Synthesis and Characterization of New Bidentate Schiff Base Ligand Type (NO) Donor Atoms Derived from Isatin and 3-Amino Benzoic Acid and Its Complexes with Co(II), Cu(II), Cd(II) and Hg(II) Ions

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Abstract

New bidentate Schiff base ligand (L) namely [(Z)-3-(2-oxoindolin-3ylildeneamino)benzoic acid] type (NO) was prepared via condensation of isatin and 3-amino benzoic acid in ethanol as a solvent in existence of drops of (glac. CH₃COOH). The new ligand (L) was characterized base on elemental microanalysis, FT-IR, UV-Vis, ¹H-NMR spectra along with melting point. Ligand complexes in general formula $[M(L)_2Cl_2]$. H₂O, where: $M^{II} = Co$, Cu, Cd, and Hg; $L = C_{15}H_{10} N_2O_3$ were synthesized and identified by FT-IR, UV-Vis, ¹H-NMR (for Cd complex only) spectra, atomic absorption, chloride content along with molar conductivity and magnetic susceptibility. It was found that the ligand behaves as bidentate on complexation via (N) atom of imine group and (O) atom of carbonyl group of five member ring. The electronic spectral data and μ_{eff} values (Co, Cu) of the complexes exhibited octahedral geometry around Co^{II} , Cu^{II} , Cd^{II} , Hg^{II} metal ions. The results of antibacterial activity showed that only Hg(II) complex has high activity (35 mm) for staphylococcus aureas.

Keywords: Schiff base, isatin, 3-aminobenzoic acid, complexes.

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Introduction

1H-indol-2,3-dione (Isatin) is an important substrate, where it can be used for preparing of a wide variety of heterocyclic compounds, such as indoles and a raw material for drug synthesis. Isatin, due to its cis α -dicarbonyl moiety, is a good substrate for complexes formation, either alone or deprotonated, isatin was found in mammalian tissues[1]. A wide numbers of Schiff bases and their complexes have been studied for significant class of ligand in coordination chemistry[2]. The structure and bonding of Schiff bases complexes assist understanding the complexes. Most of the biological systems possess metal ions in unsymmetrical environments. These structures can be modified through condensation with aldehyde and ketones[3-5]. Isatin derivatives are reported to show many biological activities like antibacterial, antifungal, antiviral, anti-allergic and anti- inflammatory activities[6-10]. The complexes of Schiff bases of pyrazoline derivative have a diversity of applications in biological, analytical, pharmacological areas, and clinical[11]. In this paper we synthesized new Schiff base from reacted isatin and 3-aminobenzoic acid and its complexes with Co(II), Cu(II), Cd(II) and Hg(II).

Experimental

Chemicals

Regent grade isatin, 3- aminobenzoic acid were obtained from Fluka and used as received while metal salt (Cobalt chloride hexahydrate, Cupper chloride dihydrate, Cadmium chloride monohydrate and Mercury dichloride.

Instruments

The IR spectra for prepared compounds were recorded as KBr discs using a FT-IR tests can Shimadzu (FT-IR) 8300 series spectrophotometer in range (4000 – 400 cm⁻¹). Electronic spectra were measured in region (200 – 1000 nm) for 10^{-3} M solution in DMSO at 25 °C using Shimadzu 160 A spectrophotometer with 1.000 \neq 0.001 cm matched quartz cell. Elemental micro analysis were performed on a (C. H. N) analyzer from (Elemental micro analyses, Euro vector, model EA 3000 origin). ¹H-NMR spectra of prepared ligand (L) and cadmium complex were recorder in DMSO-d₆ using model: NMR Ready Pro 60 MHz, Origin: Canada and reported in ppm (S). Electrical conductivity measurements of the complexes were recorded at 25 °C for 10^{-3} M solution of the sample in DMSO using Eutech 150 conductivity meter. Magnetic susceptibility measurement were obtained by balance magnetic susceptibility model MSB - MKI. Metal contents of all complexes were determined by atomic absorption technique by using Shimadzu (AA 680 G). TG were obtained using apparatus type STA PT - 1000 L₁ NSEIS as the temperature range of 30 - 600 CO, the measurement obtained in central laboratory. In addition melting points were obtained using (Stuart Melting Point Apparatus) type Dig melt (MSRS).

Synthesis of ligand (L)

The ligand was synthesized by heating of 3- aminobenzoic acid (0.137 g, 0.001 mol) and isatin (0.147 g, 0.001 mol) in 50 ml (C₂H₅OH) on aqueous bath for (3 - 4) hrs in existence of drops of (glac. CH₃COOH)[12]. The resulting was yellow soiled which was filtered, washed with (C₂H₅OH) and recrystalized from ethanol, scheme (1), yield (90 %), m.p. (258 - 260) °C.

Synthesis Co(ll), Cu(ll), Cd(ll) and Hg(ll) complexes

(1 mmole) of metal chloride salt in (20 ml) of (C_2H_5OH) was mixed with (2 mmole) of new ligand (L) in (20 ml) of warm ethanol in round bottom flask, the pH of reaction mixture is regulated to (7.2). Resulting reaction mixture is refluxed (1-2) hrs on water bath. The solid complex precipitated was filtered off, washed with ethanol and dried under vacuum to give metal complex, scheme (2).

Antimicrobial study of biological activity

Antimicrobial activities of the ligand and the complexes have been carried out against the pathogenic bacteria like Escherichia coli and staphylococcus aureus using nutrient agar medium by disc diffusion method. The test solution was prepared in DMSO and soaked in filter paper of 5 mm diameter and 1 mm thickness. The discs were placed on the already seeded plates and incubated 37 °C for 24 hrs. The diameter (mm) of the inhibition zone around each disc was measured after 24 hrs. Streptomycin was used as standard[13].

Results and discussion

New ligand (L) and its complexes [Co(II), Cu(II), Cd(II) and Hg(II)] have been synthesized and identified by spectral measurements and elemental analytical data. They are stable in air. The elemental micro analysis results for the ligand and their metal complexes are in a good agreement with the calculated values. Table (1) shows the stoichometry of the metal (II) complexes and the molar conductance of 10^{-3} M of the complexes in DMSO solvent 25 °C measured.

¹H-NMR

¹H-NMR spectrum of ligand indicated signals at $\delta(6.18-7.74)$ ppm for aromatic protons (m, 7H, Ar-H) and at $\delta(7.86)$ ppm assign to (NH) of isatin moiety. A medium at $\delta(10.96)$ ppm assignable (s, 1H) belongs to (OH) of carboxylic group, Figure (1). In spectrum of Co(II) complex signals corresponding to Ar-H have been shifted to lower region. Signal of (NH) group shifted at $\delta(7.74)$ ppm to up field region [14], Figure (2). A new signal due to presence of water molecules at $\delta(3.3)$ ppm is observed in Cd(II) complex[15].

IR spectra

The IR spectrum of the free ligand (L) shows strong band at 3384 cm⁻¹ refers to v(O-H) stretching corresponding to carboxylic group, Table (2). The stretching frequency of carbonyl v(C=O) group at 1732 cm⁻¹ in spectrum of ligand shifted to lower frequency in spectra of all complexes, Figure (3). This shifting indicated the coordination between (O) atom of carbonyl group and metal ion[16]. The band at 1689 cm⁻¹ in spectrum of free ligand attributed to v(C=N) of imine group which was shifted to lower frequency region suggesting that this group was involved in coordination via (N) atom of imine group was involved in coordination[17,18]. Similarly a band v(N-H) stretching frequency at 3205 cm⁻¹ in ligand remains unaffected in complex, Figure (4) providing evidence for its non-coordination. A band at 3448 cm⁻¹, 3363 cm⁻¹, 3338 cm⁻¹, 3240 cm⁻¹ in Cd(II), Co(II), Cu(II)[19] metal complexes refers to overlap band of hydrated water molecules and v(O-H) of carboxyl group. The appearance of new band in the spectra of Cd(II), Co(II), Cu(II) and Hg(II) metal complexes at range (567, 677, 606, 594) cm⁻¹ due to and (487, 577, 471, 484) cm⁻¹ due to v(M-N)[20] and v(M-O)[21] respectively.

Electronic spectra and magnetic moment

The electronic spectra of the new Schiff base ligand (L), exhibited two peaks around 298 nm, 430 nm related to electronic transition for π - π *, n- π *[22,23]. The electronic spectrum of Co(II) complex, Figures (5,6), Table (3) showed two peaks at 297 nm, 412 nm due to ligand field. Other three peaks at 613 nm 681 nm and 816 nm were found to be caused by (d-d) electronic transition type ${}^{4}T_{1g}(F) \rightarrow {}^{4}T_{1g}(p)$, ${}^{4}T_{1g}(F) \rightarrow {}^{4}A_{2g}(F)$ and ${}^{4}T_{1g}(F) \rightarrow {}^{4}T_{2g}(F)$ respectively[24]. The μ_{eff} value (4.86) B. M refers to high spin octahedral geometry around Co(II).

The spectrum of Cu(II) complex gave absorption peak at 300 nm, 406 nm due to ligand field. The μ_{eff} value (1.72) B.M was a good evidence for octahedral formula. The peak at 700 nm was caused by electronic transition[25] ${}^{2}E_{g} \rightarrow {}^{2}T_{2g}$. The (d-d) electronic transition of Co(II) and Cu(II) complexes were a good agreement with octahedral geometry around these ions[26]. The electronic spectra of Cd(II) and Hg(II) complexes show two absorption peaks at (299 nm and 295 nm) and at (416 nm) due to intra-ligand this indicates no (d-d) electronic transition happened (d¹⁰-system) in visible region. These complexes are diamagnetic as expected, that is a good result for Cd(II) and Hg(II) octahedral geometry around this ions[27].

Chloride content

The chloride content measurement for all complexes, Table (1) gave approximated values for theoretical values.

Antibacterial activity

Antibacterial activity of ligand and Co, Cu, Cd and Hg complexes against one strain of gram (+ ve) bacteria (Staphylococcus aureus) and gram (-ve) bacteria (Escherichia coli) were recorded. The results indicate only Hg(II) complex have a high activity (35 mm) for staphylococcus aureus. This can be attributed to an Hg(II) complex solution which have ability to solvate the fit layer of bacteria cell and destroy it, Figure (7), Table (4).

Thermal analysis studies

The TGA thermal analysis curves for $[Co(L)_2Cl_2]$.H₂O and $[Cd(L)_2Cl_2]$.H₂O data are listed in Table (5), Figure (8). The Co(II) complex decomposes in two steps. The first stage is the loss (H₂O + Cl + C₈H₅N₂O) with mass losses of 15.7539 % (CaCl= 16.1554%) within temperature range of (= 392.5 °C). The second step involves the loss of the organic fraction, (C₇H₆O₂ + H₂O + NO + Cl₂ + NH + COO + CO₂H + N₂ + C₂H₆ + C₈H₅N₂O) within the temperature range of (392.5-597.5) °C with mass losses of 49.7619 % (CaCl= 53.838 %).

The Cd(II) complex decomposes in three steps. Stage one in the loss Cl_2 within the temperature range of (= 170 °C), within mass losses of 5.405 % (CaC=5.1383 %). The two stages is the loss (Cl₂) within the temperature range of (170-330) °C within mass losses of 5.405 % (CaCl= 5.20351 %). The final weight of the compound observed at 42.439 % (CaCl= 44.9305 %) within temperature range of (330-595) °C.

The Cu(II) complex decomposes in three steps. The first stage is the loss (Cu) with mass losses of (CaCl= 4721 %) within a temperature range of (=215 °C) the second stage is loss (CuO + H₂O + Cl₂ + N₂ + C₂H₆ + C₈H₅N₂O) within temperature range of (215-365) °C with mass losses of 42.8232 % (CaCl= 42.2992 %), the final weight of the compound observed at 11.9019 % (CaCl= 11.6367 %) within temperature range of (365-595) °C.

The Hg(II) complexes decomposes in three steps. Stage one is the loss (H₂O) within the temperature range of (= 100 °C) with mass losses of 1.2847 % (CaCl= 1.50742 %). The

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stage two is the loss (HgO + COOH + NO + $C_6H_6 + C_8H_5N_2O + Cl_2 + H_2O + NH + Hg + C_7H_5O_2$) within the temperature range of (100-347.5) °C with mass losses of 67.0949 % (CaCl= 79.023 %). The final is the losses ($C_7H_6O_2$) within the temperature range of (347.5-592.5) °C with mass losses of 15.5315 % (CaCl= 16.8217 %). The difference in the calculation in observe of the residue weight may be related to the sublimation up on thermal decomposition[28].

Conclusion

In this research, we have reported the synthesis of new Schiff base ligand achieved from the reaction of isatin and 3- aminobenzoic acid and then prepared its complexes with [Co(II), Cu(II), Cd(II) and Hg(II)]. The structure of the ligand and its prepared complexes were confirmed via FT-IR, U.V-Visible, ¹H-NMR, elemental micro analysis, thermal analysis and chloride content. The ligand behaves as bidentate ligand with metal ions through its (N) atom of azomethine group and (O) atom of carbonyl group of five member ring, forming octahedral geometry around [Co(II), Cu(II), Cd(II) and Hg(II)].

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Compounds	M. wt	Yield%	Colure	M. p. °C	Found % (Calculated %)				
					С	Н	N	Metal	Cl
$(L) = C_{15} N_2 O_3 H_{10}$	266	60	Yellow	260 - 258	67.669 (67.391)	3.759 (4.102)	10.526 (10.029)	-	-
[Co(L) ₂ Cl ₂].H ₂ O	680	70	Green	>300	52.941 (51.531)	3.235 (2.224)	8.235 (7.068)	8.676 (7.818)	10.441 (9.232)
[Cu(L) ₂ Cl ₂].H ₂ O	684	65	Reddish brown	>300	52.593 (50.310)	3.214 (2.021)	8.181 (7.131)	9.276 (8.341)	10.372 (9.462)
[Cd(L) ₂ Cl ₂].H ₂ O	733.5	76	Yellow	>300	49.079 (48.022)	2.999 (1.481)	7.934 (6.089)	15.337 (14.013)	9.679 (8.614)
[Hg(L) ₂ Cl ₂].H ₂ O	821	71	Yellow	>300	43.848 (42.781)	2.679 (1.031)	6.820 (5.793)	24.360 (23.679)	8.647 (7.375)

Table (1): Analytical and physical data of ligand (L) and its complexes

Table (2): Infrared spectral data of ligand and their Co(II), Cu(II), Cd(II) and Hg(II) metal complexes

Compounds	v(OH), v(H ₂ O)	v(C=O) amide	v(C=C) arom.	v(C=N)	v(N-H)	v(M-N)	v(M-O)
L	3384	1732	1583,1458	1689	3205	498	561
[Co(L) ₂ Cl ₂].H ₂ O	3363	1714	1581,1446	1612	3072	677	577
[Cu(L) ₂ Cl ₂].H ₂ O	3240	1701	1560,1516	1595	3075	606	471
[Cd(L) ₂ Cl ₂].H ₂ O	3338	1712	1595,1556	1666	3066	567	486
[Hg(L) ₂ Cl ₂].H ₂ O	3448	1724	1547,1452	1670	3070	594	484

Compounds	λ (nm)	$v (cm^{-1})$	ε (L. mol ⁻¹ .cm ⁻¹)	Transition	Geometrical
					$\mu_{\rm eff}$ B.M
L	298	33557	2486	π -π*	
	430	23256	2394	n -π*	
[Co(L) ₂ Cl ₂].H ₂ O	297	33670	2436	L.F	Oh
	412	24272	1176	L.F	4.86
	613	16313	66	${}^{4}T_{1g}(F) \rightarrow {}^{4}T_{1g}(F)$	
	681	14684	71	${}^{4}T_{1g}(F) \rightarrow {}^{4}A_{2g}(F)$	
	816	12255	6	${}^{4}T_{1g}(F) \rightarrow {}^{4}T_{2g}(F)$	
$[Cu(L)_2Cl_2].H_2O$	300	33333	1918	L.F	Distorted Oh
	406	24630	482	L.F	
	700	14286	62	${}^{2}E_{g} \rightarrow {}^{2}T_{2g}$	1.72
$[Cd(L)_2Cl_2].H_2O$	299	33445	2269	L.F	
	416	24038	1297	L.F	Oh
$[Hg(L)_2Cl_2].H_2O$	295	33898	2191	L.F	
	416	24038	1028	L.F	Oh

Table (3): Electronic spectral data of ligand and metal complexes

Table (4): The inhibition zone of ligand and Co(II), Cu(II), Cd(II) and Hg(II) complexes

Compound	Escherichia coli	Staphylococcus aureus	
L	-	-	
$[Co(L)_2Cl_2].H_2O$	-	-	
$[Cu(L)_2Cl_2].H_2O$	-	-	
$[Cd(L)_2Cl_2].H_2O$	-	24	
$[Hg(L)_2Cl_2].H_2O$	28	35	
Control	-	-	

Table (5): Thermal analysis data of Co(II), Cu(II), Cd(II) and Hg(II) complexes

Complexes	Mass loss % observed (calculated)	Temperature °C	
	16.1554 (15.7539)	R.T - 392	
$[Co(L)_2Cl_2].H_2O$	53.838 (49.7619)	392.5 - 597.5	
	5.47215 (5.0217)	R.T - 215	
$[Cu(L)_2Cl_2].H_2O$	42.2992 (42.823)	215-365	
	11.6367 (11.9019)	365-595	
	5.13834 (5.405)	R.T - 170	
$[Cd(L)_2Cl_2].H_2O$	5.20351 (5.405)	170-330	
	44.9405 (42.439)	330-595	
	1.50742 (1.2847)	R.T - 100	
$[Hg(L)_2Cl_2].H_2O$	69.623 (67.0949)	100-347.5	
	16.8217 (15.5315)	347.5-592.5	





Figure (2): ¹H-NMR spectrum of Cd(II) complex



Figure (3): FT-IR spectrum of ligand (L)



Figure (4): FT-IR spectrum of Cd(II) complex



Figure (5): Electronic spectrum of ligand (L)



Figure (6): Electronic spectrum of Co(II) complex





Figure (8): Thermal analysis of Co(II), Cu(II), Cd(II) and Hg(ll) complexes



Scheme (1): Synthesis route of the ligand (L)



Scheme (2): Synthesis route of Co(II), Cu(II), Cd(II) and Hg(II) complexes