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SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL STUDY OF SOME BIVALENT TRANSITION METAL IONS COMPLEXES WITH HETEROCYCLIC SCHIFF BASE

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Abstract

Complexes of Ni(II), Cu(II), Zn(II), Co(II) and Mn(II) have been prepared with a heterocyclic Schiff base ligand. The heterocyclic Schiff base ligand was prepared from furfural and 2-amino-3-hydroxypyridine. The ligand and the complexes formed are microanalyzed. Conductivity measurement of the complexes shows that all the complexes are neutral. The FTIR spectroscopic method has inferred the bonding type of the ligand and complexes. Magnetic moment measurement and UV spectroscopy have inferred that Ni(II), Co(II) and Mn(II) complexes are distorted octahedral in geometry. Cu(II) complex is square pyramidal and Zn(II) complex is tetrahedral in geometry. The Schiff base and its metal complexes were examined for antimicrobial activity against Gram-positive and Gram-negative bacteria. Penicillin G was taken as the standard.

Keywords: Heterocyclic Schiff base, FTIR, UV spectroscopy, geometry, Antimicrobial property.

Introduction

Schiff bases have been taken as unique ligands in coordination chemistry due to their incorporation of diverse donor sites and exhibiting good flexibility [1,2]. Metal complexes of Schiff bases are significant because of their stability, capability to form metal chelates and wide range of biological applications [3]. Schiff bases and their metal complexes have biological activities due to the presence of the azomethine (C=N) group, which serves as a binding site for metal ions to interact with various biomolecules [4]. Schiff bases exhibited potent antibacterial, anticancer, antifungal and diuretic activities and were widely used in the food and dye industry, catalysts, analytical chemistry and agro activities [5].

Heterocyclic derivatives of Schiff bases were largely studied because of their therapeutic potential against certain types of tumors [6,7]. Transition metal complexes with hydroxypyridine ligands have been the focus of extensive research recently. Hydroxypyridine-based Schiff base ligand and their metal complexes are popular in research due to their importance in biomimetic catalysts in the oxygenation process [8].

The present paper deals with the preparation, characterization and antimicrobial studies of the Schiff base ligand derived from furfural and 2-amino-3-hydroxypyridine and their metal ion complexes of Co(II), Ni(II), Cu(II), Zn(II) and Mn(II).

Experimental

Materials and method

Furan-2-carbaldehyde (furfural), 2-amino-3-hydroxypyridine and metal acetate salts were of AR-grade.
The C, H and N contents were determined on a CHNS analyzer in IIT Patna. The metal contents were determined using a standard method [9]. The FTIR Spectra were measured using KBr discs on a Shimadzu 8300 FTIR spectrophotometer. The magnetic moments of complexes were measured by Gouy's method using $Hg[Co(SCN)_4]$ as calibrant. Molar conductance of ligand and their metal complexes were determined in DMSO at room temperature using a Toa CM 405 conductivity meter. The electronic spectra were obtained on a Shimadzu UV spectrophotometer. The metal analysis was carried out by titration with EDTA [10].

Synthesis of Ligand and complexes

Furan-2-carbaldehyde (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 recrystalised from hot ethanol.

The ethanolic solution of HL (0.02 mol), a water solution of Ni(II), Cu(II), Zn(II), Co(II) and Mn(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 $CaCl_2$.

Result and Discussion

The analytical data for ligand and complexes are given in Table 1.

Table 1

Composition of the ligand and complexes in percentage:

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 ($C_{10}H_8N_2O_2$)	63.177 (63.7673)	3.998 (4.2826)	14.026 (14.8861)	16.822 (17.0035)		Light yellow		
02.	$[NiL_2(H_2O)_2]$	50.876 (51.2114)	3.278 (3.8674)	11.382 (11.9441)	20.133 (20.4645)	12.101 (12.5125)	Brown	13.21	2.98
03.	$[CuL_2(H_2O)]$ H_2O	50.167 (50.6870)	3.068 (3.8278)	11.299 (11.8218)	20.098 (20.2549)	13.088 (13.4083)	Dark Brown	11.25	1.7
04.	$[ZnL_2]$	54.068 (54.6286)	3.001 (3.2087)	12.124 (12.7411)	14.120 (14.5533)	14.687 (14.8681)	Brown	6.8	0
05.	$[CoL_2(H_2O)_2]$ $\cdot 3/2\ H_2O$	47.890 (48.3985)	3.996 (4.2641)	11.786 (11.2880)	23.906 (24.1756)	11.522 (11.8736)	Light grey	13.64	4.36
06.	$[MnL_2(H_2O)_2]$	51.202 (51.6246)	3.363 (3.8986)	11.885 (12.0404)	20.023 (20.6296)	11.227 (11.8065)	Dark	7.84	5.44

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

FTIR

The important I.R. band for ligand and metal complexes are υ(OH), υ(C=N), υ(C-O), υ(M-N), υ(M-O), υ(C-N-C)py and υ(C-O-C)furan. The I.R. frequencies of these important bands are shown in Table 2.

Table 2

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

S. No.	**Ligand/ Complexes**	**υ(OH)**	**υ(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 ($C_{10}H_8N_2O_2$)	3288	1609	1225			υ(C=N)/ υ(C=C)py ring breathing(1528) & deformation (1089)	1237	1017
02.	$[NiL_2(H_2O)_2]$	3308	1589	1207	492	568	1531/1092	1244	1021
03.	$[CuL_2(H_2O)]$ H_2O	3248	1569	1213	478	598	1532/1093	1241	1022
04.	$[ZnL_2]$		1599	1203	452	589	1529/1090	1242	1019
05.	$[CoL_2(H_2O)_2]$ $\cdot 3/2\ H_2O$	3323	1550	1228	466	572	1533/1091	1258	1035
06.	$[MnL_2(H_2O)_2]$	3317	1551	1224	454	564	1530/1093	1256	1026

Identification of significant bands has been based on their relationship to other comparable complexes [11-14]. The ligand shows a strong band at 3288 cm^{-1}, which can be attributed to the stretching vibration of the -OH group in the ligand [15]. The involvement of deprotonated –OH moiety in the complex formation was confirmed by the shift of υ(C-O) stretching band towards lower frequency to the extent 12-22 cm^{-1} [16]. This decrease in the frequency of υ(C-O) to lower range suggests the weakening of the C-O bond and formation of a stronger M-O bond.

The frequency at 1609 cm^{-1}, in the I.R. spectra of ligand, may be assigned to azomethine group (C=N) for the ligand. It is observed that azomethine group frequency shifts towards lower frequency side. Thereby suggesting that the coordination of ligand to the metal is through (C=N) azomethine nitrogen atom [17].

All other bands as shown as υ_{asym}(C-O-C) furan, υ_{sym}(C-O-C) furan, υ(C-N-C)py ring deformation and breathing bands, remain particularly unchanged on complex formation in all the complexes, indicating non-coordination with metal ions.

Bands observed in Ni(II), Cu(II), Co(II) and Mn(II) complexes in the region 3323 to 3248 cm^{-1} assigned for υ(OH/H_2O), show the presence of coordinated water molecules [18]. The existence of water in the coordination in the I.R. spectra of Ni(II), Cu(II), Co(II) and Mn(II) complexes render it difficult to conclude the υ(OH) group of the ligand, which will be overlapped by 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 Ni(II), Cu(II), Co(II) and Mn(II).

Electronic Spectra and magnetic moment of ligand and complexes

The electronic spectra of all the complexes were recorded in DMSO. The spectral bands and their assignments are given in Table 3.

The spectra of all complexes show bands above 25000 cm^{-1} (400 nm) in addition to the $n \rightarrow \pi^*$ transition in the ligand. In the complexes, $n \rightarrow \pi^*$ transitions due to azomethine group are shifted to the lower energy, which show nitrogen atom of imine group involved in coordination to the metal ion [19]. In the absence of X-ray studies, the electronic spectra can be used for the determination of the structure of the complexes, as the number and position of spectral bands provide good insight into the geometry of metal complexes [20-22].

Table 3

Electronic spectral data and Magnetic moment of the ligand and complexes:

S. No.	Ligand/ Complexes	Band position λ(nm)	Band position υ(cm^{-1})	Molar Extinction Coefficient ε_{max}(L·mol^{-1}·cm^{-1})	Band Assignment	Magnetic moment μ (B.M.)	Suggested structure
01.	$[NiL_2(H_2O)_2]$	274 424 476 558 654 276 415	36490 23580 21000 17920 15290 11920 36230 24094	3.46 3.41 1.72 1.66 1.59 3.99 3.57	Intra ligand transition Intra ligand transition C.T. $^3A_{2g}(F)\rightarrow{}^3T_{1g}(P)$ $^3A_{2g}\rightarrow{}^3T_{1g}(F)$ $^3A_{2g}\rightarrow{}^3T_{2g}$ Intra ligand transition Intra ligand transition	3.05	Octahedral
02.	$[CuL_2(H_2O)]$ H_2O	267 283 534 665 790 290 371	37450 35330 18720 15030 12650 34480 26950	3.92 3.99 1.3 0.53 0.23 3.63 3.57	Intra ligand transition Intra ligand transition $^2B_{1g}\rightarrow{}^2E_g(dx^2\text{-}y^2\rightarrow dxz, dyz)$ $^2B_{1g}\rightarrow{}^2B_{2g}(dx^2\text{-}y^2\rightarrow dxy)$ $^2B_{1g}\rightarrow{}^2A_{1g}(dx^2\text{-}y^2\rightarrow dz^2)$ Intra ligand transition Intra ligand transition	1.7	Square pyramidal
03.	$[ZnL_2]$	293 430 266	34120 23250 37590	3.80 3.57 4.00	$\pi\rightarrow\pi^*$ C.T. $\pi\rightarrow\pi^*$	0	Tetrahedral
04.	$[CoL_2(H_2O)_2]\cdot\frac{3}{2}H_2O$		25315 22990 16395 14525 12223 19415		Intra ligand transition $n\rightarrow\pi^*$ $^4T_{1g}(F)\rightarrow{}^4T_{1g}(P)$ $^4T_{1g}(F)\rightarrow{}^4A_{2g}(P)$ $^4T_{1g}(F)\rightarrow{}^4T_{2g}(F)$ C.T.	4.36	Octahedral
05.	$[MnL_2(H_2O)_2]$		37604 35463 32071 28407		C.T. L.F. L.F. C.T.	5.58	Octahedral

Ni(II) complex: At room temperature, the magnetic moment of the Ni(II) complex is found to be 3.05 B.M. This value is greater than the expected value from two unpaired electrons (μ_s=2.828 B.M.) for the d^8 system of Ni(II) complex. The high value of the Ni(II) complex compared to the expected value is explained by spin-orbit coupling and is given by $\mu_{eff} = \mu_0\ (1\text{-}4\lambda/10Dq)$. Since λ is negative for the d^8 system, the μ_{eff} becomes greater than μ_s values [22-27]. This value of μ_{eff} is in agreement with the reported value for octahedral complexes of Ni(II) [28-29].

The electronic spectra of Ni(II) complex display three absorption at 17920, 15290 and 11920 cm^{-1}. These transitions may be assigned to $^3A_{2g}(F)\rightarrow{}^3T_{1g}(P)$ and $^3A_{2g}(F)\rightarrow{}^3T_{1g}(F)$ and $^3A_{2g}(F)\rightarrow{}^3T_{2g}$. The position of these bands suggests a distorted octahedral geometry for $[NiL_2(H_2O)_2]$ with the reported value for octahedral complexes of Ni(II) [30].

Cu(II) complex: The electronic spectra of Cu(II) complex display absorption bands in the range 18720-12650 cm^{-1}, may be assigned due to d-d transitions [31]. The possible spin allowed transitions shown as $^2B_{1g}\rightarrow{}^2E_g(dx^2\text{-}y^2\rightarrow dxz, dyz)$, $^2B_{1g}\rightarrow{}^2B_{2g}(dx^2\text{-}y^2\rightarrow dxy)$ and $^2B_{1g}\rightarrow{}^2A_{1g}(dx^2\text{-}y^2\rightarrow dz^2)$. The magnetic moment value of Cu(II) complex is 1.84 B.M. The magnetic moment value and these transitions for square planar or square pyramidal copper complexes are with the $dx^2\text{-}y^2$ ground state [32].

Zn(II) complex: $[ZnL_2]$ is diamagnetic and has completely filled d^{10} configuration, therefore, it would have tetrahedral geometry [33].

Co(II) complex: The magnetic moment of Co(II) complex is 5.14 B.M. This value is higher than the expected for three unpaired electrons (μ_s=3.88 B.M.) for a high-spin octahedral complex. The high value may be explained by appreciable orbital contribution from the triply degenerate ground term $^4T_{1g}$ [34,35]. The three spectral bands are obtained in electronic spectra of Co(II) complex at 16395, 14525 and 12223 cm^{-1}. These three bands may be assigned as the transitions $^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$, $^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$ and $^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$. These values are in accordance with the distorted octahedral geometry of Co(II) complex [36].

Mn(II) complex: The magnetic susceptibility of $[MnL_2(H_2O)_2]$ complex is found to be 5.58 B.M. This value displays the fact that Mn(II) complex is a high spin magnetically dilute octahedral complex [37]. The electronic spectra of the Mn(II) complex display spin-forbidden weak bands. Results of molar conductivity, magnetic moment, and FTIR spectra suggested distorted octahedral geometry of Mn(II) complex [38].

Antimicrobial Activity

The bio-efficacy of the ligand and its metal complexes has been determined against the growth of bacteria. In vitro, to evaluate their antimicrobial activities, the various 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 [39]. The antibiotic Penicillin G has been taken as the standard for comparison. The antimicrobial testing was performed by laboratory and clinical standard methodology [40]. The ligand and its complexes were prepared in Dimethylformamide (DMF) and tested against the selected microbial strains. Each experiment was performed 3 times. The experimental results have been given in Table 4.

It is evident from the data obtained that complexes exhibit greater biological activity than the ligand but smaller than the standard Penicillin G at all the concentrations.

Table 4

Antimicrobial 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)	50 100 150	0 0 7±0.02	0 6±0.06 8±0.06	0 0 7±0.14	0 0 6±0.14
02.	Ni(II)	50 100 150	0 0 6±0.06	0 6±0.14 8±0.12	11±0 13±0 15±0	6±0.14 8±0.14 10±0.03
03.	Cu(II)	50 100 150	0 0 6±0.14	0 0 6±0.3	6±0.03 9±0.03 12±0	6±0.06 8±0.06 10±0.03
04.	Zn(II)	50 100 150	0 0 6±0.12	0 0 6±0.12	7±0.04 9±0.03 9±0.12	0 0 6±0.04
05.	Co(II)	50 100 150	0 0 5±0.12	0 0 7±0.06	0 0 7±0.14	0 0 0
06.	Mn(II)	50 100 150	0 0 7±0.14	0 0 0	0 6±0.03 8±0.03	0 0 6±0.14
07.	Penicillin G	50 100 150	8±0 10±0 13±0	10±0.03 12±0.03 16±0	9±0.06 10±0.03 12±0.03	0 0 0

Conclusion

The Schiff base ligand (HL) is found to act as a bidentate monoanionic coordinated through azomethine nitrogen (C=N) and OH of the pyridine ring. The complexes of Ni(II), Co(II) and Mn(II) are found to have a distorted octahedral geometry. The positions along z-axis are occupied by neutral water molecule. The complex of Cu(II) is found to possess square pyramidal geometry with water along z-axis. The complex of Zn(II) possesses a tetrahedral geometry. The ligand and its complexes are found biologically active against the Gram-positive bacteria (Bacillus cereus and Staphylococcus aureus) and Gram-negative bacteria (Escherichia Coli and Pseudomonas aeruginosa).

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