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RESEARCH ARTICLE

Synthesis, Spectral and Antibacterial Activity of Mixed Ligand from Lanthanium (III) Complexes of Schiff Base Ligand with Some Amino Acids

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Abstract

In this work, lanthanium (III) complexes were synthesized using by Schiff base ligand (L) derived from benzaldehyde and o-aminoaniline with five amino acids (AA) from glycine (Gly), L-alanine (Ala), L-valine (Val), L-asparagine (Asp) and DL- phenylalanine (Phe). The Schiff base ligand has been characterized by elemental analysis, (MASS, FTIR, ^{1}HNMR, ^{13}CNMR, UV-VIS) electronic spectra. The structures of the new complexes have been described of analysis of elements, molar conductivity, (UV-Vis electronic, FT-IR, mass) spectra also magnetic moment. The molar conductivity values of the complexes indicat this every of complexes are electrolytes and other analytical studies reveal octahedral geometry for La (III) ion. The Schiff base ligand, five amino acids, La(III) salt and mixed ligand complexes were tested against four pathogenic bacteria such as G^{+}- bacterial and such as G^{-}- bacterial. These bacteria were found that compounds show, it has no effect on five amino acids and Schiff base ligand, while it has been shown to have a clear effect on lanthanum complexes.

Keywords: *Schiff base ligand, Amino acids, Mix ligand complexes, Spectral studies and antibacterial studies.*

Introduction

Schiff base have known since 1864 when Hugo Schiff reported the condensation of primary amines with carbonyls compounds [1-2]. Coordination chemistry appoints Schiff bases that fulfilled primary significance on this epoch [3-4]. Intensive studies have been done at complexation from Schiff rule for metals because the cute physic-chemical characteristics from metal complexes as well wide range for employment on diverse regions for science [5-6].

Amino acids are molecules including a carboxylic acid group, and an amine group aside series that diversity between various amino acids. Molecules are especially significant on bio-chemistry [7-8], where this idiom indicates into amino acids for the generic formula HNCHROOH; whither R is an organic substituent. Amino acids consisting uncharged amino groups, on the value of the degree of acidity physiological, are likewise submit Schiff base formation whom present other mechanism with metal complexes.

The characterization at quantitative investigation of the binding features from amino acids towards transition metal ions plays an important role in biological activity. Amino acids of metal complexes were long from interest a model for metal-ligand system and interaction which may occur in nature [9-13]. Rare earth ions possess the properties of antibacterial, antitumor and antivirus agents when co-ordinated with organic ligands and participate effectively in many important life processes.

It has studied many researchers preparation, features, antimicrobial, as well as suspended with toxins activity from mixed ligand complexes from metal transition elements, lanthanide metal ions [14-19]. In this paper, is to synthesis and characterization of mixed ligand of Schiff base ligand (L) with some amino acids (HAA) as glycine, L-alanine, L-valine, L-asparagine and DL-phenylalanine with lanthanium (III) ion. Schiff base ligand also mixed ligand complexes with amino acids (AA) that been prepared, have been

analysed as well featured through employing various techniques like elemental analysiz, (FT.IR, mass also UV Vis) spectrum and ^{1}HNMR and ^{13}CNMR spectrum of the Schiff base. Also, the Schiff base, five amino acid and mixed ligand complexes with La (III) have been estimated with in vitro antibacterial effectiveness versus positive also negative bacterial studied.

Experimental

Instrumentation

C: H: N elemental analysis from Schiff base ligand also La (III) complexes have been achieved the micro analytical on Euro EA Elemental Analyser. The molar conductivity values have been measured on (10^{-3}M) of DMSO at 25^{o}C with Schiff base ligand, five amino acids as well La(III)- complexes on CON 510 Conductivity. Electronic absorption spectra in DMSO (10^{-3}M) at 1.000±0.001 cm matched quartiz cell in the range 200-1100 nm. From Schiff base ligand, five amino acids as well La (III) - complexes in the ultraviolet and visible range, respectively, were measured on using Shimadzu-U.V-160. FT.IR spectra from Schiff base ligand, five amino acids as well La (III) - complexes recorded in KBr discs in the range 4000-400 cm^{-1} on FT-IR-600 FT-IR Spectrophotometer. 1H also 13CNMR spectral from Schiff base ligand was recorded at NMR broker 500mhz. Stuart Melting Point Apparatus was used to determined melting point from Schiff base as well La (III) - complexes. Mass spectra from Schiff base as well La(III)- complexes have been registered by Agilent 5975c vl msd Mass Spectrometer selection Technology (HP) with Triple-Axis Detector by the analyzer Quadrupole at 230 ^{0}C.

Materials

Most of the chemicals used were of Fluk, B.D.H, Merk, Sigma and Alderich have been clarified through standard methods. Solvents such as C_2H_5OH, CH_3OH, $CHCl_3$, $(CH_3)_2CO$, $(C_2H_5)_2O$, petroleum ether, CCl_4, C_6H_6, $(CH_3)_2NC$ (O) H (DMF) and $(CH_3)_2SO$ (DMSO) have been refined also dried relative to standard procedures.

Preparation of Schiff base and La (III) Complexes

The synthesis of the ligand in Figure (1), benzaldehyde and o-phenylendiamine were prepared of Schiff base ligand (L) reported method in literatures [20-21].

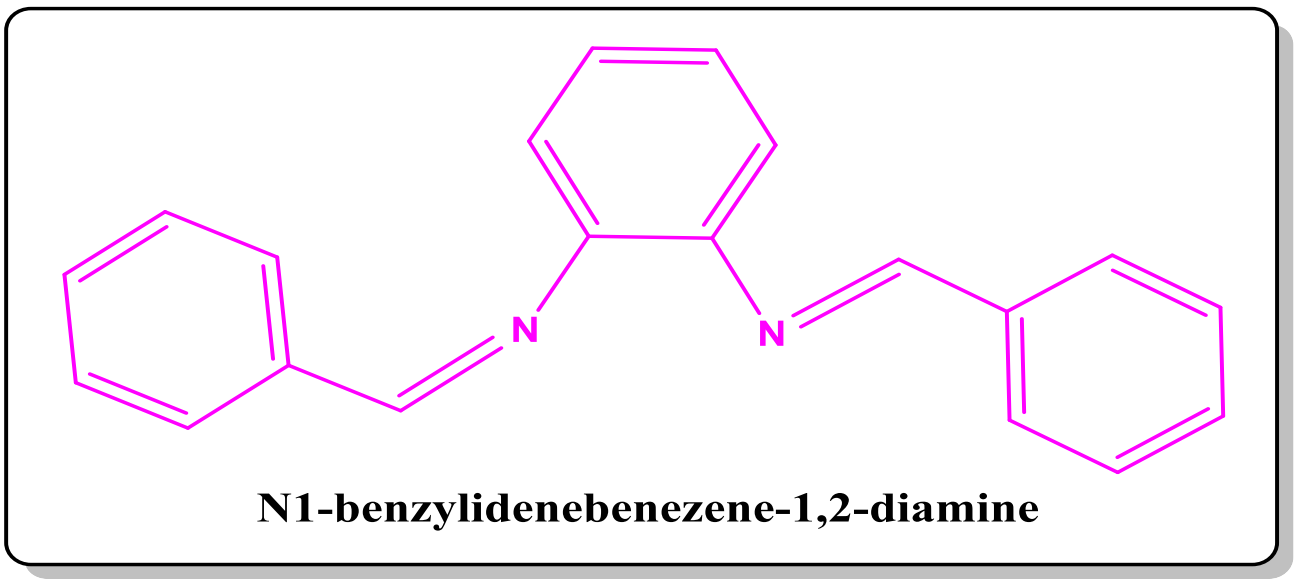


Figure 1: Structure of the Schiff Base Ligand (L)

The metal complexes were prepared from lanthanium (III) chloride pentahydrate, Schiff base ligand also five amino acids (HAA) like glycine, L-alanine, L- valine, L-asparagine and DL- phenylalanine. An aqueous solution (10ml) of lanthanium(III) chloride pentahydrate (0.371gm,1mmole) was mixed with ethanolic solution (10ml) of Schiff base ligand (0.284 gm,1 mmole) and (10ml) of ethanolic solution at each of the glycine (0.150 gm, 2mmole), Lalanine (0.196 gm, 2mmole), L-valine (0.234 gm, 2 mmole), L-aspargine (0.300 gm, 2 mmole) and DL-phenylalanine (0.330 gm, 2 mmole)which is added to each amino acid sodium hydroxide (0.08 gm) dissolved in the water solution.

The reaction mixture (1:1:2), (La: L: 2AA) molar proportion was taken in water bath and heated for about 15 minutes on the temperature to 10^0C. Then mixture has been cooled also colored solid complex acquired has been filtrated, washed for acetone.

The complexes so synthesized have been drain under vacuum.

Instrumentation

C: H: N elemental analysiz from Schiff base ligand also La (III) complexes have been achieved the micro analytical on Euro EA Elemental Analyser.. The molar conductivity values have been measured on (10-3M) of DMSO at 25°C with Schiff base ligand, five amino acids as well La (III)- complexes on CON 510 Conductivity. Electronic absorption spectra in DMSO (10^{-3}M) at 1.000±0.001 cm matched quartiz cell in the range 200-1100 nm. From Schiff base ligand, five amino acids as well La (III) - complexes in the ultraviolet and visible range, respectively, were measured on using Shimadzu-U.V-160.

FT.IR spectra from Schiff base ligand, five amino acids as well La (III) - complexes recorded in KBr discs in the range 4000-400 cm^{-1}on on FT-IR-600 FT-IR Spectrophotometer. ^{1}H also ^{13}CNMR spectral from Schiff base ligand was recorded at Nmr broker 500 mhz. Stuart Melting Point Apparatus was used to determined melting point from Schiff base as well La (III) - complexes. Mass spectra from Schiff base as well La(III)- complexes have been registered by Agilent 5975c vl msd Mass Spectrometer selection Technology (HP) with Triple –Axis Detector by the analyzer Quadrupole at 230 ^{0}C.

Antibacterial Assay

Antibacterial activities of the tested for Schiff base, La(III) salt, five amino acids and mixed ligand complexes have been estimated through using well agar diffusion method [22] using G^+bacteria *Staphylococcus aureas* and *Bacillus subtilis*, G^-bacteria *Burkholderia cepacia* and *Escherichia coli*.

The chemical compounds against growth isolated one colony of bacterial from the media of nutrient agar in bacteria and placed in sterile distal water and compares with the turbidity µc Farm land tubes containing 1.5×10^{-8} cell / ml. Then 100 microleter from suspension bacterial is taken by micropipette, then transport to muller hinton agar plate in bacteria. Chemical compounds are dissolving in DMSO and leave the plate 15 minute degree temperature of laboratory, then incubated at 37°C for 24hr. and diameters were measured the inhibition zone of bacterial growth replicated the test three times.

Results and Discussion

Characterization of Schiff Base and Mixed La (III) Complexes

Schiff base ligand (L was synthesized through reaction from benzaldehyde with o-phenylenediamine as well as characterized by ^{1}HNMR, ^{13}CNMR, FT.IR, mass and UV-Vis spectral. The attended from mixed ligand La(III)- complexes with five amino acids (HAA) such as glycine (Gly), L-alanine (Ala), L-valine(Val), L-asparagine(Asp) and DL-phenylalanine(Phe). The synthesis from mixed ligand La (III) - complexes can be explained accordingly follows:

$$LaCl_3.7H_2O + L + 2HAA + 2NaOH \longrightarrow [La(L)(AA)_2]Cl.H_2O + 2NaCl + 8H_2O$$

1 : 1 : 2 (mixed ligand complexes)

In general the complexes are coloured, non-hygroscopic, thermal stable solids according to the Table (1). The complexes are soluble in water, ethanol, methanol, DMSO and DMF, but insoluble in diethylether, acetone, benzene, carbon tetrachloride and chloroform. The element analysis data in Table (1) of Schiff base as well as La (III) - complexes are proportionate for generic formation accordingly (1:1:2) mixed ligand La (III)-complexes for the assort [La (L) (AA) $_2$] Cl.H_2O. Molor conductivity values from complexes on DMSO when the (10^{-3}M) condensation described to electrolytic type [23] shown in Table (1).

Table 1: Physical Characteristics for Schiff Base Ligand and Mixed Ligand La (III) – Complexes

Compounds	Empirical formula	Molecular weight (g/mol)	Color	M.P °C	Λm (S.cm.mol^{-1}) in DMSO	Elemental micro analysis, Found (Calc.)%		
						C	H	N
Schiff base ligand (L)	$C_{20}H_{16}N_2$	284.4	Yallow	(82-85)	6.34	83.8 (84.48)	5.33 (5.67)	8.96 (9.85)
Glycine	$C_2H_5NO_2$	75.07	White	233	2.92	-	-	-
L-Alanine	$C_3H_7NO_2$	89.09	White	258	2.98	-	-	-
L-Valine	$C_5H_{11}NO_2$	117.15	White	298	3.63	-	-	-
L-Asparagine	$C_4H_{10}N_2O_4$	150.13	White	233-235	4.87	-	-	-
DL-Phenylalanine	$C_9H_{11}NO_2$	165.00	White	105	5.92	-	-	-
[La(L)(Gly)$_2$].Cl.H$_2$O	[$LaC_{24}H_{24}N_4O_4$]Cl.H_2O	624.99	Deep green	240	30.2	45.34 (46.12)	2.04 (3.87)	7.12 (8.96)
La(L)(Ala)$_2$].Cl.H$_2$O]	[$LaC_{26}H_{28}N_4O_4$]Cl.H_2O	671.04	Deep green	238	31.2	45.81 (46.54)	3.09 (4.21)	7.43 (8.35)
La(L)(Val)$_2$].Cl.H$_2$O]	[$LaC_{30}H_{36}N_4O_4$]Cl.H_2O	709.15	Reddish brown	248	46.0	49.84 (50.81)	4.33 (5.12)	8.40 (7.90)
[La(L)(Asp)$_2$].Cl.H$_2$O	$LaC_{28}H_{34}N_6O_8$	775.12	Reddish brown	246	30.92	42.61 (43.39)	3.04 (4.42)	9.71 (10.84)
La(L)(Phe)$_2$].Cl.H$_2$O	[$LaC_{38}H_{36}N_4O_4$].$4H_2O$	804.86	Greenish brown	230	66.2	55.38 (56.71)	3.73 (4.51)	6.47 (6.91)

^{1}HNMR and 13CNMR Spectral for Schiff Base Ligand (L)

The ^{1}HNMR also ^{13}CNMR spectra of the Schiff base ligand were obtained in DMSO-d_6 by employing TMS an internal standard at ambient temperature. The ^{1}HNMR and ^{13}CNMR spectra of the ligand are shown in Figure (2) and (3), respectively. The ^{1}HNMR spectral from ligand offer many signals on (δ=7.00-8.02) ppm (m, 14H, C-H) assigned to aromatic protons. Signal obtained at (δ=8.24-8.26) ppm due to δ (HC=N)) of imine group, the signal observed at (δ=2.50) ppm lead to DMSO-d_6 [24-25]. ^{13}CNMR spectrum of the ligand shows resonance at (δ=166.98) ppm described to carbon of (C=N) of imine group. Various signals at (δ= 136.36, δ=135.38, δ=132.14, δ=130.68, δ=129.63, δ=128.52 and δ=125.54) ppm lead to carbon atoms from aromatic rings. Signal on δ=38.98 ppm due to DMSO-d_6 [26-27].

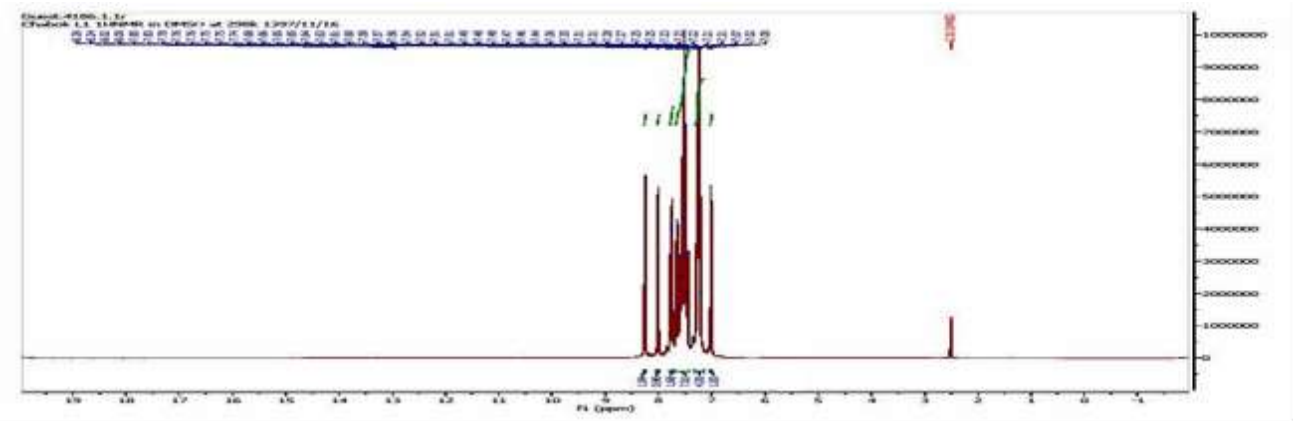


Figure 2: 1HNMR of the Schiff Base Ligand

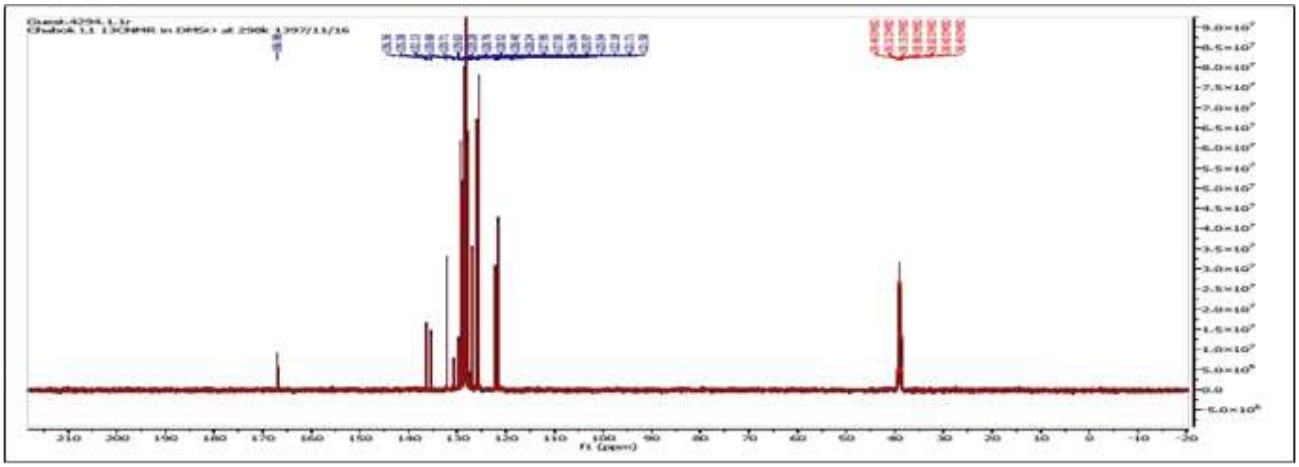


Figure 3: ^{13}CNMR of the Schiff Base Ligand

Electronic Absorption Spectra for Schiff Base, Five Amino Acids and Mixed Ligand La La (III)-Complexes

The electronic spectra of Schiff base, five amino acids and mixed ligand complexes were recorded in the UV-Visible region at Table (2). The spectrum of Schiff base ligand shows two peaks at 300 nm and 340 nm which were assigned to (π-π*) and (n-π*) electronic transitions respectively, connected for the azomethine chromosphere (HC=N) [28]. The electronic spectra of amino acids display peaks at (264-261) nm [29] due to ligand field, the peaks at the range (991-762) nm which were assigned to f-f electronic transitions [30-31].

Table 2: UV-Vis Datum with Schiff Base Ligand, Five Amino Acids as well Mixed Ligand La (III) – Complexes

Compounds	(λ nm)	ABS	Wave number(cm^{-})	C_{max} ($L.mol^{-1}.cm^{-1}$)	Assignments
Schiff base ligand (L)	300 340	2.385 0.387	33333 29411	2385 387	π–π* n–π*
Glycine	260 361	2.232 1.545	38461 27700	2232 1545	π–π* n–π*
L-Glanine	261 362	1.472 0.737	38314 27624	1472 737	π–π* n–π*
L-Valine	261 362	1.204 0.885	38314 27624	1204 885	π–π* n–π*
L-Aspargine	261 362	1.387 0.933	38314 27624	1387 933	π–π* n–π*
DL-Phenylalanine	261 362	1.443 0.687	38314 27624	1443 687	π–π* n–π*
[La(L)(Gly)$_2$].Cl.H$_2$O	263 765 805	0.927 0.017 0.017	38022 13071 12422	927 17 17	L.F F-F F-F
[La(L)(Ala)$_2$].Cl.H$_2$O	264 991	1.007 0.011	37878 10090	1007 11	L.F F-F
[La(L)(Val)$_2$].Cl.H$_2$O	263 762 852	1.023 0.033 0.031	38022 13123 11737	1023 33 31	L.F F-F F-F
[La(L)(Asp)$_2$].Cl.H$_2$O	261 841	1.215 0.021	383314 11890	1215 21	L.F F-F
[La(L)(Phe)$_2$].Cl.H$_2$O	264 989	0.941 0.039	37878 10111	941 39	L.F F-F

Infra-Red Spectra from Schiff Base Ligand, Five Amino Acids as well Mixed Ligand La (III) – Complexes

FT.IR spectra from Schiff base, amino acids and La (III) - complexes have been registered at KBr discs on a scale 4000-400 cm^{-1} at Table (3) and Figure (4) of the La(III) complex with glycine. The band appointed into azomethine group at Schiff base ligand has been watched on 1678 cm^{-1} as well shifted into lower frequency on La (III) - complexes (45-95) cm^{-1}. This evidences the involvement for nitrogen atom from azomethine group on coordination [32]. Broad band observed at (3149-3062) cm^{-1} and (2995-2927) cm^{-1} attributable into (N-H) asymmetric as well (N-H) symmetric vibrations for free amino acids moiety are shifted into wave numbers on range (3126-3040) cm^{-1} as well (3022-2031) cm^{-1} respectively, at spectral for complexes, proposing coordination from amino group during nitrogen for metal ion.

The asymmetric (COO^{-}) band for free amino acids which (1595-1527) cm^{-1} is shifted at range (1633-1583) cm^{-1} as well the symmetric (COO^{-}) mode watched on (1394-1311) cm^{-1} at spectral for free amino acids is observed to be shifted into lower wave number at range of (1365-1325) cm^{-1}, at spectra for complexes pointing out the coordination for carboxylic acid group through oxygen for the metal ion[33-34]. C-N symmetrical stretching frequency watched about (987-912) cm^{-1} on spectral for amino acids is found to be shifted to lower wave numbers at range of (976-850) cm^{-1} at spectral for complexes, confirming coordination during the amino group for amino acids [35]. As well a medium adversity band at higher frequency area is appointed into ν (OH) vibration for water on the complexes [30]. IR spectral for complexes offers a weak band at Table (3) assigned into M-N and M-O stretching vibration which attributed the chelation between the nitrogen atom and the studies metals for azomethane

group as well the coordination from metals for nitrogen from amino group oxygen atom for carboxylate anions group from studies amino acids [17]. At study from the research suggest that the La (III)- complexes have octahedral geometry shown in Figure (5).

Table 3: Some Important Infrared Bands (cm^{-1}) of Schiff Bases Ligand, Amino Acids and Mixed Ligand Complexes

Compounds	υ(OH) H_2O	υ(N-H) Asym.	υ(N-H) Sym.	υ(C=N)	υ(COO) Asym.	υ(COO) sym.	υ(C-N)	υ(M-N)	υ(M-N)	υ(M-O)
Ligand (L)	-	-	-	1678	-	-	974	-	-	-
Glycine	-	3105	2962	-	1595	1394	976	-	-	-
L-Alanine	-	3093	2995	-	1527	1338	912	-	-	-
L-Valine	-	3149	2954	-	1538	1311	945	-	-	-
L-Asparagine	-	3116	2927	-	1583	1387	987	-	-	-
DL-Phenylalanine	-	3062	2951	-	1576	1358	945	-	-	-
$[La(L)(Gly)_2]Cl.H_2O$	3365	3053	2980	1610	1458	1363	908	584	505	467
$[La(L)(Ala)_2]Cl.H_2O$	3377	3070	2985	1583	1477	1365	850	538	486	459
$[La(L)(Val)_2]Cl.H_2O$	3354	3040	2978	1603	1523	1325	893	592	542	490
$[La(L)(Asp)_2]Cl.H_2O$	3377	3107	2943	1633	1435	1356	972	557	521	459
$[La(L)(Phe)_2]Cl.H_2O$	3354	3110	3022	1599	1493	1346	916	598	530	482

Figure4: Spectra for La (III) Complex with Schiff Base Ligand and Glycine

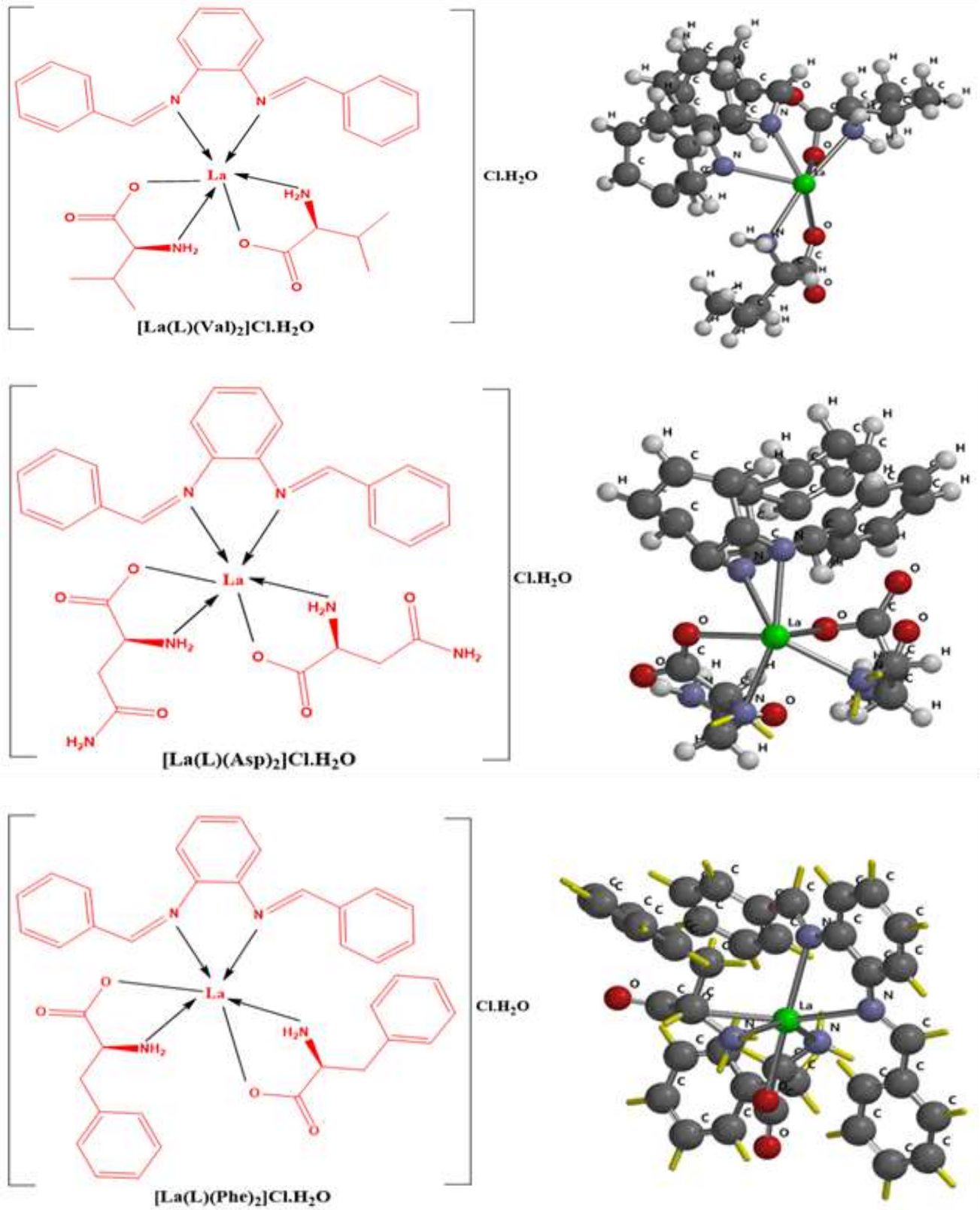
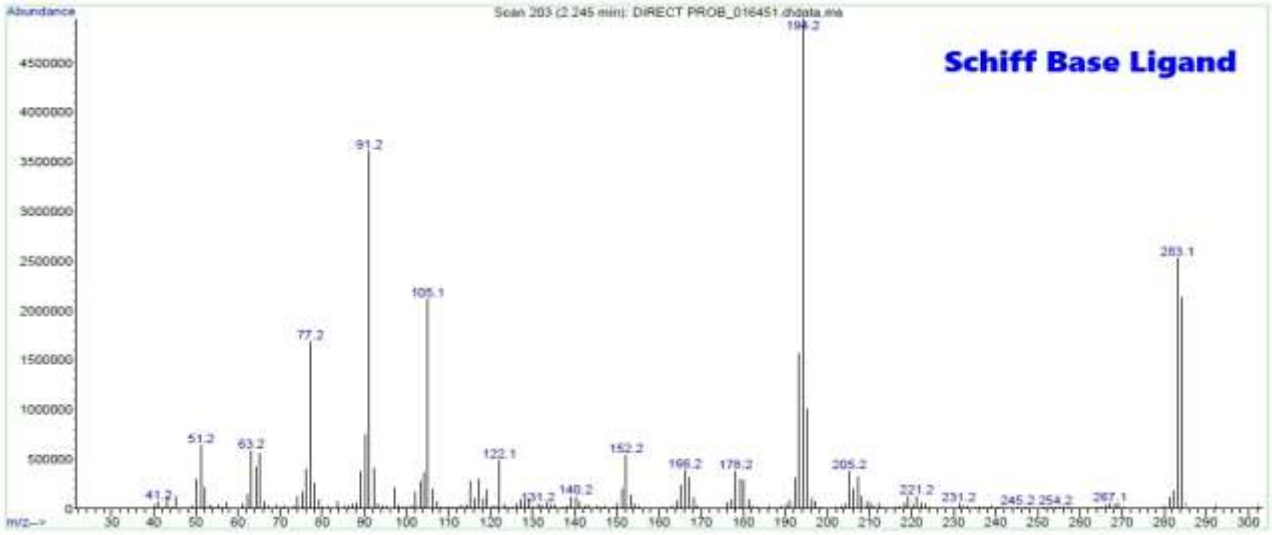


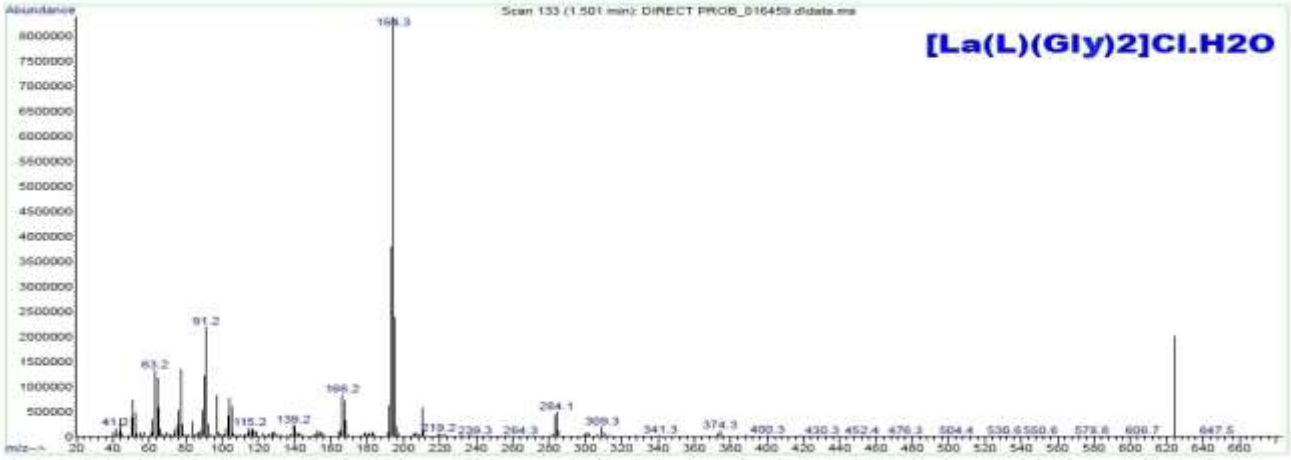
Figure 7: The Proposed Structure and 3D- Geometrical Structure to the all Mixed Ligand Complexes with La (III) Ion

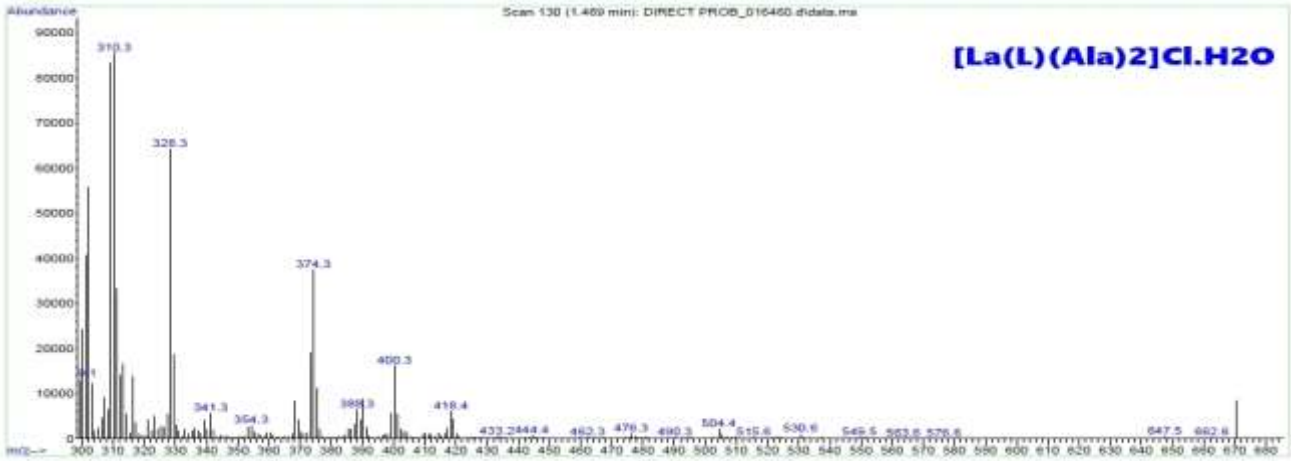
Mass Spectra for Schiff Base Ligand as well Mixed Ligand La (III) - Complexes

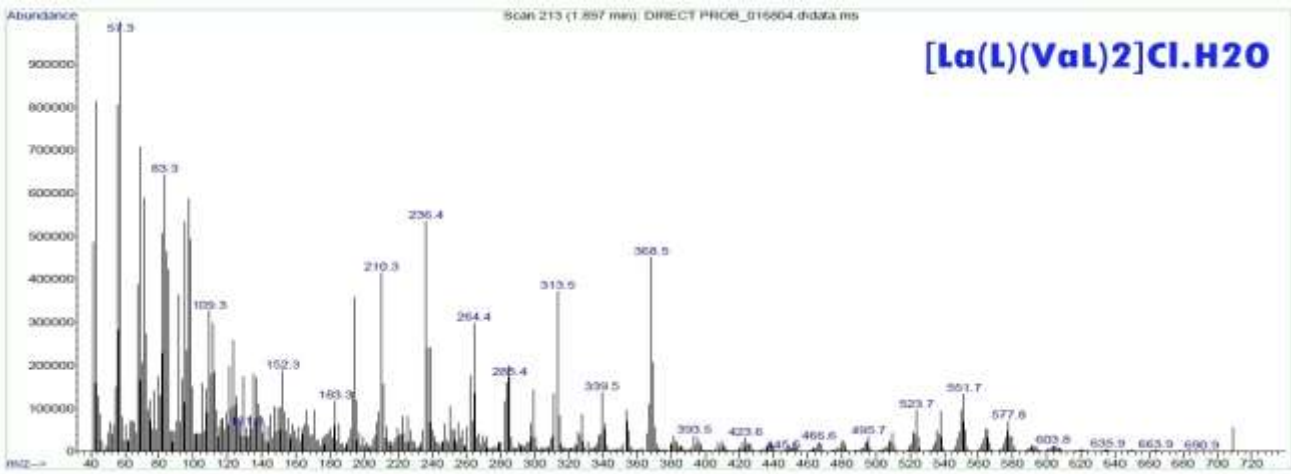
Mass spectrum for Schiff base ligand also mixed ligand La (III)-complexes are offered at Figure (6). Spectra of the Schiff base ligand as well mixed ligand La(III)-complexes have been registered also they are utilized to liken stoichiometry installation. Schiff base offers a molecular ion. Peak on m /z 284.4.

Molecular ion peak with mixed ligand La(III)- complexes, watched with glycine on m / z 624.99, alanine at m / z 671.04, valine at m / z 709.15, asparagine at m / z 775.12 and phenylalanine at m / z 804.86 confirmed stoichiometry from metal chelates. This installation is as well backed up by the spectra of mass from complexes. It is in good harmonization for the micro-analytical datum.









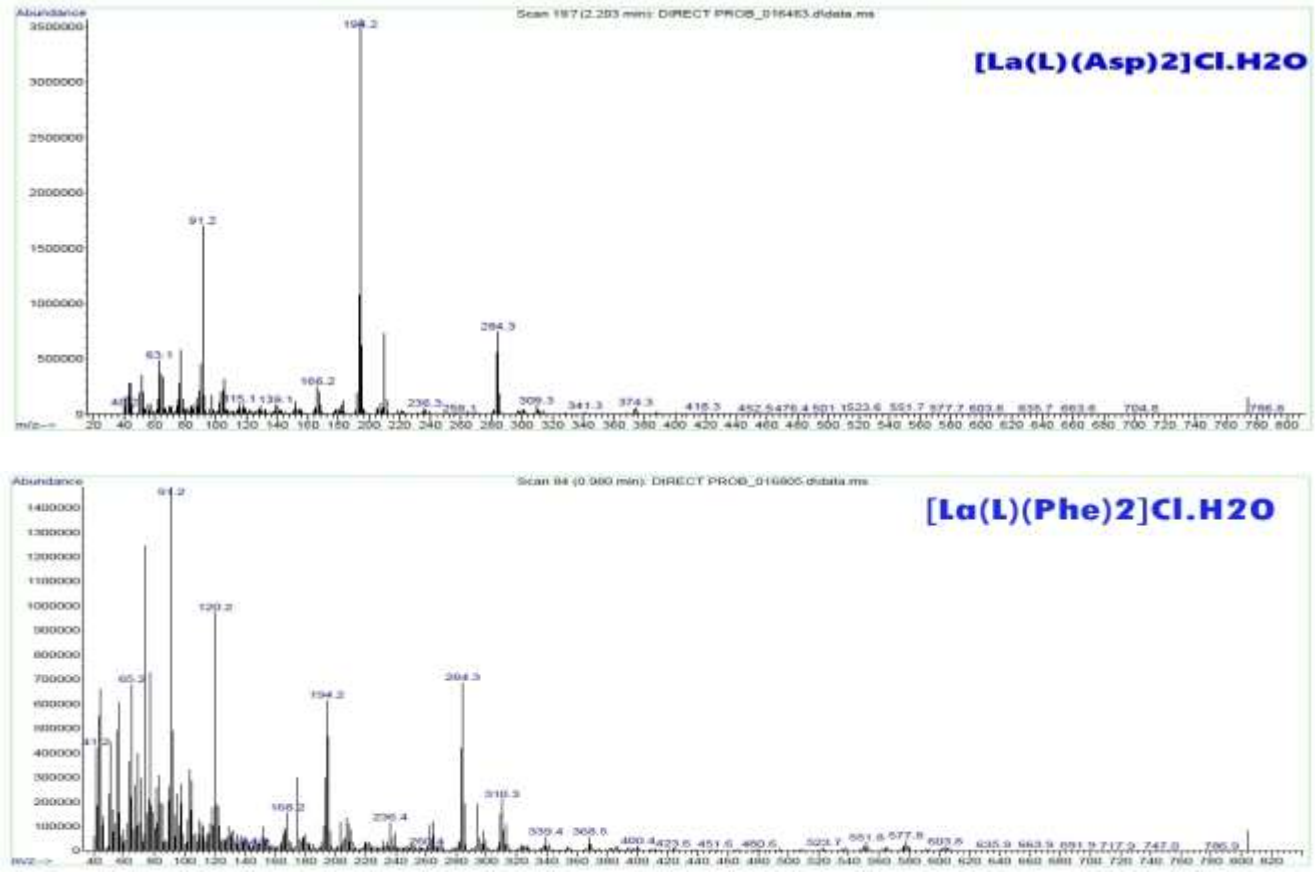


Figure 6: Mass Spectra for Schiff Base Ligand and all La (III) Complexes

Biological Studies

The antibacterial activities for Schiff base ligand, La(III) salt, five amino acids and mixed ligand complexes have been estimated through the agar diffusion method against G^+-bacteria *taphylococcus aureas, Bacillus subtilis*, G^--bacteria *Burkholderia cepacia* and *Esherichia coli*. The outcomes, expressed like the diameter for growth inhibition region at millimeters, are given on Table (4) and Figure (7). Indicate the Schiff base ligand and five amino acids show non-activity against the four species of bacteria. On the other hand, the all mixed ligand complexes with La (III) show good activity against the gram negative (*Burkholderia cepacia*) and the gram positive (*Bacillus subtilis*) and the La (III) salt with four bacteria. Also the gram positive (*Staphylococcus aureas*) with the La (III) complexes with valine, asparagine and phenylalanine, i.e. are labeled can be into carboxyl group from each amino acid [23]. As well as, for the outcomes, it was watched that the complexes explain that they have more as well better efficiency than the five amino acids and Schiff base ligand. The confirmed efficacy for the complexes may be demonstrated at expression for the nature for the cell membrane. It points out that chelating accretion the antibacterial efficiency.

Table 4: Results of Antimicrobial Activity of the Schiff Base Ligand, La (III) Salt, Five Amino Acids and Mixed Ligand Complexes in DMSO

Compounds	*Burkholderia cepacia* G^-Ve	*Esherichia coli* G^-Ve	Staphylococcus aureas G^+Ve	Bacillus subtilis G^+Ve
L	-	-	-	-
Glycine	-	-	-	-
Alanine	-	-	-	-
Valine	-	-	-	-
Aspargine	-	-	-	-
Phenylalanine	-	-	-	-
$LaCl_3$	30	28	28	29
La-L-Gly	13	-	-	16
La-L-Ala	14	-	-	13
La-L-Val	14	-	12	15
La-L-Asp	14	-	13	16
La-L-Phe	16	-	16	17
Control(DMSO)				

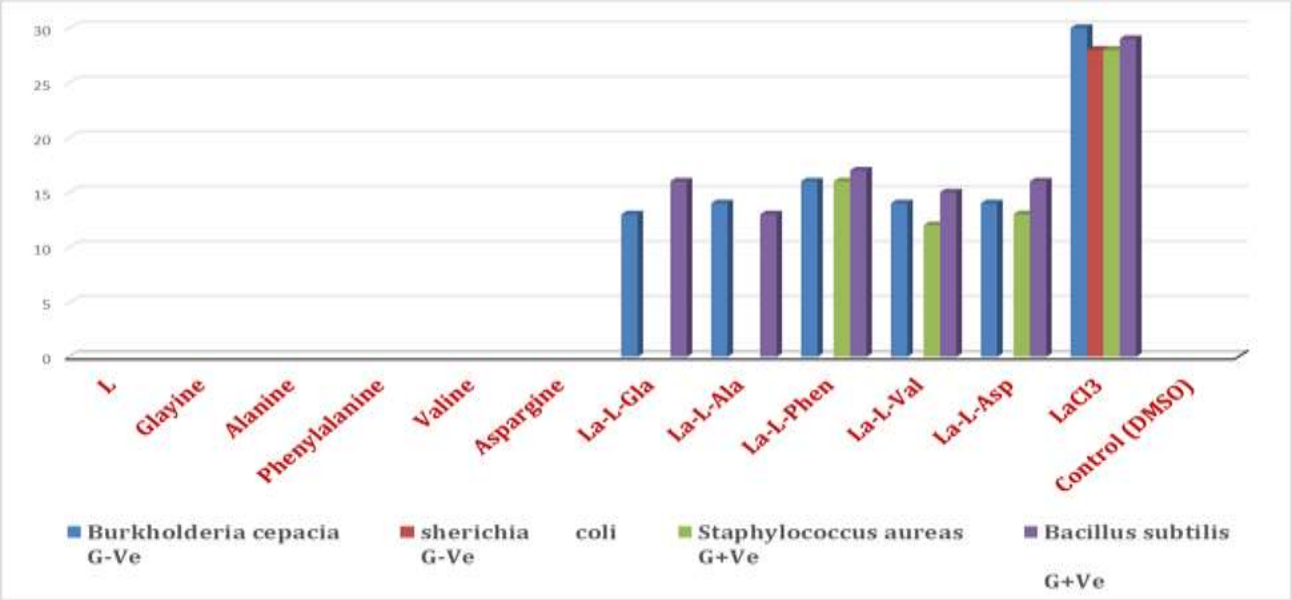


Figure 7: Antibacterial Evaluation of the Investigated Schiff Base Ligand, La(III) Salt, Five Amino Acids and Mixed Ligand Complexes Against *Burkholderia cepacia, Esherichia coli, Staphylococcus aureas* and *Bacillus subtilis* Bacteria

Conclusion

The current search reports about synthesis description, ^{1}HNMR, ^{13}CNMR, electronic absorption, mass and FT.IR spectral for Schiff base ligand (L) from benzaldehyde and o-phenylenediamine as well mixed ligand complexes of La(III) with five amino acids (AA) such as glycine, L-alanine, L-valine, L-spargine as well as DL-phenylalanine. The synthetic procedure at this work led to the formation for complexes to the mole ratio (1:1:2) (La: L: AA). All complexes have been distinguished with analysiz of elements, (FT.IR, mass also UV.Vis) spectra, magnetic moment and molar conductance. Newly synthesized of the five complexes participated on bonding into La (III) like mono-basic mono-dentate ligand during the azomethine nitrogen. Amino acid as well acts like mono-basic bi-dentate ligand by amino as well ionized carboxylate groups. Electronic, FT.IR and mass spectral studies suggest that the La(III) complexes have octahedral geometry .The antibacterial efficiency outcomes pointed out that cheked complexes were more energetic versus *Burkholderia cepacia, Bacillus subtilis* and *Staphylococcus aureas* bacteria compared with the Schiff base ligand and free five amino acids. It can be deduced that antibacterial efficiency for the compounds is related to cell wall structure for the bacteria [36].

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