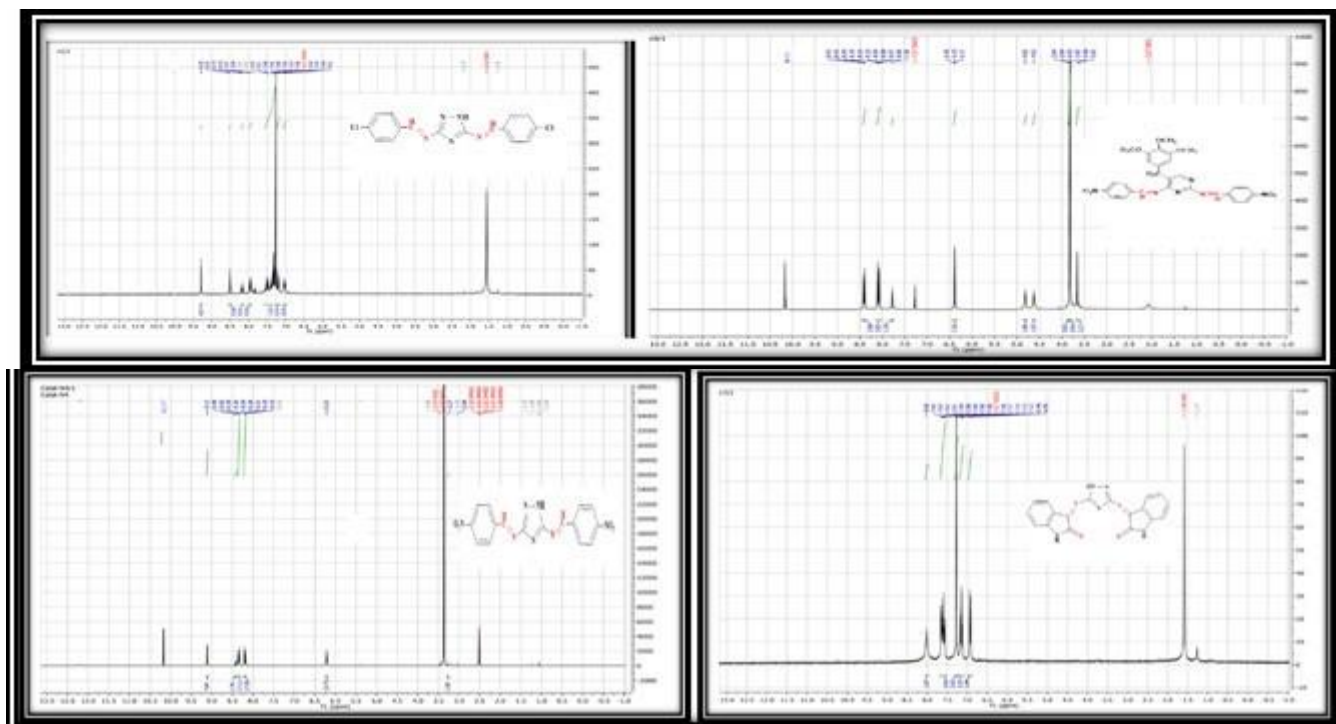


Fig. 11 : $^1\text{H-NMR}$ Spectrum for the ligand L_3 . **Fig. 12 :** $^1\text{H-NMR}$ Spectrum for The ligand L_4 . **Table 4 :** Electronic spectrum analysis

for ligand and its Metal complexes, magnetic moments, proposed structure and metal complexes.

Compounds	Wave number		ϵ_{max} $\text{L.mol}^{-1}\text{.cm}^{-1}$	Transition	Cond. Λm ($\Omega^{-1}\text{cm}^2\text{mol}^{-1}$)	μ_{eff} (B.M)	Geometry, Hybridization
	nm	cm^{-1}					
L_1	235	42553	1612	$n-\pi^*$	--	--	--
	405	24691	1061	$\pi-\pi^*$			
L_2	235	42553	1493	$\pi-\pi^*$	--	--	--
	375	26667	523	$n-\pi^*$			
L_3	235	42553	1492	$\pi-\pi^*$	--	--	--
	450	22223	1348	$n-\pi^*$			
L_4	235	42553	1492	$n-\pi^*$	--	--	--
	465	21505	1594	$\pi-\pi^*$			
C_1 $[\text{Cd}(\text{L}_1)(\text{C})(\text{X})](\text{y})_2$	235	42553	1613	Ligand Field	10.3	Dia	Octahedral SP^3d_2
	415	24096	1195	C.T			
C_2 $[\text{Cd}(\text{L}_2)(\text{C})(\text{X})_2](\text{y})_2$	235	42553	1614	C.T	72	Dia	Octahedral SP^3d_2
	393	10649	926				
C_3 $[\text{Cd}(\text{L}_3)_2(\text{H}_2\text{O})_2](\text{y})_2$	235	42553	1614	Ligand Field	71	Dia	Octahedral SP^3d_2
	461	21692	1429	C.T			
C_4 $[\text{Cd}(\text{L}_4)(\text{y})_2]$	234	42553	1546	Ligand Field	9.3	Dia	Octahedral SP^3d_2
	473	21142	1732	C.T			

$y = \text{CH}_3\text{COO}$, $X = \text{H}_2\text{O}$, $C =$ ligand my side.

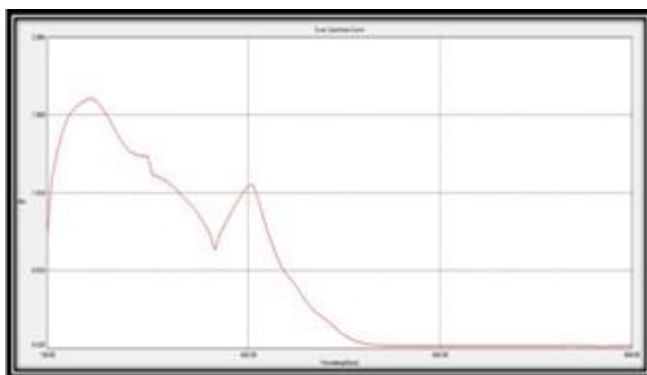


Fig. 13 : [Electronic Spectrum for ligand L₁].

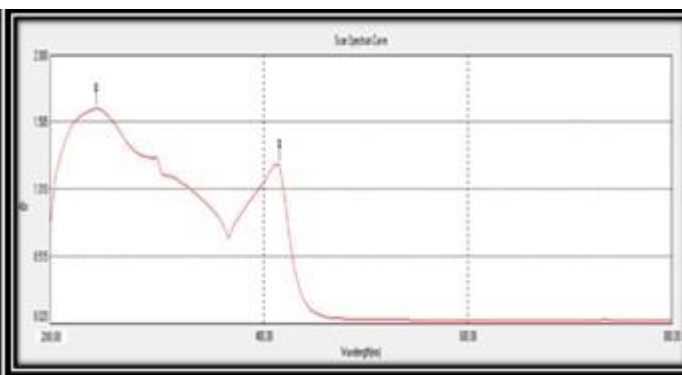


Fig. 14 : [Electronic Spectrum for Complex C₁].

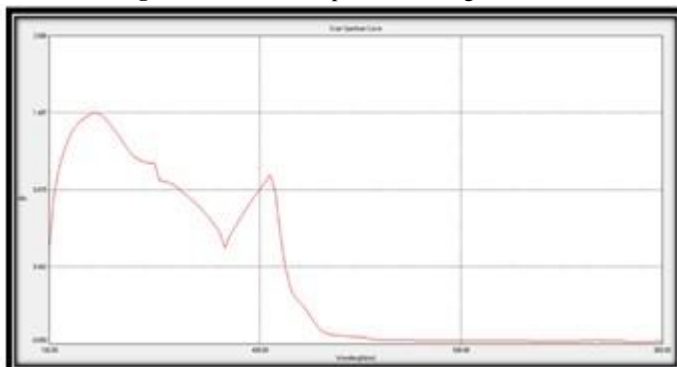


Fig. 15 : [Electronic Spectrum for ligand L₂].

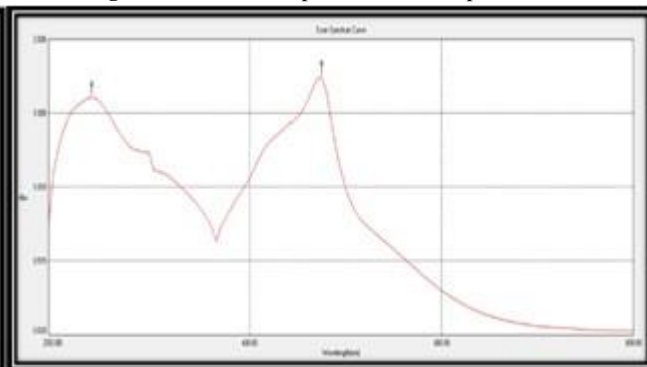


Fig. 16 : [Electronic Spectrum for Complex C₂].

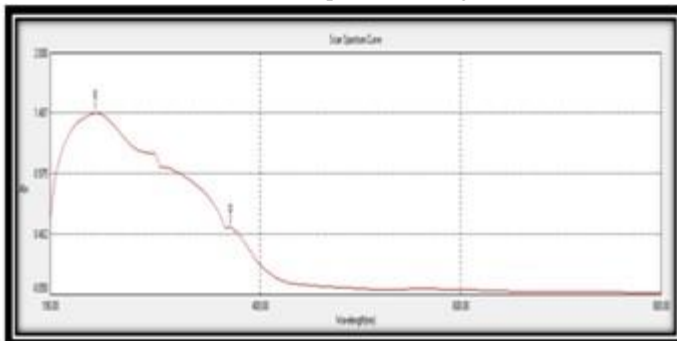


Fig. 17 : [Electronic Spectrum for ligand L₃].

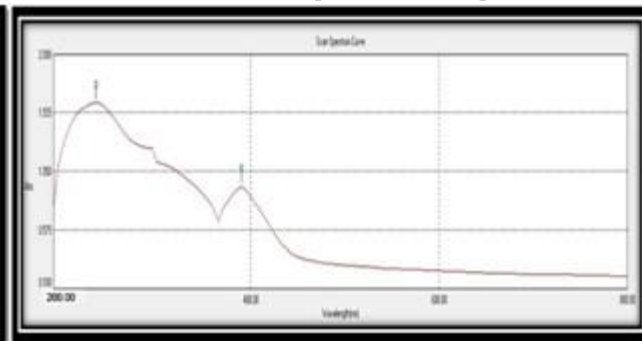


Fig. 18 : [Electronic Spectrum for Complex C₃].

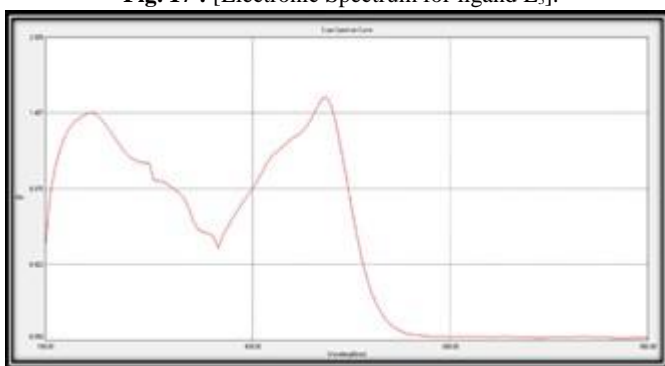


Fig. 19 : [Electronic Spectrum for ligand L₄].

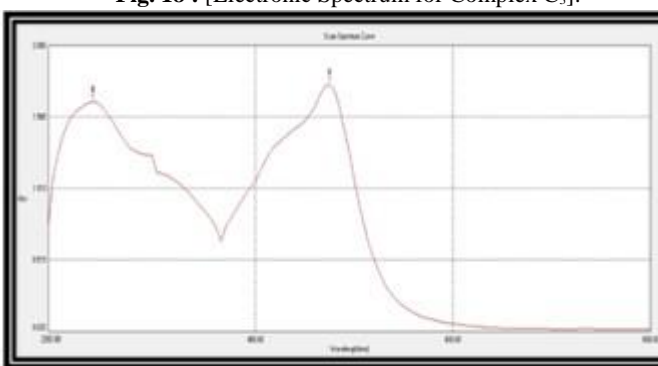


Fig. 20 : [Electronic Spectrum for Complex C₄].

the second peak determined in the range (390-450) nm to $n \rightarrow \pi^*$ for azomethine group (Rashee *et al*, 2015) that changed at the complexity of their towards shorter or longer wavelengths by sending an electron from the orbital ligand to the orbital metals called charge

transitions (Mohammed *et al*, 2015). The electronic spectrum of Cd (II)- complexes peaks in the U.V region at (235)nm for Cd(II)- complexes respectively assigned to the ligand field. While the peak other at

(401-474) nm are assigned for the charge transfer transitions, Cadmium (II)

Table 5 : GC.Mass data of the Complex C₄ [Cd(L₄)(CH₃COO)].

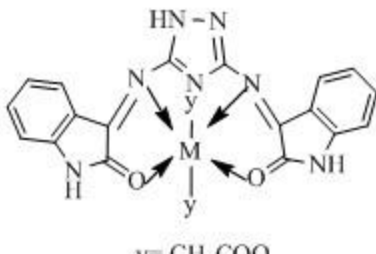
Complex	Fragmentation	Mass/charge (m/z)	Relative abundance
 <p>y = CH₃COO</p> <p>[Cd(L₄)(CH₃COO)₂]</p>	M ⁺ =[C ₂₂ H ₁₇ N ₇ O ₆ Cd] ⁺	589	55%
	[C ₂₀ H ₁₄ N ₇ O ₄ Cd] ⁺	530	20%
	[C ₁₈ H ₁₁ N ₇ O ₂ Cd] ⁺	471	28%
	[C ₁₄ H ₉ N ₇ O ₂ Cd] ⁺	420	30%
	[C ₁₁ H ₅ N ₅ O ₃ Cd] ⁺	372	25%
	[C ₁₈ H ₁₁ N ₇ O ₂] ⁺	357	31%
	[C ₁₀ H ₇ N ₅ Cd] ⁺	325	15%
	[C ₁₃ H ₉ N ₆ O ₂] ⁺	281	27%
	[C ₄ H ₄ N ₄ OCd] ⁺	236	25%
	[C ₁₆ H ₅ N ₆ O] ⁺	226	77%
	[C ₁₁ H ₅ N ₄ O] ⁺	213	24%
	[C ₁₀ H ₅ N ₅] ⁺	198	61%
	[C ₂ H ₃ O ₂ Cd] ⁺	171	24%
	[C ₅ H ₅ N ₂ O] ⁺	143	73%
	[C ₄ H ₄ N ₄ O] ⁺	124	26%
	[C ₃ H ₃ N] ⁺	119	76%
	[Cd] ⁺	112	98%
	[C ₂ H ₂ N ₅] ⁺	97	50%
	[C ₅ H ₅ N] ⁺	82	53%
	[C ₅ H ₅] ⁺	68	70%
	[C ₂ H ₃ O ₂] ⁺	59	37%
[C ₄ H ₇] ⁺	55	100%	
[C ₄ H ₅] ⁺	53	21%	

Table 6 : The inhibition (mm) after (42 h) at 30 gm/ml and 15 mg/ml concentration for the studied complexes.

No.	Complexes	<i>Candida albicans</i>		<i>Aspergillus niger</i>	
		30mg/ml	15mg/ml	30mg/ml	15mg/ml
C	DMSO (Control)	0	0	0	0
A ₁	Itraconazole	9	5	6	4
A ₂	Fluconazole	14	9	10	5
L ₁	C ₁₆ H ₁₁ Cl ₂ N ₅	0	0	0	0
L ₂	C ₂₈ H ₂₄ N ₆ O ₇	11	14	18	10
L ₃	C ₁₆ H ₁₁ N ₇ O ₄	0	0	0	0
L ₄	C ₁₈ H ₁₁ N ₇ O ₂	11	0	7	0
C ₁	[Cd(L ₁)(C)(H ₂ O)(CH ₃ COO)]	43	30	32	21
C ₂	[Cd(L ₆)(C)(X) ₂ .(y) ₂]	40	33	28	13
C ₃	[Cd(L ₃)(H ₂ O) ₂](CH ₃ COO) ₂	35	35	20	12
C ₄	[Cd(L ₄)(CH ₃ COO) ₂]	40	34	22	11

complexes do not include transformations (d-d) because **GC. Mass spectrum** : Mass spectrum for the their orbitals are completely occupied by electrons ($3d^{10}$), prepared complex display the ionic peak $[M+1]$ at m/z Based on other measurements, the proposed geometrical $z=589$ equivalent to mass molar for complex, respectively. is octahedral (Numan *et al*, 2016). Spectrum also shows peaks resulting from complex

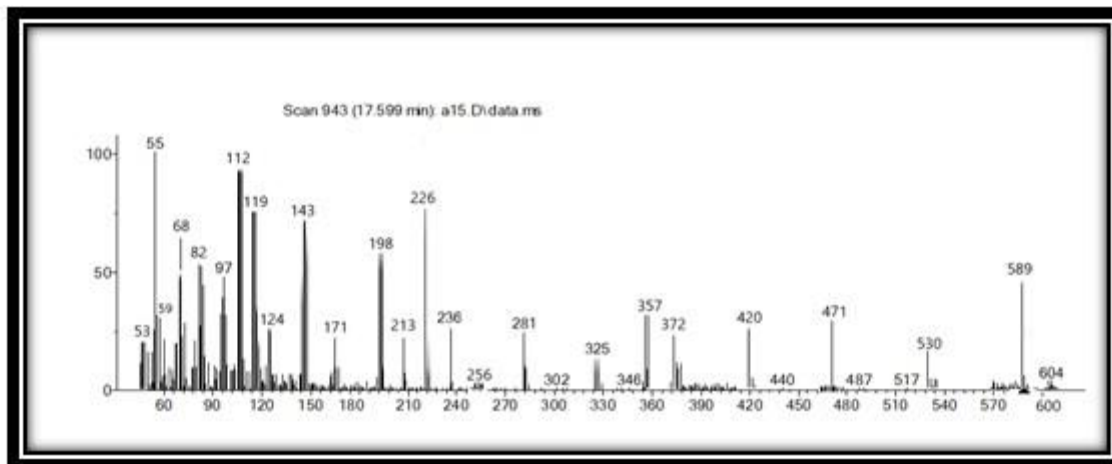


Fig. 21 : [GC-Mass Spectrum for Complex C₄].



Fig. 22 : Bioactivity of Ligands against *C. albicans* and *A. niger*.

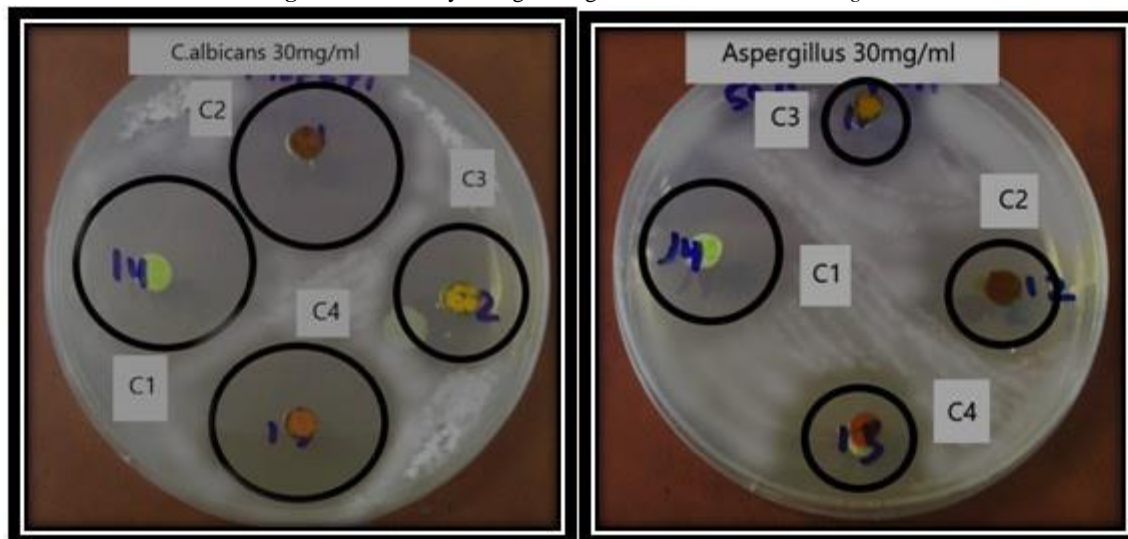


Fig. 23 : Bioactivity of complexes against *C. albicans* and *A. niger*.

fragmentation. These fragmentation are due to the ligands and their transition complex (Vogel, 1968; Silverstein *et al*, 2005).

Antifungal activity : Antifungal Potential: The biological activity was studied employing the antifungal method, Pathogenic strains were used) *C. albicans* and *A. niger* (MIC values for complexes show strong activity compared with azomethine compounds and antibiotics (Itraconazole and Fluconazole standard) against fungi *C. albicans* and *A.niger* (Tawfeeq *et al*, 2019). The values are depicted in Table 6.

Based on the results of spectroscopic diagnosis, the compound (L₄) acted as a tetrahedron ligand (N₂O₂) by nitrogen atoms from groups (C=N) and group oxygen (C=O) for esatin with a complex (C₄). While compounds (L₁, L₂, L₃) the behavior of di dentate ligands via nitrogen atoms from groups (C=N) of complexes (C₁, C₂, C₃).all these results indicate that the proposed formula is octahedral for all complexes (C₁, C₂, C₃, C₄). These complexes showed significant inhibitory activity against tested fungi compared to antibiotics.

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