Original Research Article

Interaction of schiff base derived from 4-chloro-5-sulfamoyl-2',6'-salicyloxylidide with Cu(II) and Mn(II)

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Abstract

A bidentate sulfonamide Schiff base ligand have been prepared by refluxing 4-chloro-5-sulfamoyl-2',6'-salicyloxylidide (Xipamide) and salicaldehyde in methanol water (1:1) for 4 to 5 hours. The synthesized Schiff base was used as a chelating agent for Cu (II) and Mn (II) complexes. The physico-chemical properties were characterized by elemental analysis, molar conductance and by magnetic susceptibility method. Molar conductance values supported the non-ionic nature of the complexes. The synthesized Schiff base and its metal complexes were also analyzed and characterized by IR electronic spectra studies. The spectral studies confirm the bidentate nature of the ligand. The microbiological studies of Schiff base and their metal complexes were studied against gram +ve and gram –ve bacteria by filter paper disc method. Increased activities of metal complexes as compared to the ligand and parent drug suggest that complexation increases the antibacterial activity.

Key Words: Xipamide, ligand, bidentate, Schiff base, elemental analysis, molar conductance.

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INTRODUCTION

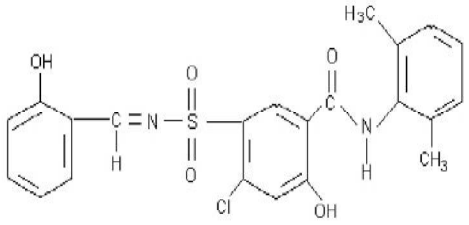
Chemically Schiff base is a functional group called azomethine group, that contains a carbon-nitrogen double bond (CH=N-). The chemistry of azomethine has occupied a place of considerable importance because of their well-established biological activities[1,2]. Schiff bases are an important class of ligand, which give excellent result on coordination with transition metal ions. After chelation with biologically suitable metal ions, Schiff base gains much more importance due to their enhanced biological and industrial applicationa[3,4]. Diuretics are described as medicine that help to reduce the amount of water in the body or include all kinds of drugs that promote the formation of urine by kidney. Schiff base derived from diuretic drugs have gained much importance and interest due to their useful applications in various fields[5,6]. The study of transition metal complexes is of great interest due to their biological activities. Transition metal complexes with Schiff base ligands are reported by many workers[7,8]. In view of the interest involved in these compounds and continuation of our previous work[9-12] in this field, Cu (II) and Mn (II) complexes have been synthesized with Schiff base ligand (xipamide-salicylaldimine) derived from the condensation of salicylaldehyde and 5-aminosulfonyl-4-chloro-N-2,6-dimethyl phenyl-2- hydroxybenzamide(xipamide-salicylaldimine) derived from 5-aminosulfonyl-4-chloro-N-2,6-dimethyl phenyl-2- hydroxybenzamide(Xipamide), a diuretic drug. The solid complexes have been synthesized and studied by elemental analyses, spectroscopic characterization and biological studies.

MATERIALS AND METHODS

All the chemicals and solvents used were analytical reagent grade and were used without any purification. Xipamide drug was provided by Dishman Pharmaceuticals which was used as such for the synthesis of ligand. Elemental analyses were carried out on model 240 Perkin elemental analyzer at CDRI, Lucknow. Metal

contents were determined gravimetrically using standard methods[13].Conductivity measurements were made in anhydrous DMF on a Systronics model 305 (India) Conductivity Bridge. Magnetic susceptibility measurements of the complexes in the solid state were determined by vibrating sample magnetometer at Centre for Advance Technology, Indore at room temperature. The electronic spectra of the metal complexes in DMF were recorded on a Perkin-Elmer UV Win Lab Spectrophotometer at School of Studies in Chemistry and Biochemistry, Vikram University, Ujjain. The infrared spectra of the ligand and complexes were recorded in KBr pellets using Perkin-Elmer FT- IR spectrophotometer in the range of 4000-400 cm^{-1} at School of Studies in Chemistry and Biochemistry, Vikram University, Ujjain. The melting points of the ligand and complexes were recorded in open capillaries on a capillary melting point apparatus. The biological activities were tested in vitro and in vivo. In vitro tests were conducted for growth inhibitory against *Escherichia coli* and *Bacillus subtilis* using Streptomycin as standard by filter paper disc method[14].

Synthesis of the ligand (xipamide-salicylaldimine): The Schiff base is synthesized by using equimolar (0.01M) solutions of pure drug (0.22 gm) and salicylaldehyde (0.14ml) separately dissolved in methanol-water mixture (1:1) and refluxed for four hours and kept for a day. Peach colour crystals of xipamide- salicylaldimineSchiff base were formed in the reaction mixture which were filtered and washed thoroughly with 50% methanol-water mixture, dried over vacuum and weighed. Melting point of Schiff base was recorded. The structure of the synthesized ligand is shown in Fig.1



Xipamide–Salicylaldimine Schiff Base

Figure 1: Structure of Ligand

Synthesis of complexes: For the synthesis of complexes, 0.006 M ligand solution was prepared in 50% acetone-water solvent and refluxed for four hours with 0.003 M solution of metal salts separately. The refluxed solutions were kept for some days. Solid crystalline compounds appeared in the solution, which were filtered, washed with 50% acetone-water mixture, dried and weighed. Melting points of the complexes were recorded.

RESULTS AND DISCUSSION

All these complexes are analyzed for 1:2 stoichiometry of the type ML_2. The chelating ligand and its metal complexes were insoluble in water bur fairly soluble in DMSO and DMF. The elemental analyses data of the chelating ligand and its metal complexes were in good agreement with calculated value. It has been found that all the complexes are non-hygroscopic and stable at room temperature. The molar conductance values of synthesized complexes were and their too low value account for their electrolytic behavior[15,16]. The analytical data of the complexes and their molar conductance values are given in Table 1.

Table 1: Analytical data and molar conductance values for ligand and metal complexes

Sl. no.	Ligand/ Complexes	Elemental analysis (%): Found (Calcd.)					M.pt. (°C)	Color	Molar Conductance $\Omega^{-1}cm^2mol^{-1}$
		C	N	S	Cl	M			
1	L	56.87 (57.57)	5.84 (5.96)	6.91 (6.97)	7.50 (7.61)	----	250	Peach	----
2	MnL_2	52.26 (52.38)	5.58 (5.55)	6.27 (6.34)	7.12 (7.04)	5.33 (5.45)	208	Off-White	15.2
3	CuL_2	61.36 (61.78)	6.68 (6.55)	7.32 (7.48)	8.88 (8.30)	7.28 (7.43)	260	Bluish green	10.6

Spectral studies of ligand and its complexes IR spectra: The characteristic vibrations and assignments of ligand and its complexes are described in Table 2. The IR spectra of the complexes indicate that the ligand behaves as bidentate and the metal coordinates via azomethine nitrogen and phenolic –OH groups[17]. The shift of $\nu_{C=N}$to lower wave number by 30-40 cm^{-1} in the complexes indicates that these groups are involved in complexation[18]. The ligand shows strong band at 3386 cm^{-1} due to phenolic –OH group[19]. This band is absent in all the metal complexes indicating the involvement of this group in complex formation[20]. Moreover, the shift of theν_{C-O} phenolic bands from 1282 cm^{-1} in ligand to 1282-1327 cm^{-1} in the spectra of metal complexes supports the coordination of the phenolic oxygen atom to the metal ion[21]. The bands for ν_{M-O}modes appeared in the range of

470 cm^{-1} - 582 cm^{-1} in all the complexes[22]. The presence of sharp band in the region 505-521 cm^{-1} in the spectra of all the complexes assigned to ν_{M-N} mode[23] further support the involvement of nitrogen atom in coordination.

Table 2: IR spectral data (cm^{-1}) of ligand and its complexes

Sl. No.	Ligand/Complexes	ν_{N-H}	$\nu_{C=N}$	ν_{C-O}	$\nu_{C=O}$	ν_{M-N}	ν_{M-O}
1	$C_{22}H_{19}N_2O_5ClS$	3302	1639	1282	1671	----	-----
2	$C_{44}H_{40}N_4O_{12}Cl_2S_2Mn$	3376	1611	1325	1665	521	582
3	$C_{44}H_{40}N_4O_{12}Cl_2S_2Cu$	3322	1617	1327	1710	505	470

Electronic spectra: Electronic spectra of the ligand and its metal complexes displayed in DMF solution. Electronic spectra of the ligand shows high intensity bands at 40328cm-1 and 4243 cm-1 which indicate n-n* and л-л* transition. The electronic spectra of manganese (II) complex showed four weak bands 16000, 19780, 20680 and 26720 cm^{-1} which have been assigned to $^6A_{1g}\rightarrow{}^6T_{1g}$, $^6A_{1g}\rightarrow{}^4T_{2g}$, $^6A_{1g}\rightarrow{}^4E_g$ and $^6A_{1g}\rightarrow{}^4T_{1g}$ respectively[24]. The electronic spectra of Cu(II) complex shows two energy bands at 33070cm-1 and 25870 cm-1 due to $^4T_{2g}(F)\rightarrow{}^4A_{2g}(F)$ and $^4T_{1g}(F)\rightarrow{}^4T_{2g}(P)$ transitions respectively indicating an octahedral geometry around Cu(II)[25].The above results supported octahedral geometry around the central metal ion.

Magnetic susceptibility: The manganese (II) complex exhibits magnetic moment value 5.50 B.M., which is close to spin only value of 5.90 B.M.for high spin octahedral, manganes e(II) complexes[26]. The magnetic moment obtained for copper (II) complex is 1.83 B.M. which supports the expected octahedral configuration.

ESR Spectra: The ESR spectra of Cu (II) complex was recordedin DMSO at LNT (Liquid nitrogen temperature) and RT (room temperature).The spectrum of the Cu complex at RT shows one intense absorption band in the high field. At LNT Cu (II) complex shows four well-resolved peaks with low field region. The $g_{\parallel}$ and $g_{\perp}$ components have been calculated respectively from the low and high intensity envelopes. The values obtained for Cu (II) complex are $g_{\parallel} > g_{\perp} > 2$ indicating that the Cu (II) lies predominantly in the $dx^2\text{-}y^2$ orbital.[28]

Antibacterial Activity: Antibacterial activity of ligand and their metal complexes are given in Table 3. Antibacterial activity of the ligand and complexes reveal that the activity zone of inhibition for all the complexes against *Escherichia coli* and *Bacillus subtilis* are higher as compared to ligand and pure drug whereas the standard drug Streptomycin showed 55mm and 61 mm inhibition respectively at the same concentration. On the basis of these observations it can be said that complexation or chelation increases the antibacterial activity[29,30].

Table 4: Antibacterial screening data of the ligand L and its complexes

Sl. No.	Ligand/complexes	Dose	Antibacterial activity zone of inhibition (in mm)	
			Escherichia coli	Bacillus subtilis
1	Xipamide	500 ppm	63	68
2	XM-SA (L)	500 ppm	68	71
3	$[MnL_2(H_2O)_2]$	500 ppm	69	73
4	$[CuL_2(H_2O)_2]$	500 ppm	71	75
5	Streptomycin (Standard)	500 ppm	55	61

CONCLUSION

Hence on the basis of elemental analysis, magnetic moment data, conductivity measurements and spectral studies we propose octahedral structure for all the complexes shown in Fig.2. The present work will be extended to the synthesis of other metal complexes and their biological activities

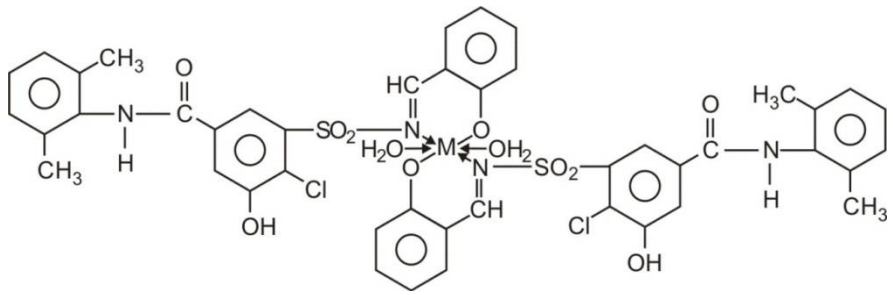


Figure 2: (M= Mn/Cu)

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