Synthesis, Characterization and Antimicrobial Study of Mn (II), Co (II), Ni (II), Cu (II) And Zn (II) Heterocyclic Schiff Base Complexes

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***Abstract*—The nitrogen and oxygen containing heterocyclic Schiff base ligand was synthesized from 5-methylfurfural and 2-amino-3-hydroxypyridine. The prepared ligand has been used for complexation with Mn(II), Co(II), Ni(II), Cu(II) and Zn(II). The microanalysis of the complexes and ligand formed has been done in laboratory. From the microanalysis and conductivity measurement of ligand and complexes, it has been found that both are neutral. The mode of linkage from ligand atom to metal ion has assigned with FTIR spectroscopic study. The magnetic moment measurement and electronic spectroscopy of the complexes reveals that Cu(II) complex is square pyramidal and Zn(II) complex is tetrahedral in geometry. Other all complexes are distorted octahedral in geometry. The ligand and its metal complexes were examined for antimicrobial activity against Gram-positive and Gram-negative bacteria. Penicillin G was taken as the standard.**

***Index Terms*—FTIR, UV spectroscopy, Heterocyclic Schiff base, antimicrobial property.**

INTRODUCTION

In coordination chemistry Schiff base ligands are an important class of ligands and come across vast area of applications in organometallic compounds and in various aspects of bio-inorganic chemistry[1-5]. Schiff bases act as important ligands due to their amazing donor qualities, ease of synthesis and high solubility in common solvents[6,7]. π -system of Schiff bases often imposes geometrical constrains and influences the electronic structure. The biological activities of Schiff bases are noteworthy, encompassing antifungal, antibacterial, food, leather, textile, pharmaceutical and cosmetic applications[8-10]. Due to azomethine compounds of Schiff base can form complexes with transition metal ions that bind 2-DNA bases by the formation of coordinate bonds with nitrogen atom of DNA bases to create chelation ring. Their potential as antibacterial agents has been established in pharmaceutical field[11,12]. Recently, researchers focus on his studies on transition metal complexes with hydroxyl pyridine ligands derived from 2,4-dihydroxybenzaldehyde. These metal complexes of hydroxyl pyridine related Schiff bases are popular in research due to their valuable importance as biomimetic catalysts in oxygenation process[13]. Schiff bases derived from various aldehydes with 4-aminopyridine or 2-aminophenol were reported[14-20].
Present communication reports synthesis, characterization and antimicrobial studies of heterocyclic Schiff base ligand derived from 5-methylfurfural and 2-amino-3-hydroxypyridine and their metal complexes of Mn(II), Co(II), Ni(II), Cu(II) and Zn(II),. The antimicrobial activities of the present Schiff bases are reported.Experimental

II. MATERIAL AND METHODS

All chemicals were of A.R. grade and used without further purification.
The ligand and complexes were micro-analyzed at IIT Patna. The metal contents were determined in laboratory by using standard method[1]. The magnetic moment of complexes was measured by Guoy's instrument using Hg $[Co(SCN)_4]$ as calibrant. The electronic spectra were obtained on a shimadzu UV Spectrometer. The molar conductance of ligand and

complexes were measured in DMSO at room temperature using a Toa CM 405 conductivity meter. The mode of linkage from ligand atom to metal ion is determined by FTIR spectra using KBr discs on a Shimadzu 8300 FTIR spectrophotometer.

III. SYNTHESIS OF THE LIGAND AND THE COMPLEXES

5-methylfurfural (1.2 ml, 0.01 mol) was added drop-wise to an ethanolic solution of 2-amino-3-hydroxypyridine (0.01 mol), then a few drops of glacial acetic acid were added. The reaction mixture was heated under reflux for 6 hours, where a faint yellow precipitate was formed. The precipitate was filtered off, washed with ethanol and recrystallized from hot ethanol.

The ethanolic solution of HL (0.02 mol), a water solution of Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) acetate (0.01 mol) were added drop-wise with stirring. The reaction mixture was refluxed for 3-5 hours. The precipitate was filtered off, washed with hot ethanol and dried in a vacuum desiccator over $CaCl_2$.

IV. RESULT AND DISCUSSION

The micro-analytical data for ligand and metal complexes are shown in Table 1.

Table 1% Composition of the ligand and complexes:

S. No.	Ligand/ Complexes	Carbon (%) Found (calculated)	Hydrogen (%)	Nitrogen (%)	Oxygen (%)	Metal (%)	Colour	Molar conductance C= ($ohm^{-1}\cdot cm^2\cdot mol^{-1}$)	Magnetic moment μ=(B.M.)
01.	HL ($C_{10}H_8N_2O_2$)	65.257 (65.3382)	4.901 (4.9841)	13.726 (13.8535)	15.799 (15.8240)		Faint yellow		
02.	[$MnL_2(H_2O)_2$]	53.376 (53.5582)	4.316 (4.4940)	11.267 (11.3558)	19.376 (19.4566)	10.891 (11.1351)	Light Orange	7.92	5.41
03.	[$CoL_2(H_2O)_2$] ·3/2 H_2O	50.319 (50.3903)	4.466 (4.5165)	10.516 (10.6841)	22.761 (22.8822)	10.991 (11.2384)	Green	13.68	4.32
04.	[$NiL_2(H_2O)_2$]	52.996 (53.1536)	4.276 (4.4601)	11.022 (11.2700)	19.017 (19.3096)	11.723 (11.8064)	Light Brown	13.29	2.86
05.	[$CuL_2(H_2O)$] H_2O	52.363 (52.6398)	4.298 (4.4169)	10.906 (11.1611)	18.979 (19.1230)	12.450 (12.6590)	Dark Brown	11.18	1.68
06.	[ZnL_2]	56.306 (56.4878)	3.735 (3.8780)	11.853 (11.9770)	13.483 (13.6806)	13.795 (13.9764)	Yellow	6.78	0

All the complexes are soluble in common organic solvents. The ligand and complexes were found to be stable for some period. The non-electrolytic nature of all the complexes is due to lower molar conductance value. From the molar conductance value and molecular weight data of the complexes, the general formula of the complexes is found to be [$ML_2(H_2O)_n$], where M = Mn(II), Co(II), Ni(II), Cu(II) and Zn(II); HL = 2N-(5-methylfurfural)-amino-3-hydroxypyridine; n = 2 for Mn(II), Co(II) and Ni(II); n = 1 for Cu(II) and n = 0 for Zn(II). The general composition of Cu(II) and Zn(II) complexes are [CuL_2H_2O] and [ZnL_2].

FTIR

The important I.R. band of ligand and metal complexes are shown in Table 2.

Table 2Important I.R. bands of ligand (HL) and the isolated metal complexes (cm^{-1}):

S. No.	Ligand/ Complexes	υ_{OH}	$\upsilon_{C=N}$	υ_{C-O}	υ_{M-N}	υ_{M-O}	υ_{C-N-C} (py)	υ_{C-O-C} (asym.)	υ_{C-O-C} (sym.)
01.	HL ($C_{11}H_{10}N_2O_2$)	3292	1612	1228			$\upsilon_{C=N}/\upsilon_{C=C}$ (py) ring breathing (1528) & deformation (1089)	1242	1022
01.	$[ML_2(H_2O)_2]$	3314	1556	1227	461	559	1532/1096	1262	1031
02.	$[CoL_2(H_2O)_2] \cdot 3/2\ H_2O$	3321	1558	1232	472	568	1528/1093	1264	1039
03.	$[NiL_2(H_2O)_2]$	3206	1594	1209	499	566	1529/1096	1248	1026
04.	$[CuL_2(H_2O)]\ H_2O$	3244	1572	1216	486	594	1537/1098	1244	1028
05.	$[ZnL_2]$		1604	1206	462	582	1526/1092	1246	1021

The ligand shows a strong band at 3292 cm^{-1}, which can be assigned to the stretching vibration of the –OH group in the ligand[22]. The involvement of deprotonated –OH moiety in the complex formation was confirmed by the shift of C-O band towards lower frequency[23]. The shift of υ_{CO} frequency towards lower frequency suggests the weakening of C-O bond and formation of a stronger M-O bond. The frequency at 1612 cm^{-1}, in the I.R. spectra of ligand may be assigned to azomethine group for the ligand. In all the complexes, this azomethine group frequency shifted towards lower frequency side. This decrease in frequency suggesting that the coordination of ligand to the metal through (-C=N-) azomethine nitrogen atom[24]. All other bands remain particularly unchanged on complex formation. In all the complexes, indicating the non-coordination with metal ions. In complexes of Mn(II), Co(II), Ni(II) and Cu(II), υ bands appeared in the region 3321-3349 cm^{-1}. These bands assigned for $\upsilon_{(OH/H2O)}$, show the presence of coordination with water molecules[25]. The existence of water in the coordination in the complexes of Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) render it difficult to conclude the υ_{OH} group of ligand, which will be overlapped of those of the water molecules. But in case of Zn(II) complexes, the band due to –OH vibration is absent, supports the coordination of OH/H_2O in the complexes of Mn(II), Co(II), Ni(II) and Cu(II).

V. ELECTRONIC SPECTRA AND MAGNETIC MOMENT OF LIGAND AND COMPLEXES

The electronic spectra of all the complexes are shown in Table 3.

Table 3Electtronic spectral data and magnetic moment of the ligand and complexes:

S. No.	Ligand/ Complexes	Band position $\upsilon(cm^{-1})$	Molar Extinction Coefficient $\varepsilon_{max}(L \cdot mol^{-1} \cdot cm^{-1})$	BandA ssignment	Magnetic moment M (B.M.)	Suggested structure
01.	$[ML_2(H_2O)_2]$	37832 35668 34092 28614		C.T. L.F. L.F. C.T.	5.62	Distorted Octahedral
02.	$[CoL_2(H_2O)_2] \cdot 3/2\ H_2O$	27326 23188 16598 14623 12428 19626		Intra ligand transition $\pi^* \leftarrow n$ $^4T_{1g}(P) \leftarrow {}^4T_{1g}(F)$ $^4A_{2g}(P) \leftarrow {}^4T_{1g}(F)$ $^4T_{2g}(F) \leftarrow {}^4T_{1g}(F)$ C.T.	4.48	Distorted Octahedral

03.	$[NiL_2(H_2O)_2]$	36684 23782 21212 18192 15422 12168 36434 25214	3.52 3.48 1.76 1.72 1.64 4.02 3.65	Intra ligand transition Intra ligand transition C.T. $^3T_{1g}(P)\leftarrow{}^3A_{2g}(F)$ $^3T_{1g}(F)\leftarrow{}^3A_{2g}$ $^3T_{2g}\leftarrow{}^3A_{2g}$ Intra ligand transition Intra ligand transition	3.07	Distorted Octahedral
04.	$[CuL_2(H_2O)]\ H_2O$	37652 35542 18916 14921 12826 34652 27212	3.99 4.11 1.41 0.64 0.34 3.74 3.68	Intra ligand transition Intra ligand transition $^2E_g(dx^2\text{-}y^2\rightarrow dxz,dyz)\leftarrow{}^2B_{1g}$ $^2B_{2g}(dx^2\text{-}y^2\rightarrow dxy)\leftarrow{}^2B_{1g}$ $^2A_{1g}(dx^2\text{-}y^2\rightarrow dz^2)\leftarrow{}^2B_{1g}$ Intra ligand transition Intra ligand transition	1.7	Square pyramidal
05.	$[ZnL_2]$	34321 23560 37786	3.91 3.68 4.11	$\pi^*\leftarrow\pi$ C.T. $\pi^*\leftarrow\pi$	0	Tetrahedral

The electronic spectra of complexes were recorded in DMSO. All the complexes show bands above 25000 cm^{-1} in addition to the $\pi^*\leftarrow$n transition in the ligand. $\pi^*\leftarrow$n transitions due to azomethine group shifted towards lower frequency in all the complexes. This shift towards lower energy show nitrogen atom of imine group involved in coordination to the metal ion[26]. The electronic spectra can be used for the determination of the structure and geometry of the complexes with the help of number and position of spectral bands in the absence of X-rays studies[27-29].

Mn(II) Complex: The no. of unpaired electron for Mn(II) ion in weak crystal field is 5 and is paramagnetic in nature. The magnetic moment of $[MnL_2(H_2O)_2]$ complex is found to be 5.62 B.M. This value of magnetic moment shows that Mn(II) complex is high spin magnetically dilute octahedral complex[30]. The ground state term for Mn(II) is ^6S. The electronic spectra of $[MnL_2(H_2O)_2]$ complex display spin forbidden weak bands. Result of magnetic moment, molar conductivity and FTIR spectra suggested distorted octahedral geometry of $[MnL_2(H_2O)_2]$ complex[31].

Co(II) Complex: The no. of unpaired electrons in Co(II) complex is found to be 3 and is paramagnetic in nature. The magnetic moment of complex is found to be 4.48 B.M. This value is higher than the expected for 3 unpaired electron (μ_s = 3.88 B.M.) for a high-spin octahedral complex. The high value of magnetic moment is explained by appreciable orbital contribution from triply degenerate ground term $^4T_{1g}$[32,33]. The following three bands are obtained in electronic spectra of Co(II) complex at different frequencies as shown below.

$^4T_{1g}(P)\leftarrow{}^4T_{1g}(F)$, υ_1 = 18916 cm^{-1};
$^4A_{2g}(P)\leftarrow{}^4T_{1g}(F)$, υ_2 = 14921 cm^{-1};
$^4T_{2g}(F)\leftarrow{}^4T_{1g}(F)$, υ_3 = 12826 cm^{-1}.

These values are in good agreement with the distorted octahedral geometry around Co(II) in the complex[34].

Ni(II) Complex: The magnetic moment of Ni(II) complex is found to be 3.07 B.M. at room temperature. This value is greater than the expected value from two unpaired electrons (μ_s = 2.828) for d^8 system[35-40]. The high value of Ni(II) complex is explained by spin-orbit coupling and is given by $\mu_{eff} = \mu_s(1\text{-}4\lambda/10\ Dq)$. Since λ is negative for d^8 system, the μ_{eff} is in good agreement regarding distorted octahedral complex of Ni(II)[41-42]. The following three spectral bands display by $[NiL_2(H_2O)_2]$ complex:

$^3T_{1g}(P)\leftarrow{}^3A_{2g}(F)$, υ_1 = 18192 cm^{-1};
$^3T_{1g}(F)\leftarrow{}^3A_{2g}$, υ_2 = 15422 cm^{-1};
$^3T_{2g}\leftarrow{}^3A_{2g}$, υ_3 = 12168 cm^{-1}.

The position of these spectral bands indicates a distorted geometry for $[NiL_2(H_2O)_2]$ complex[43].

Cu(II) complex: The Cu(II) is d^9 system. The magnetic moment of Cu(II) complex is found to be 1.68 B.M. and is paramagnetic in nature. The electronic spectra of Cu(II) shows absorption bands in the range 12826-18916 cm^{-1} may be assigned due to d-d transitions[44]. The possible spin allowed transitions shown as:

$^2E_g(dx^2\text{-}y^2\rightarrow dxz,dyz)\leftarrow{}^2B_{1g}$, υ_1 = 181916 cm^{-1};
$^2B_{2g}(dx^2\text{-}y^2\rightarrow dxy)\leftarrow{}^2B_{1g}$, υ_2 = 14921 cm^{-1};

$^2A_{1g}(dx^2\text{-}y^2 \rightarrow dz^2) \leftarrow {}^2B_{1g}$, $\upsilon_3 = 12826\ cm^{-1}$.

The magnetic moment value and these electronic transitions support that the complex is square pyramidal in geometry[45].

Zn(II) Complex: Zn(II) is d^{10} system. Hence ZnL_2 is diamagnetic and it would have tetrahedral geometry[46].

VI. ANTIMICROBIAL ACTIVITY

The bio-efficacy of the free ligand and their metal complexes have been examined against the growth of bacteria. In-vitro, to evaluate their antimicrobial activities, the different bacterial strains such as Bacillus cereus and Staphylococcus aureus (Gram-positive bacteria) as well as Escherichia Coli and Pseudomonas aeruginosa (Gram-negative bacteria) were used to find antibacterial activity[47]. The antibiotic Penicillin G has been taken as Standard for comparision. The antimicrobial testing was performed by standard methodology[48]. The ligand and its complexes were prepared in DMSO and tested against the selected microbial strains. Each process was performed for 3 times. The experimental results were shown in Table 4.

Table 4Antimicrobial activity of Schiff base ligand their metal complexes:

S. No.	Ligand/ Complexes	Concentration (µg/ml)	Gram-positive bacteria		Gram-negative bacteria	
			Bacillus cereus	Staphylococcus aureus	Escherichia Coli	Pseudomonas aeruginosa
01.	Ligand (HL)	50	0	0	0	0
		100	0	6±0.04	0	0
		150	7±0.01	8±0.05	7±0.15	6±0.16
02.	Mn(II)	50	0	0	0	0
		100	0	0	6±0.04	0
		150	7±0.15	0	8±0.04	6±0.13
03.	Co(II)	50	0	0	0	0
		100	0	0	0	0
		150	5±0.13	7±0.08	7±0.15	0
04.	Ni(II)	50	0	0	11±0.03	6±0.16
		100	0	6±0.17	13±0.02	8±0.16
		150	6±0.08	8±0.13	15±0.06	10±0.05
05.	Cu(II)	50	0	0	6±0.05	6±0.08
		100	0	0	9±0.05	8±0.07
		150	6±0.16	6±0.5	12±0	10±0.04
06.	Zn(II)	50	0	0	7±0.06	0
		100	0	0	9±0.05	0
		150	6±0.14	6±0.14	9±0.13	6±0.03
07.	Penicillin G	50	8±0	10±0.03	9±0.06	0
		100	10±0	12±0.03	10±0.03	0
		150	13±0	16±0	12±0.03	0

It is evident from the data that complexes exhibit greater antimicrobial activity than ligand but smaller than the standard taken at all the concentration.

VII. CONCLUSION

The Schiff base ligand [HL = 2N-(5-methylfurfural)-amino-3-hydroxypyridine] is found to act as monoanionic bidentate coordinated through azomethine nitrogen (-C=N-) and OH of pyridine ring except Zn(II) and Cu(II) complexes of Ni(II), Co(II) and Mn(II) are found to have distorted octahedral geometry. The position along z-axis is occupied by water molecule. The Cu(II) complex is found to be possesses square pyramidal geometry with water molecule along z-axis. The Zn(II) complex have tetrahedral geometry. The ligand and its complexes are found to be biologically active against the Gram-positive (Bacillus cereus and Staphylococcus aureus)

and Gram-negative (Escherichia Coli and Pseudomonas aeruginosa) bacteria.

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