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Synthesize, Characterization and Thermal behavior of some New Mercury and Cadmium halides Coordination compounds of Recently synthesized Schiff base

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Abstract

Some new cadmium (II) and mercury (II) halides complexes of the bidentate Schiff base ligand N,N-bis [(E)-3-(2-nitrophenyl)allylidene)]benzene-1,2-diamine (L) were synthesized, and characterized by physical and spectral study such as elemental analysis, molar conductance, UV–visible spectra, FT-IR spectra, ^{1}H NMR and ^{13}C NMR spectra. All complexes were stable in DMF solvent and the low molar conductivity confirms their stabilization and the non-electrolytic nature of them. The changes in the location and shape of the peaks in UV-visible, FT-IR, and the ^{1}H and ^{13}C NMR spectra of complexes rather than free ligand are the other evidence to form Schiff base complexes. The suggested structure of the complexes is pseudo-tetrahedral. Thermal behaviors of complexes were also investigated.

Keywords: Schiff base, complex, diamine, spectra, thermal.

Introduction

Schiff bases are characterized by the HC=N- (imine) group which is important in illustrating the mechanism of the reactions in biological systems[1-3]**.** A wide range of Schiff bases have been synthesized due to the great flexibility and diverse structural aspects. Schiff base and its complex derivatives, especially heterocyclic amine family, have been an important field in drug research and development due to their broad bioactivities such as antitumor, antibacterial, and antiviral activities[4,5].

These complexes have also applications in clinical, analytical and industrial in addition to their important roles in reversibly bind oxygen in epoxidation reactions[6], biological properties[7-9] catalytic role in hydrogenation of olefins[10,11], photochromic properties[12], analytical determination[13-15] and organic synthesis[16]. In these years, attention to group XII metal complexes with a stable d^{10} electronic configuration have been increased in the field of inorganic chemistry, biochemistry and environmental chemistry[17,18].

In continuation of our research[19-21], the aim of this work is to prepare and investigate the some complexes of Cd(II) and Hg(II) with recently synthesized bidentate Schiff base ligand of N,N-bis [(E)-3-(2-nitrophenyl) allylidene)] benzene-1,2-diamine (L) with nitrogen atoms as donor sites[21]. The general formula of these complexes are MLX_2 in which M= Cd(II) and Hg(II), L= Schiff base ligand and X= chloride, bromide, iodide. The ligand and complexes were characterized by physical and spectral data including microanalysis, FT-IR, UV-visible, ^{1}H and ^{13}C NMR and conductivity measurements.

Material and Methods

General: 2-Nitrocinnamaldehyde, 1,2-phenylendiamine, cadmium(II) and mercury(II) halides and other chemicals were purchased from either Aldrich, Merck or BDH Chemicals. All the chemicals used were of analytical grade. Solvents were purified and dried before use according to the standard method. FT-IR spectra in KBr pellets were recorded on a JASCO FT/IR-680 spectrometer in the 4000–400 cm^{-1} range. Electronic spectra were recorded in DMF solutions on a JASCO-V570 model spectrometer with quartz cells of 0.5cm path length. ^{1}H and ^{13}C NMR spectra were obtained using a Brucker DPX FT-NMR spectrometer at 500MHz with the samples dissolved in DMSO-d_6 mixture using TMS as internal standard. MS (m/z) of the ligand was recorded on Shimadzu model GC–MS QP5050. Carbon, hydrogen and nitrogen of dried samples were performed using an elemental analyzer. The melting points (°C) of the complexes were recorded on BUCHI melting point B-545 instrument. Conductivity measurements of the ligand and their complexes were made on freshly prepared 10^{-3}M solutions in DMF at room temperature with a Metrohm 712 conductometer with a dip-type conductivity cell made of platinum black.

Preparation of N,N-bis [(E)-3-(2-nitrophenyl) allylidene)] benzene-1,2-diamine (L): The ligand was prepared as in our previous report[21]. The structure of the Schiff-base is presented in figure-1.

Synthesis of complexes: The Schiff base ligand (1 mmol) in 20 mL methanol was added gradually to a same amount of metal salt (1 mmol) (MX_2, where M= Cd(II) and Hg(II); X= chloride, bromide and iodide) in methanol or ethanol and then the mixture was vigorously stirred at room temperature for 4–6 h. When the solid product formed, the precipitate as complex was collected by filtration and then washed several times with warm methanol. The product was dried in air and stored in a desiccator over anhydrous $CaCl_2$ under vacuum. All the metal complexes were stable to air and moisture.

$CdLCl_2$: FT-IR(KBr), cm^{-1}: 3420(m), 3064(m), 2927(w), 2852(w), 1625(s), 1607(s), 1577(s), 1523(vs), 1440(m), 1343(vs), 1167(s), 986(m), 957(m), 787(m), 743(s), 677(m), 458(w). UV-visible(DMF), λ(nm) (ε,$M^{-1}cm^{-1}$): 284(47983) and 364(19422). ^{1}HNMR spectra (in DMSO-d_6): 8.51(bs, $2H_a$), 8.06(t, $4H_{f,g'}$, J= 9.16 Hz and J= 9.38 Hz), 7.79(m, $2H_{f'}$), 7.69(d, $2H_c$, J= 14.36 Hz), 7.66(t, $2H_g$, J= 6.85 Hz and J= 7.80 Hz), 7.56(bs, $2H_b$), 7.33(bs, $2H_d$), 7.21(bs, $2H_e$). ^{13}CNMR spectra (in DMSO-d_6): 163.32(C_8), 149.08(C_3), 143.82(C_4), 139.04(C_9), 134.54(C_{11}), 133.38(C_{10}), 131.44(C_{12}), 130.82(C_5), 129.37(C_6), 127.77(C_7), 125.57(C_2), 120.77(C_1).

$CdLBr_2$: FT-IR(KBr), cm^{-1}: 3443(m), 3063(m), 2912(w), 2854(w), 1624(s), 1607(s), 1576(s), 1523(vs), 1441(m), 1343(vs), 1166(s), 984(m), 955(m), 786(m), 742(s), 678(m), 458(w). UV-visible(DMF), λ(nm) (ε,$M^{-1}cm^{-1}$): 284(48436) and 366(19586). ^{1}HNMR spectra (in DMSO-d_6): 8.49(bs, $2H_a$), 8.06(t, $4H_{f,g'}$, J= 7.31 Hz and J= 6.52 Hz), 7.80(t, $2H_{f'}$, J= 7.03 Hz and J= 7.24 Hz), 7.69(d, $2H_c$, J= 13.84 Hz), 7.66(t, $2H_g$, J= 6.87 Hz), 7.48(bs, $2H_b$), 7.33(bs, $2H_d$), 7.22(bs, $2H_e$).

$CdLI_2$: FT-IR(KBr), cm^{-1}: 3442(m), 3063(m), 2924(w), 2852(w), 1623(s), 1606(s), 1573(s), 1518(vs), 1443(m), 1344(vs), 1267(w), 1171(s), 990(m), 950(m), 786(m), 744(s), 680(m), 466(w). UV-visible(DMF), λ(nm) (ε,$M^{-1}cm^{-1}$): 284(45912) and 366(18863). ^{1}HNMR spectra (in DMSO-d_6): 8.56(d, $2H_a$, J= 7.06 Hz), 8.07(d, $2H_f$, J= 8.24 Hz), 8.05(d, $2H_{g'}$, J= 8.15 Hz), 7.82(t, $2H_{f'}$, J= 7.51 Hz and J= 7.60 Hz), 7.69(d, $2H_c$, J= 15.59 Hz), 7.67(t, $2H_g$, J= 7.78 Hz and J= 7.73 Hz), 7.57(bs, $2H_b$), 7.36(bs, $2H_d$), 7.29(bs, $2H_e$). ^{13}CNMR spectra (in DMSO-d_6): 163.08(C_8), 149.09(C_3), 143.82(C_4), 139.09(C_9), 134.56(C_{11}), 133.17(C_{10}), 131.52(C_{12}), 130.72(C_5), 129.29(C_6), 127.77(C_7), 125.62(C_2), 120.90(C_1).

$HgLCl_2$: FT-IR(KBr), cm^{-1}: 3442(m), 3066(m), 2922(w), 2851(w), 1620(s), 1606(s), 1574(s), 1518(vs), 1439(m), 1343(vs), 1264(w), 1167(s), 984(s), 951(m), 789(m), 743(s), 678(m), 464(w). UV-visible(DMF), λ(nm) (ε,$M^{-1}cm^{-1}$):262(29106) and 318(19744). ^{1}HNMR spectra (in DMSO-d_6): 8.68(bs, $2H_a$), 8.07(d, $2H_f$, J= 8.50 Hz), 8.01(d, $2H_{g'}$, J= 10.50 Hz), 7.80(m, $4H_{f',c}$), 7.68(m, $6H_{g,b,d}$), 7.34(m, $2H_e$). ^{13}CNMR spectra (in DMSO-d_6): 164.01(C_8), 149.07(C_3), 142.62(C_4), 139.10(C_9), 134.80(C_{11}), 133.02(C_{10}), 131.64(C_{12}), 130.68(C_5), 129.11(C_6), 128.92(C_7), 125.62(C_2), 121.01(C_1).

$HgLBr_2$: FT-IR(KBr), cm^{-1}: 3466(m), 3065(m), 2919(w), 2854(w), 1620(vs), 1607(s), 1573(vs) 1520(vs), 1440(m), 1343(vs), 1262(w), 1166(s), 984(s), 951(m), 788(m), 742(s), 678(m), 461(w). UV-visible(DMF), λ(nm) (ε,$M^{-1}cm^{-1}$): 264(36556) and 316(17292). ^{1}HNMR spectra (in DMSO-d_6): 8.71(d, $2H_a$, J= 8.50 Hz), 8.07(m, $4H_{f,g'}$), 7.83(m, $2H_{f'}$), 7.70(m, $6H_{c,g,b}$), 7.43(m, $2H_d$), 7.39(m, $2H_e$).

$HgLI_2$: FT-IR(KBr), cm^{-1}: 3466(m), 3062(m), 2919(w), 2854(w), 1623(s), 1605(s), 1574(s), 1517(vs), 1443(m), 1344(vs), 1169(s), 986(m), 951(m), 786(m), 759(m), 745(s), 679(m), 457(w). UV-visible(DMF), λ(nm) (ε,$M^{-1}cm^{-1}$): 262(45476) and 310(28862). ^{1}HNMR spectra (in DMSO-d_6): 8.08(m, $4H_{a,f}$), 7.83(m, $2H_{g'}$), 7.73(m, $6H_{f',c,g}$), 7.41(m, $2H_b$), 7.38(m, $4H_{e,d}$).

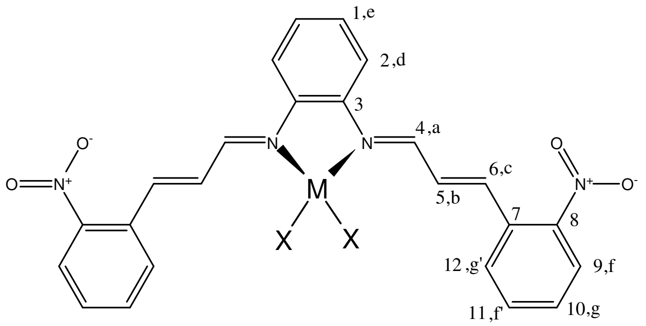

Figure-1
Suggested structure for the ligand and complexes (M=Cd(II), Hg(II) and X=Cl^-, Br^-, I^-)

Results and Discussion

Synthesis: N,N-bis-[(E)-3-(2-nitrophenyl)allylidene)]benzene-1,2-diamine(L) as a Schiff base ligand was synthesized by the condensation reaction between 2-nitrocinnamaldehyde and 1,2-phenylendiamin as reported in previous report[20,21]. This ligand was used as chelate ligand to prepare complexes of Cd(II) and Hg(II) with general formula MLX_2 in which X= Cl^-, Br^-, I^-. All complexes are insoluble in common solvent such as dichloromethane, chloroform, acetone and alcohols but they are soluble in dimethylsulfoxide and dimethylformamide. According to the information recorded by elemental analyses, the stoichiometry of ligand and its complexes was confirmed. The analytical and physical data of the Schiff base ligand and its complexes are given in table-1. The analytical data show that the 1:1 ratio (metal: ligand) is correct for all complexes. The molar conductivity was measured in DMF (10^{-3} M) at room temperature and low range of the obtained values 19.43-32.44 $cm^2\Omega^{-1}mol^{-1}$ indicates that the nature of all complexes is non-electrolyte[22-24].

IR spectra: The most characteristic absorptions of the bidentate Schiff base ligand and its complexes are summarized in table-2. In the IR spectrum of ligand, the absorption peak assigned to azomethine (-CH=N-) as a functional group, is appeared at 1609 cm^{-1}. This stretching frequency shifted to higher frequency in all mercury and cadmium complexes. The shifting to the higher frequency 1620-1625 cm^{-1} indicated this group was affected by complexation [24, 25]. In the IR spectrum of ligand, the absorption band of $CH_{aromatic}$ and $CH_{aliphatic}$ appeare at 3057 and 2916 cm^{-1} respectively that after complexation shifted to higher frequency. The peak at 2856 cm^{-1} assigned to iminic CH shifted smoothly to lower frequency after complexation. Two very sharp peaks assigned to asymmetric (ν_{asym}) and symmetric (ν_{sym}) streching band of nitro-group exist at 1519 and 1347 cm^{-1} that smoothly change in the IR spectra of complexes. The very strong out-of-plane bending of the aromatic C-H and C-C at 734 and 698 cm^{-1} are shifted to higher or lower frequencies after coordination. The important absorption band in the spectra of complexes attributed to stretching frequency of M-N bound are appeared in the region of 457-466 cm^{-1} [26]. This peak confirmed that the nitrogen of ligand is successfully coordinated to the metal ion.

Electronic Spectral Studies: Electronic spectra of the ligand and its complexes were recorded in DMF solvent at room temperature and the spectral data including the maximum wavelength are summarized in table-2. In the spectrum of ligand two absorption bands were appeared. An absorption band at 278 nm assigned to $\pi \rightarrow \pi^*$ transition of the aromatic ring moieties that shifted to the higher wavelength in the spectra of cadmium complexes while in mercury complexes this band has a shift to the lower wavelength. The other band at 374 nm attributed to $\pi \rightarrow \pi^*$ transition of imine groups which is mainly localized within the iminic chromophore. In the electronic spectra of all complexes, shifting to the lower wavelength (310-366 nm) suggest the coordination of the iminic nitrogen to the metal ions. The electronic spectra of d^{10} elements generally consists ligand to metal charge transfer (MLCT) that in our complexes were not separately observed (probably overlapped with internal transition of the ligand)[27]. The suggested structure of d^{10}-four coordinated complexes based our evidences and with considering our previous report on this type of ligands [28-30] is pseudo-tetrahedral as drawn in figure-1.

The FT-IR spectra of $CdLCl_2$ and $HgLCl_2$ are illustrated in figure-2.

^1H and ^{13}C NMR spectra: The ^1H and ^{13}C NMR spectra of complexes were recorded in DMSO-d_6 at 300 MHz and were assigned based on figure-1. Detailed assignment of NMR spectra has been brought in experimental section. The ^1H NMR spectrum of the ligand as in our previously report [21] showed the signal of iminic proton resonance as a functional group for Schiff base compound at 8.33 ppm as a doublet due to coupling with H_b. This signal has a red shift at all of complexes spectra and also in the ^1H NMR of $CdLCl_2$, $CdLBr_2$ and $HgLCl_2$ is appeared as broad singlet. At $HgLI_2$ spectra, this peak was overlapped with the signal of H_f and was exhibited as a multiplet signal. The change in the location and shape of iminic proton resonance signals confirm the coordination of imine nitrogen to metal. In the spectrum of ligand, the resonances of H_f and $H_{g'}$ were shown at 8.03 and 8.01 ppm as a doublet signals that after complexation smoothly shifted to the weaker fields except for $HgLI_2$ that in which, the signal of $H_{g'}$ shifted to 7.83 ppm as a multiplet signal. In the spectrum of ligand, H_b is observed at 7.24 ppm as a doublet of doublet due to coupling with H_a and H_c respectively. The signal of H_c is seen at 7.61 ppm as a doublet due to coupling with H_b. The signal of H_b and H_c in the spectra of complexes have a smoothly shift to the down fields with respect to free ligand. The doublet of doublet signals at 7.23 and 7.10 ppm attributed to H_d and H_e of ligand are shifted to weaker fields in spectra of complexes as broad singlet and multiplet signals in Cd(II) and Hg(II) complexes respectively. In the spectrum of the ligand, $H_{f'}$ is observed at 7.75 ppm as triplet due to coupling with the H_g and $H_{g'}$. H_g is appeared similar to $H_{f'}$ at 7.59 ppm as triplet due to its couplings with H_f and $H_{f'}$. After coordination ligand to metals, these signals have smoothly shifted to the weaker fields. The ^{13}C NMR spectrum of the ligand shows the iminic carbon (C_4) resonances as functional group signal at 144.36 ppm. This peak is shifted to 142.62-143.82 ppm in the complexes, suggesting well coordination of the iminic nitrogens to metal ions. The other peaks of aromatic and ethylenic carbons resonance are observed at 162.74(C_8), 148.109(C_3), 137.62(C_9), 133.57(C_{11}), 132.80(C_{10}), 130.10(C_{12}), 129.94(C_5), 128.56(C_6), 126.58(C_7), 124.53(C_2), 120.21(C_1) that shifted to the down field after formation of Schiff base complexes. The good ^{13}CNMR spectra were not recorded for some complexes as seen their absence in experimental section.

Table-1
Synthetic, analytical and conductivity data for the mercury and cadmium complexes.

Compound	Color	M.p.(^oC)	Yield(%)	Found (Calcd.) (%)			Λ_M ($cm^2\ \Omega^{-1}M^{-1}$)
				C	N	H	
$CdLCl_2$	Yellowish White	294(dec.)	71	47.18 (47.28)	9.22 (9.19)	2.82 (2.98)	19.43
$CdLBr_2$	Yellowish White	247(dec.)	77	-	-	-	20.06
$CdLI_2$	Yellowish White	258(dec.)	73	-	-	-	30.51
$HgLCl_2$	Yellowish White	184(dec.)	71	40.84 (41.30)	8.32 (8.03)	2.55 (2.60)	28.10
$HgLBr_2$	Cream	178(dec.)	85	-	-	-	32.44
$HgLI_2$	Yellow	198(dec.)	78	-	-	-	31.68

Table-2
FT-IR (cm^{-1}) and UV–visible (nm) spectral data of the Schiff-base ligand[21] and its mercury and cadmium complexes

Compounds	νCH (arom.)	νCH (aliph.)	νCH (imin.)	νC=N	νC=C	ν(–NO_2)	νCH arom(oop)	νC-C arom(oop)	νM–N	λ_{max}
Ligand	3057	2916	2856	1609	1583	1519, 1347	734	698	-	278, 374
$CdLCl_2$	3064	2927	2852	1625	1607	1523, 1343	743	677	458	284, 364
$CdLBr_2$	3063	2912	2854	1624	1607	1523, 1343	742	678	458	284, 366
$CdLI_2$	3063	2924	2852	1623	1606	1518, 1344	744	680	466	284, 366
$HgLCl_2$	3066	2922	2851	1620	1606	1518, 1343	743	678	464	262, 318
$HgLBr_2$	3065	2919	2854	1620	1607	1520, 1343	742	678	461	264, 316
$HgLI_2$	3062	2919	2854	1623	1605	1517, 1344	745	679	457	262, 310

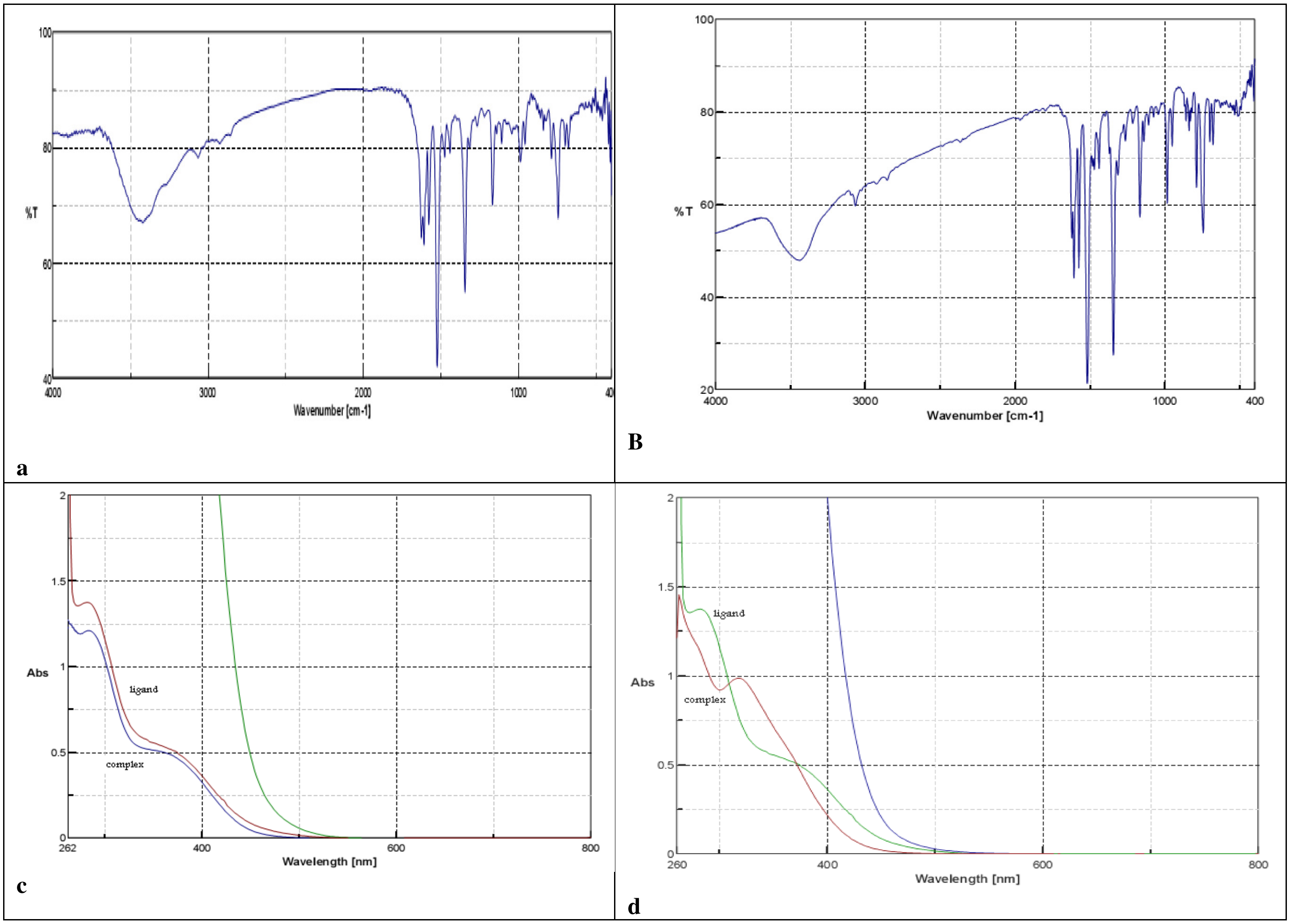

Figure-2
FT-IR of $CdLCl_2$(a), $HgLCl_2$(b) and UV-visible of $CdLCl_2$(c), $HgLCl_2$(d) in compared to ligand

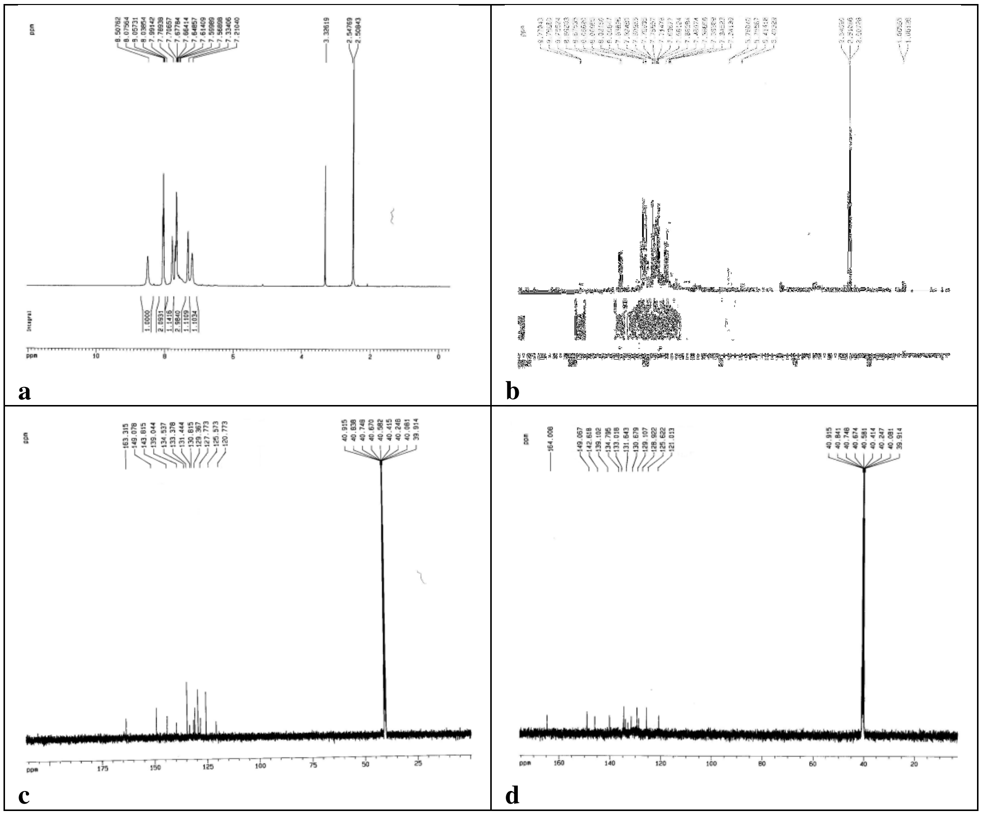

Figure-3
^{1} H NMR of $CdLCl_2$(a), $HgLCl_2$(b) and ^{13} C NMR of $CdLCl_2$(c), $HgLCl_2$(d)

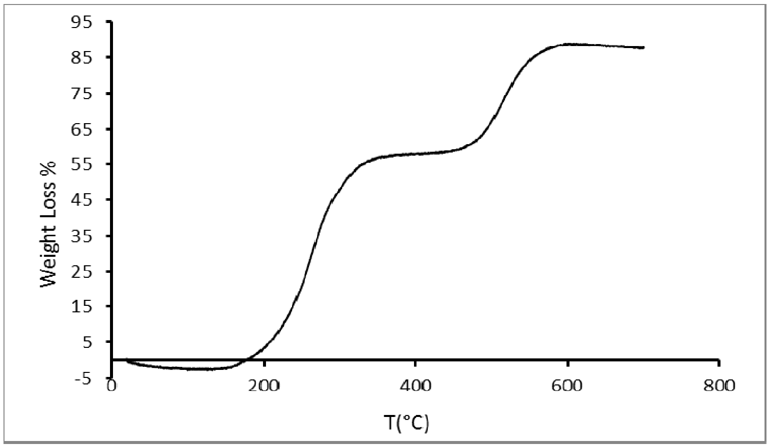

Figure-4
TGA plot of $HgLCl_2$ complex

Thermal behavior of the complexes: Thermal decomposition of the titled complexes was runned from room temperature to 700^0C at the heating rates of 10 (^{o}C/minute) under oxygen atmosphere. For instance, TGA plot of $HgLCl_2$ is shown in figure-4. The lack of weight loss under 200 ^{o}C states absence of water molecules in their structures. Complexes lose 78.4-96.7% of their weight via two temperature steps at the applied temperature range. It is suggested that mercury(II) and cadmium(II) oxide are main metal residue. The weight loss% of complexes at various temperature steps are summarized in table-3.

Conclusion

In this paper we reported synthesis and identified a bidentate ligand and its mercury and cadmium complexes. The compounds were characterized by physical, spectral data and conductivity measurement. It was found that the complexes are non-electrolyte in DMF solution. Thermal investigation of the complexes showed that they are decomposed via two temperature steps in the range of room to 700°C.

Table-3

Thermal weight loss% of the mercury and cadmium complexes

Compound	Temperature step(°C)	Weight loss%	Total weight loss%
$CdLCl_2$	200-400 400-600	38.6 58.1	96.7
$CdLBr_2$	200-420 420-660	13.7 64.7	78.4
$HgLCl_2$	20-420 420-605	58.0 30.9	88.9
$HgLBr_2$	200-420 420-660	60.9 29.4	90.3
$HgLI_2$	200-425 425-600	65.0 24.2	89.2

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