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Molybdenum(VI) complexes of N, N′-bis isatin diamine Schiff base: Synthesis and characterization

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ABSTRACT

Schiff base ligand of N, N′-bis isatin diamine was synthesized from various diamine and isatin through condensation reaction in 1:2 molar ratio. Mo(VI) complexes of isatin-diamines were synthesized by the reaction between the sodium molybdate dehydrate and N,N′ bis isatin diamines with the reflux method. The synthesized ligand and complexes were characterized using spectroscopic techniques. The FT-IR band of azomethine group at 1600- 1610 cm^{-1} indicated the formation of the ligand. The UV-vis spectra clearly indicated that the prepared complex is a genuine Mo(VI) with d^0 electronic configuration. The formation of a coordination bond was revealed by the downfield chemical shift of the complex in the NMR data as compared to the ligand. The UV results were corroborated by molar conductance data, which demonstrated their non-electrolytic character. The experimental evidence primarily supported the synthesis of both ligands and corresponding Mo(VI) complexes.

Keywords: Schiff base ligand; N, N′-bis isatin diamine; Mo(VI) complex; Imine group

Introduction

Many medications have enhanced pharmacological and toxicological characteristics in the form of Schiff base ligands that comprise metal complexes. Schiff bases are useful C=N (imine) containing compounds possesses comprehensive bioactivity and metal Schiff base complexes showed to a certain extent of antibacterial, antifungal, antitumor and anti-inflammatory activity (Gupta *et al*. 1998). A Schiff base acts as a flexi-dentate ligand and generally co-ordinates through the ‘O’ atom of the de-protonated phenolic and N atom of azomethine group (Assefa *et al.* 2009). Schiff base has often been used as a chelating ligand in metal-complex chemistry (Shibuya *et al*. 2008; Roth *et al*. 2006). The presence of supporting and stabilizing -OH group adjacent to -HC=N- group enhances the chelating efficiency of this type of ligands. Transition metal complex containing Schiff base ligand has the interest for many years (Chang *et al*. 1998).

Over the past few years, there have been many reports on their applications of metal complexes in catalysis. Metal complex compounds [Co(II), Cu(II) and Zn(II) ions] of Schiff base play a crucial role in the advancement of co-ordination chemistry. The attractive DNA binding and cleavage properties of transition metal-complexes have raised the inquisitiveness of the scientific community.

Schiff base ligand of salicylaldehyde and 2, 3-diaminopyridine using chromium, iron and cobalt have been synthesized in absolute ethanol solvent with reflux method (Zare and Ataeinia, 2012). Due to the use of thiocyanate bridging in this study, fairly novel structures were produced. Octahedral structured Cobalt(II) complex was synthesized via condensation of salicylaldehyde, o-phenylenediamine, and isatin/acetyl acetone /naphthaldehyde using a tetradentate N_2O_2 type Schiff base ligand (Nagajothi *et al*. 2012). Sodium pyruvalidene hydrazine [NaHL], a novel ligand, and its complexes with Zn(II), Cu(II), Ni(II) and Co(II) were investigated (Yousif, 2008). For the complexes under study, a distorted tetrahedral geometric structure is suggested. Cobalt(II) and manganese(II) Schiff base complexes have been explored (Parrey and Hashmi, 2015).

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Using hydrogen peroxide as an oxidant, the catalytic activity of both complexes was examined in cyclohexane and benzyl alcohol. The cobalt(II) complex exhibits greater activity in this study than the manganese(II) complex does. The synthetic procedure of Cd(II), Cu(II), Ni(II) and Zn(II) complexes of 2-thiophenecarboxaldehyde with 1,2-diamino propane and 2-amino thiophenol have already been reported (Uddin *et al*. 2014). The synthesised complexes were claimed to have an octahedral structure based on the findings of magnetic susceptibility and electronic spectra. Values for conductivity point to the non-electrolytic behaviour of the synthesised compounds. It has been proposed that $[ML_2]$ and $[ML_1Cl_2]$ represent the stoichiometry of the prepared complexes.

Co(III), Mn(II), Fe(III), Ni(II) and Cu(II) complexes of a new Schiff base ligand, nicotinohydrazide was prepared. Through the quinolone carbonyl, azomethine nitrogen, and amide oxygen, the ligand serves as an ONO tridentate coordinating to the metal in each of the prepared complexes (Kumbalpuri *et al*. 2015). Molybdenum coordination compounds have a significant role in both plant and animal metabolism. Nitrogenase, aldehyde oxidase, nitrate reductase, xanthin oxidase, and other enzymes contain molybdenum as a component. A number of molybdenum representative complexes have been synthesized and explored to better understand the mechanism underlying enzymetic activity (Syamal and Maurya, 1985.

Despite numerous reports, to our knowledge there have been no reports on Schiff bases based on complexes of N, N′-bis isatin diamine, although they are biologically important. In this study, we therefore, aim at synthesizing and characterizing molybdenum(VI) complexes of N, N′-bis isatin diamine Schiff bases.

Materials and methods

Chemicals

Methanol, ethanol, diamines (1,6-diamino hexane, 1,3-diamino propane and 1,2-diamino ethane) were obtained from Scharlau Spain; isatin from BDH Chemicals Ltd., UK; sodium molybdate dehydrate from Germany, DMF and DMSO from m/s. E. Merk, Germany. Without any additional purification, all of the analytical grade chemicals were employed in the synthesis.

Preparation of N, N′-bis isatin diamine Schiff base ligand

The Schiff base ligands were synthesized by the reaction of different diamine with isatin by the published procedure (Gupta and Gupta, 1983; Chowdhury *et al*. 2006). Isatin (2 mmol) in 25 cm^3 methanol was introduced in a 250 cm^3 round bottom flask and then it was placed in a hot plate magnetic stirrer. Isatin was dissolved completely and allowed to cool. 1,2-diamino ethane (1 mmol) was introduced in the prepared mixture taken in a 250 cm^3 round bottom flask. The mixture was refluxed until the desired product obtained. The resulting precipitate (N, N'-bis isatin 1,2-diamino ethane; L1) was filtered, washed with methanol, and dried in a silica gel containing vacuum desiccator. N, N′-bis isatin-1,3-diamino propane (L^2), N, N′-bis isatin-1,6-diamino hexane (L^3) Schiff base ligands were also prepared following similar method.

Ligand

^{1}H NMR (solvent: DMSO, frequency: 400 MHz) δ 2.11-2.32 (m, 2H), 3.28-3.71 (m, 4H), 6.82-6.93 (m, 2H), 7.00-7.08 (m, 2H), 7.38-7.51 (m, 2H), 7.84-7.91 (m, 2H), 10.81 (s, 2H).

Preparation of molybdenum complexes with N, N′-bis isatin diamine

A solution of sodium molybdate dehydrate ($Na_2MoO_4.2H_2O$) (1 mmol) in distilled water (30 cm^3) was mixed with the solution of L^1 (1 mmol) in acidic condition (pH= 1.90). The resulting mixture was allowed to reflux on a magnetic stirrer for desired product. An orange precipitate was formed immediately. The colored precipitate was separated by filtration, washed using distilled water, dried and stored in a vacuum desiccator. Similar procedure was used for the preparation of molybdenum complex of N, N′-bis isatin-1,3-diamino propane and N, N′-bis isatin-1,6-diamino hexane.

Molybdenum complex

^{1}H NMR (solvent: DMSO, frequency: 400 MHz) δ 2.50-2.51 (m, 2H), 3.48 (t, 4H), 6.91-7.05 (m, 2H), 7.07-7.09 (d, 2H), 7.50-7.51 (d, 2H), 7.57-7.61 (m, 2H), 11.04 (s, 2H)

Analysis

Melting point apparatus, model KRUSS, KSPINN was used to obtain the melting points of the ligand and complexes. Elementar (Vario-EL cube) was used for the elemental analysis of the sample and metal analysis was done by atomic absorption spectrophotometer (model: AA-7000, Shimadzu, JAPAN) equipped with GFA-7000. Conductance of the samples was measured by CTS-406K model conductometer using dimethylformamide as solvent. The FTIR spectrum was taken by FTIR 8400 S Shimadzu spectrophotometer using KBr within wavenumber 4000- 400 cm^{-1} at spectrum resolution 2 cm^{-1} and 50 scanning times. Nuclear magnetic resonance spectrometer (model: AscendTM 400 MHz, Bruker, Switzerland) was used to record ^{1}H-NMR spectra. In the ^{1}H-NMR analysis, DMSO-d6 was employed as a solvent. The electronic spectra of the prepared samples in DMF were collected using a Shimadzu UV 1800 model ultra violet-visible spectrophotometer equipped with a 1 cm cell. Physical properties of the ligands and complexes are listed in Table I.

From Table I, it is observed that the experimental values for Mo, C, N, and H of the prepared ligands and complexes were almost similar to the calculated values.

Table I. Physical parameters and elemental analysis of the prepared N, N′-bis isatindiamine ligands and molybdenum(VI) complexes

Name of the samples	Colour	Yield (%)	m.p. ^{0}C	Mo%	N%	C%	H%	Molar conductance Am ohm^{-1} cm^2 mol^{-1} (in DMF)
L^1	Deep Yellow	70	206		17.447 (17.61)	66.67 (67.92)	4.441 (4.4)	0
L^2	Yellow	72	190		16.777 (16.87)	69.46 (68.67)	4.887 (4.82)	0
L^3	Yellow	72	182		14.872 (14.97)	69.46 (70.59)	5.951 (5.88)	0
MoO_2L^1	Orange	74	>192	20.28 (21.56)	8.557 (12.58)	40.39 (48.54)	2.98 (3.15)	0
MoO_2L^2	Light orange	75	>178	19.08 (20.86)	8.320 (12.17)	40.70 (49.57)	3.207 (3.48)	0
MoO_2L^3	Orange	75	>170	18.82 (19.11)	8.276 (11.16)	42.89 (52.59)	3.883 (4.38)	0

Calculated values are given in parentheses

Results and discussion

Elemental analysis

Elemental analysis of the synthesized ligands and their molybdenum(VI) complexes are summarized in Table I. The analytical data obtained for C, H, N elements for ligands and their complexes are almost similar to the calculated values. It indicates that the prepared ligand and complexes may have almost similar elemental structure to the expected one.

Conductance measurements

The complexes are insoluble in water solvent and soluble in dimethylsulphoxide, dimethylformamide solvents and slightly soluble in MeOH and EtOH. Molar conductance values were measured and are listed in Table I. The molar conductance values of the synthesized N, N′-bis isatin diamine ligands and their resultant molybdenum(VI) complexes were found zero. The zero molar conductivity data of the schiff base ligands and subsequent complexes indicate their non-electrolytic nature. Thus the observed zero molar conductance can be treated as good evidences in support of the Mo(VI), d^0 cation, and basic nature of the coordinated Schiff base ligands.

FTIR spectral study

The FTIR spectral data help to determine the presence and examine the effect of coordination of the ligands and assume structural properties of the complexes (Zare and Ataeinia 2012; Demir *et al*. 2008). The ligands L^1, L^2, L^3 and complexes MoO_2L^1, MoO_2L^2 and MoO_2L^3 were studied ($\upsilon_{C=N}$, υ_{C-N}, υ_{C-O-H}, $\upsilon_{C=O}$, υ_{O-H}, υ_{N-H} functional group regions) using FT-IR spectroscopy and plotted in Table II.

The spectra consist azomethine group (-C=N- group) in the region 1625.00-1582.00 cm^{-1} (Singh *et al*. 2012). The vibrational peak at 1610.56 cm^{-1},1608.63 cm^{-1} and 1610.56 cm^{-1} reveals azomethine group (>C=N-) for N, N′-bis isatin diamine Schiff base ligands of L^1, L^2 and L^3 , respectively. The presence of FTIR band at 1614.42 cm^{-1},1618.28 cm^{-1} and 1616.35 cm^{-1} for MoO_2L^1, MoO_2L^2 and MoO_2L^3, respectively indicates the existence of ligands' >C=N- group in the complexes.

The band for the C-N stretching frequency literally assigned in the region 1250-1020 cm^{-1} (Uddin *et al*. 2012). In the present work, the existence of peak at 1203.58 cm^{-1}, 1203.58 cm^{-1} and 1209.37 cm^{-1} showed the existence of the C-N stretching frequency in the ligands L^1, L^2 ,L^3, respectively and 1199.72 cm^{-1}, 1201.65cm^{-1} and 1201.65 cm^{-1} for complexes MoO_2L^1, MoO_2L^2 and MoO_2L^3, respectively.

The band for the C-O-H bending frequency is reported in the region 1321-1371 cm^{-1} (Xavier and Srividhya, 2014). Bands at 1340.53 cm^{-1}, 1334.74 cm^{-1} and 1336.67 cm^{-1} correspond to C-O-H bending frequency for ligands L^1, L^2 and L^3, respectively. It indicates the keto-enol tautomerism in the prepared ligand, which was totally absent in the complexes.

It is reported that the bands for C=O stretching frequency are in the region 1730-1750 cm^{-1} (Uddin *et al*. 2012). Strong sharp bands are observed at 1741.72 cm^{-1}, 1751.36 cm^{-1} and 1749.44 cm^{-1} are assigned to υC=O of isatin ligands L^1, L^2 and L^3, and 1732.28 cm^{-1}, 1732.28 cm^{-1} and 1728.22 cm^{-1} for complexes MoO_2L^1, MoO_2L^2 and MoO_2L^3, respectively.

The band for the O-H stretching frequency is reported in the region 3200-3500 cm^{-1} (Sarker *et al*. 2019). The bands at 3441.01, 3448.72 and 3454.51 cm^{-1} indicate the presence of O-H stretching frequency for ligands L^1, L^2 and L^3 and 3444.87, 3448.72 and 3446.79 cm^{-1} for respective aquatic complexes MoO_2L^1. H_2O, MoO_2L^2. H_2O and MoO_2L^3. H_2O, respectively.

The bands in the region 3150-3300 cm^{-1} are assigned due to the N-H stretching mode in the free ligands (Pickard and Polly, 1954). Absorption band are observed at 3267.41, 3234.62 and 3244.27 cm^{-1} relating to N-H absorption frequencies in the ligand L^1, L^2 and L^3, and 3190.26, 3199.91 and 3192.91 cm^{-1} relating to N-H absorption frequencies for complex MoO_2L^1, MoO_2L^2 and MoO_2L^3, respectively.

The band at lower than 950 cm^{-1} is assigned to the asymmetric M=O stretch and higher than 950 cm^{-1} is assigned to the symmetric M=O stretch (Nath *et al*. 2009; Al-Ajlouni *et al*. 2009) For complex (MoO_2L^1), the absorption band was at 945.12 cm^{-1} for asymmetric M=O stretch and 1093.64 cm^{-1} for symmetric M=O stretch. For complexes, MoO_2L^2 and

Table II. The obtained infrared data for ligands and prepared complexes

Complexes	υC=N cm^{-1}	υC-N cm^{-1}	υC-O-H cm^{-1}	υC=O cm^{-1}	υN-H cm^{-1}	υO-H cm^{-1}	υM=O cm^{-1}	υM-N cm^{-1}	υM-O cm^{-1}
L^1	1610.56	1203.58	1340.53	1741.72	3267.41	3441.01			
L^2	1608.63	1203.58	1334.74	1751.36	3234.62	3448.72			
L^3	1610.56	1209.37	1336.67	1749.44	3244.27	3454.51			
MoO_2L^1	1614.42	1199.72		1732.28	3190.26		945.12(as) 1093.64(s)	462.92	667.37
MoO_2L^2	1618.28	1201.65		1732.28	3199.91		937.40(as) 1095.37(s)	522.71	657.73
MoO_2L^3	1616.35	1201.65		1728.22	3192.91		948.98(as) 1093.64(s)	555.50	665.44

'as'= asymmetric 's'= symmetric

MoO_2L^3 absorption bands were observed at 937.40 and 948.98 cm^{-1} for asymmetric M=O stretch and 1095.37 and 1093.64 cm^{-1} for symmetric M=O stretch, respectively. The band for the M-N stretching frequency is reported in the region 506-570 cm^{-1} (West *et al*. 1991). The band at 462.92 cm^{-1}, 522.71 cm^{-1}, 555.50 cm^{-1} demonstrated the existence of the M-N stretching frequency for complexes MoO_2L^1, MoO_2L^2 and MoO_2L^3, respectively. The band for the M-O stretching frequency is reported in the region 450-750 cm^{-1} (Ade *et al*. 2012. The bands at 667.37 cm^{-1}, 657.73 cm^{-1}, 665.44 cm^{-1} indicate the presence of the M-O stretching frequency for complexes MoO_2L^1, MoO_2L^2 and MoO_2L^3, respectively.

Electronic (UV-Visible) Spectra

Electronic spectral data of the synthesized ligands and complexes are shown in Table III. The ligands were soluble in DMF. Most of the complexes were soluble in DMF but insoluble in MeOH, EtOH, and CCl_4. Therefore, DMF was used to record the electronic spectral data for the prepared ligand and their complexes.

Beyond 500 nm, optical density values are extremely low and there is no chance of any d-d transitions over the visible region for d^0 system (Brown and MacPherson, 1970). Electronic spectral band of untainted d-d region are not anticipated in the molybdenum(VI) complexes containing $4d^0$ configuration. Accordingly, the electronic spectrum data proved that the complexes were actually containing molybdenum(VI). But the only way the complexes were coloured was via the charge-transfer that they absorbed from the ultraviolet. In the ligand spectrum, bands were observed in the region 250-265 nm due to the π-π* electronic transitions within the aromatic ring and showed a small peak in the spectra of molybdenum complexes. As a result of the coordination of azomethine nitrogen to the molybdenum atom, a band in the 290–300 nm range is caused by the n-π* electronic transitions inside the >C=N– chromophore (Kalia *et al*. 2007). The polarisation within the >C=N- chromophore brought about by the molybdenum ligand electronic interaction resulted in a slight shift of the absorption band to the higher energy region in the electronic spectra of molybdenum complexes.

^{1}H NMR spectrum of N, N'-bis isatin1, 3-diamino propane Schiff base ligand and its Mo complexes

^{1}H NMR spectra were recorded only for L^2 and MoO_2 L^2 complex. The ^{1}H NMR spectra of the compounds were run in DMSO at room temperature with TMS as the internal standard. The characteristic resonance peaks in the obtained ^{1}H NMR spectra of the selected ligand and complex are given in Table IV. The protons of phenyl groups both in ligand and complex exhibited chemical shifts in the range 6.82-7.91 ppm indicating the presence of aromatic nucleus in both the ligand and complex (Parrey and Hashmi, 2015). However the spectral analysis also revealed the presence of eight protons for two benzene rings of isatin moiety both in ligand and complex. The N-H of both ligand and complex gives ^{1}H NMR peaks at 10.81 ppm and 11.05 ppm, respectively. This downfield chemical shifting of the complex compared to the ligand indicated the formation of coordination bond in the Mo-complex existing in lactam-lactim equilibrium which can be supported by the FTIR result (de *et al*. 2012; Jiang *et al*. 2017; Khalifa and Hassaan, 1995). We have also noticed two different ^{1}H NMR peaks from methylene groups ($-CH_2-$) both in ligand and complex with some unexpected peaks resulting from impure reagents and solvents. It is notable that the chemical shift of DMSO-*d6* in ^{1}H NMR is at 2.50 ppm (Khalajia and Das, 2011).

Table III. Electronic spectra of the prepared ligands and complexes

c	Medium	Electronic spectral bands in (nm)
L^1	DMF	258.62(sh), 260.44(sh), 296.84
L^2	DMF	251.76(sh), 260.44(sh), 295.44
L^3	DMF	251.34, 293.2(sh), 395.26
MoO_2L^1	DMF	270.74, 291.38(sh), 422.52
MoO_2L^2	DMF	255.56, 294.46(sh), 411.08
MoO_2L^3	DMF	255.56, 296.84(sh), 417.24

‘sh’=shoulder

Table IV. ^{1}H NMR data for N, N′-bis isatin(^{1}H-indole-2,3-dione) 1, 3-diamino propane Schiff base ligand and its Mo-complexes

Compounds	NMR signals for $–CH_2$- δppm	NMR signals for –N-H, δppm	NMR signal for phenyl protons, δppm
L_2	2.11-2.32 ppm, 3.28-3.71 ppm	10.81 ppm	6.82-7.91 ppm
MoO_2L_2	2.50-2.51 ppm 3.48 ppm	11.04 ppm	6.91-7.61 ppm

Conclusion

New molybdenum(VI) complexes of isatin based Schiff bases with three aliphatic diamines were synthesized successfully. The physical and analytical results of the studied complexes reveals 1:1 stoichiometry of metal to ligand. By measuring conductivity, melting point and interpreting the UV-visible, FTIR along with ^{1}H NMR spectra, the synthesized ligands and complexes were characterized. Azomethine group was present in the prepared ligands and molybdenum complexes. C-O-H bending frequency indicated the keto-enol tautomerism in the ligand, which was totally absent in the prepared complexes. The bands for the Mo-N and Mo-O stretching frequency indicated the formation of the Mo-N and Mo-0 coordination bonds in the complexes. The electronic spectra provide evidence that the complexes contain molybdenum with d^0 electronic configuration. Therefore, this result clearly endorsed the complex as molybdenum(VI). In NMR spectra, the downfield chemical shift for the NH of isatin moiety in the complex with respect to ligand indicated the possibility of the formation of coordination bond in the complex. Elemental data (Mo, C, N, and H) for both ligands and complexes concurred with the predicted values. Complexes have been found to be non-electrolytes based on their conductivity (molar conductance) responses. Nevertheless, it is difficult to propose the particular geometry of the synthesized Mo(VI) complexes without crystal structure study. Consequently, additional research and analysis are required to ascertain the precise geometry of the produced compounds.

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