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Chemistry

SYNTHESIS, STRUCTURAL ANALYSIS AND BIOLOGICAL PROPERTIES OF MN(II), CO(II), NI(II) AND CU(II) COMPLEXES OF SCHIFF'S BASE LIGAND

Ravikant Department of Chemistry, Zakir Husain Delhi College, University of Delhi, JLN Marg, New Delhi-110002, India

Sulekh Chandra* Department of Chemistry, Zakir Husain Delhi College, University of Delhi, JLN Marg, New Delhi-110002, India *Corresponding Author

ABSTRACT In this research work, the acyclic nitrogen (N) and oxygen (O) donor atoms containing Schiff's base ligand (L) acetophenonenicotinichydrazone has been derived. Ligand (L) is prepared from acetophenone and nicotinic hydrazide by following condensation reaction and its Mn(II), Co(II), Ni(II) and Cu(II) complexes also prepared and structural analysis carried out by elemental analyses, IR, UV-Vis., mass, ^{1}H-NMR and EPR spectral studied. The ligand (L) behaves as a bidentate ligand and coordinates to metal ions via nitrogen and oxygen donor atoms and complexes have mononuclear construction. Cu(II) complex exhibit distorted octahedral geometry, whereas an octahedral geometry is suggested for all other complexes. Ligand and complexes are evaluated to examine inhibition potential against bacterial and fungal strains and the assay indicated that the metal complexes exhibited remarkable antibacterial and antifungal activities against tested microorganism.

KEYWORDS : Ligand, structural analysis, mononuclear, complexes, inhibition, antibacterial, antifungal

INTRODUCTION

Due to presence of many biologically significant activities, synthesis of Schiff's base ligands and their mononuclear metal complexes is a growing field of investigation in coordination and bioinorganic chemistry [1-3]. Transition metal complexes of these ligand are expected to have a great interest because of their anticancer, antitumor, antibacterial, antiviral, antifungal, antioxidant, anti carcinogenic activity and DNA binding and cleavage [4-12]. Presence of metal ions often enhances the efficiency as well as drug action of organic biologically active agents (Free ligands) [13]. Metal complexes of these biologically active agents obstruct the pathogen's enzymes which build up interference in the cellular respiration and slow down the protein synthesis [14-15]. Besides biologically active agents, Schiff's base ligands as well as their metal complexes also used in catalytically activating small molecules in electrochemically assisted reactions [16]. In the radiance of aforesaid applications, present research paper reports synthesis, structural analysis and biological properties of Mn(II), Co(II), Ni(II) and Cu(II) complexes of Schiff's base ligand.

EXPERIMENTAL

MATERIALS AND METHODS

Analytical grade chemicals have been used for synthesis of Schiff's base ligand. Acetophenone and nicotinic hydrazide have been purchased from Sigma Aldrich and metal salts from Merck, S.D. Fine India and used as received. The microanalysis for carbon (C), H (hydrogen), N (nitrogen) has been done by using Carlo-Erba 1106 elemental analyzer. The molecular weight of ligand is determined by recording mass spectra, JEOL, JMS-DX-303 mass spectrometer. The Proton Nuclear Magnetic Resonance (^{1}H-NMR) spectra were recorded in deuterated dimethyl sulfoxide (d_6-DMSO) at room temperature on a Bruker Advanced DPX-300 spectrometer. Ultraviolet-Visible (UV-Vis) spectra in DMSO have been recorded on Shimadzu UV-visible mini-1240 spectrophotometer ranging 200-1100 nm at room temperature. The Fourier Transform Infra-Red (FT-IR) spectra for all synthesized compounds were recorded on FT-IR spectrum BX-II spectrophotometer by using KBr pellet in the range 4000-400 cm^{-1}. X-band Electron Paramagnetic Resonance (EPR) spectra of synthesized Mn(II), Co(II) and Cu(II) complexes has been recorded at room temperature on on E_4-EPR spectrometer using the DDPH as the g-marker at SAIF, IIT Bombay. Magnetic susceptibility measurements has been carried out on Gouy balance at room temperature by using $CuSO_4.5H_2O$ as calibrant.

Synthesis of Schiff's base ligand acetophenonenicotinichydrazone

In a hot stirred ethanolic solution of acetophenone (0.01 mol, 1.2015 g), ethanolic solution of nicotinic hydrazide (0.01 mol, 1.375 g) has been added dropwise with constant stirring. The reaction was refluxed for 13 hrs at 75^{0}C and then cooled at room temperature. For cooling purpose, reaction solution has been kept in an ice bath. After one hour, the precipitation took place and obtained precipitate was filtered off, washed with distilled water and re-crystallized then dried in a desiccator. The general scheme for the synthesis of ligand is shown in Figure 1. Its analytical data and physical properties are given in Table 1.

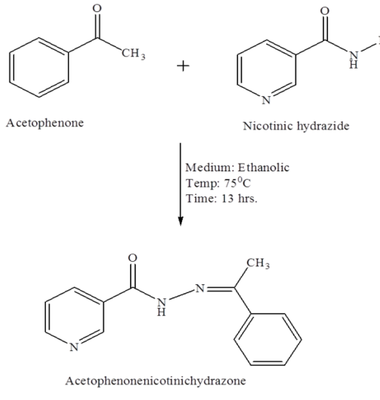


FIGURE 1: Synthesis Scheme for Schiff's base ligand

Procedure for synthesis of complexes of Schiff's base ligand

All complexes have been synthesized by using general condensation reaction. Hot ethanolic solution of synthesized ligands (0.002 mol) was added to hot ethanolic solution of corresponding metal salts (0.001 mol) with constant stirring. The resulted reaction solution was refluxed at optimum condition. On cooling, the colored solid complex was precipitated out. This colored precipitate was filtered off, washed with 50% ethanol and dried in desiccator which contains silica gel as moisture absorbent.

BIOLOGICAL ACTIVITY

The synthesized acyclic Schiff's base and its metal complexes have been screened for antifungal and antibacterial activities against some selective microorganisms. Three different bacteria and two fungi have been chosen for this purpose.

Antifungal activity: Poisoned food method

Poisoned food method is mostly used to evaluate the antifungal effect against fungi [17]. The antifungal agent is incorporated into the molten agar at a desired concentration (1000 ppm, 750 ppm, 500 ppm and 250 ppm) and mixed well. Then, the medium is poured into disposable petri dishes. After pre-incubation, the inoculation can be done by fungi (*A.niger, M.phasolina and P.glomerata*) which are deposited in the center of the plate. After further incubation under suitable conditions for the fungal strain tested, the diameters of fungal growth in control and sample plates are measured.

Antibacterial activity: Well diffusion method

The antibacterial activity was evaluated by using well diffusion method [18]. Synthesized compounds were tested against three bacteria *i.e., E. coli, S. aureus* and *P.aeruginosa*. Nutrient agar and Neomycin were used as a medium and as a standard drug, respectively. The inhibition of the bacterial growth was also expressed in (%) and determined from the growth in the test plate to the respective control plate.

RESULT AND DISCUSSION

Molar conductance, Micro analytical and other physical properties of Schiff's base ligand and metal complexes are accessible in Table 1. The molar conductance values (16-19 Ω^{-1} cm^2 mol^{-1}) clearly indicate non-electrolytic nature of all metal complexes [19]. The micro analytical data of the complexes exposed $M(L)_2X_2$ type geometry with 1:2 (metal:ligand) ratio. The ligand is soluble in ethanol and metal complexes were insoluble in water but soluble in DMSO and DMF. On the basis of spectral studies with combination of their physical data, proposed structure determination and confirmation of the complexes is well explored.

TABLE 1: Analytical, physical data of Mn(II), Co(II), Ni(II) and Cu(II) complexes with Schiff's base ligand

Compounds	Molar Ratio (M:L)	Colour	Formula Wt. (g/mol)	M.P. (℃)	Molar conductance (Ω^{-1} cm^2 mol^{-1})	Yield (%)	Elemental analysis (%) Cal. (Found)			
							C	H	N	M
Schiff's base Ligand	1:2	White	239.29	164	-	64	70.09 (70.20)	5.36 (5.43)	17.41 (17.51)	-
$[Mn(L)_2Cl_2]$	1:2	White	604.51	>260	16	59	55.58 (55.49)	4.30 (4.25)	13.89 (13.80)	9.08 (9.06)
$[Co(L)_2Cl_2]$	1:2	Light Brown	604.51	>260	16	59	27.61 (27.58)	2.13 (2.11)	6.90 (6.56)	9.68 (9.63)
$[Ni(L)_2Cl_2]$	1:2	Dark Green	718.73	>260	16	59	29.73 (29.71)	2.64 (2.60)	15.32 (15.29)	8.02 (8.00)
$[Cu(L)_2Cl_2]$	1:2	Green	652.47	>250	19	52	27.40 (27.36)	2.12 (2.09)	6.85 (6.82)	10.36 (10.34)

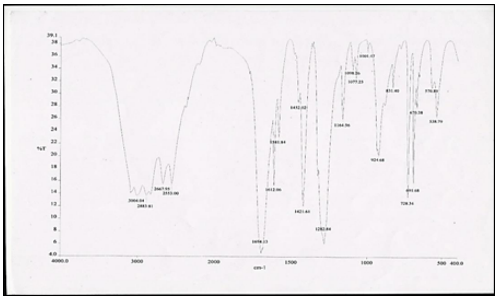


FIGURE 2: IR spectrum of Schiff's base ligand

[1]H-NMR spectral analysis

The 1H-NMR spectrum of ligand shows no signal corresponding to primary amine proton. The pyridine ring protons of ligand resonate in the range of 8.19-8.44 ppm and these signals were found between 8.18 and 8.59 ppm in 1H-NMR spectrum [22]. Signals appeared at positions 9.68 ppm and 2.25 ppm corresponding to the presence of protons of NH and -CH3 group. A signal at position 6.70 ppm is due to proton of the aromatic ring (Ar-H) [23]. These signals indicate the presence of four different types of protons in ligand as shown in Figure 3.

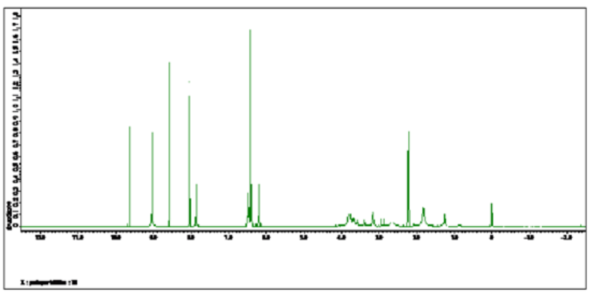


FIGURE 3: 1H-NMR spectrum of ligand

Mass spectral analysis

The obtained molecular mass value for the ligand is mutual with data of other spectral studies. A peak obtained at m/z = 238 in the spectrum of ligand is a sign of molecular ion peak M+ which firmly support the proposed molecular formula of ligand Figure 4.

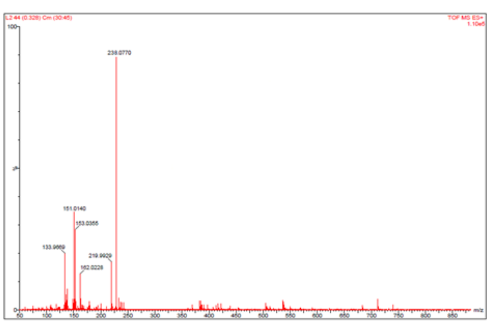


FIGURE 4: Mass spectrum of ligand

IR spectral analysis for metal complexes

The important IR spectral bands of ligand and its complexes are given in Table 2. The band υ(C=N) appeared at 1612 cm^{-1} in ligand spectrum is shifted towards lower wave number (1587-1604 cm^{-1}) in complexes indicate coordination take place through azomethine nitrogen [24]. It is also verified by the presence of bands at positions (434-445 cm^{-1}) [25]. A band appeared at position 1694 cm^{-1} which shows the presence of υ(C=O) group, also shifted towards lower wave number (1651-1680 cm^{-1}) which indicates that coordination took place through oxygen atom of C=O group [26]. Coordination through oxygen atom is also verified by presence of new bands in the range of 518-537 cm^{-1} [27]. Additionally, the IR spectra of the complexes show the bands due to coordinated anions. The chloro complex shows band at positions 330-345 cm^{-1} due to (M-Cl) bonding [28].

TABLE 2: IR spectral data of Mn(II), Co(II), Ni(II) and Cu(II) complexes with Schiff's base ligand

Compounds	υ(C=N)	υ(C=O)	υ(M-N)	υ(M-O)	υ(M-Cl)
Schiff's base ligand	1612	1694	-	-	-
$[Mn(L)_2Cl_2]$	1598	1651	436	533	345
$[Co(L)_2Cl_2]$	1604	1680	439	518	350
$[Ni(L)_2Cl_2]$	1602	1678	434	533	330
$[Cu(L)_2Cl_2]$	1587	1680	445	537	340

Magnetic moment

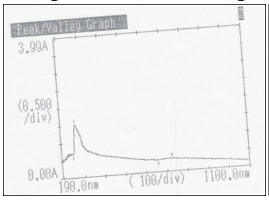
The observed values of magnetic moment for Mn(II), Co(II), Ni(II) and Cu(II) metal complexes is summarized in Table 3. The value of magnetic moment indicated that the complexes were paramagnetic in nature due to the presence of three unpaired electrons.

Electronic spectral studies

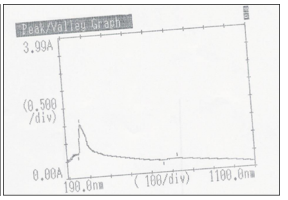
The electronic spectra of Mn(II) complexes of all synthesized ligands under study show absorption bands at positions 18685 cm^{-1}, 22973 cm^{-1}, 24989 cm^{-1} and 28924 cm^{-1}. These transition assigned to the $^6A_{1g} \rightarrow {}^4T_{1g}$ (^{4}G), $^6A_{1g} \rightarrow {}^4E_g$, $^6A_{1g} \rightarrow {}^4E_g$ (^{4}D) and $^6A_{1g} \rightarrow {}^4T_{1g}$ (P), respectively. These transitions are characteristic of the octahedral geometry of the complexes **Table 3.3** [29]. The ligand field parameter values Dq, B, C, b, F_4, and F_2 were calculated. The electron-electron repulsion in the complexes is less than that in the free ion, resulting in an increased distance between electrons and thus an effective increase in the size of the orbital. On increasing delocalization the value of b decreases and is less than one in the complexes. The numerical value 786 cm^{-1} for B of free Mn(II) ion has been used to calculate the value of β. The calculated values of β Table 4 indicate that Mn(II) complex under study have appreciable covalent character.

The electronic spectra of the complexes under study display the absorption bands in the region 9876 cm^{-1}, 20560 cm^{-1} and 26790 cm^{-1} and 37548 cm^{-1} [30]. These transitions may be assigned to the following transitions: $^4T_{1g}$ (F)→$^4T_{2g}$ (ν_1), $^4T_{1g}$ (F)→$^4A_{2g}$ (ν_2), $^4T_{1g}$ (F)→$^4T_{1g}$(P) (ν_3) transitions, respectively. These transitions corresponding to the octahedral geometry of the Co(II) complex [31].

The absorption spectra of the Ni(II) complex displayed the d-d transition at positions 10170-10738 cm^{-1}, 15274-16070 cm^{-1} and 21265-22065 cm^{-1}. These bands might be assigned to the three spin allowed transition: $^3A_{2g}$ (F) → $^3T_{2g}$ (F) (ν_1), $^3A_{2g}$ (F) → $^3T_{1g}$ (F) (ν_2), $^3A_{2g}$ (F) → $^3T_{1g}$ (P) (ν_3). These transitions revealed that the nickel complexes possessed octahedral geometry [32]. The electronic spectra of Cu(II) complex shows absorption bands at positions 11211 cm^{-1}, 17322 cm^{-1} and 25851 cm^{-1} Figure 5. These bands correspond to $^2B_{1g} \leftarrow {}^2A_{1g}$ $(d_{x^2-y^2} \leftarrow d_{z^2})$ (ν_1), $^2B_{1g} \leftarrow {}^2B_{2g}$ $(d_{x^2-y^2} \leftarrow d_{xy})$ (ν_2) and $^2B_{1g} \leftarrow {}^2E_g$ $(d_{x^2-y^2} \leftarrow d_{xz}, d_{yz})$ (ν_3) d-d transition. These transitions revealed that Cu(II) complex possessed distorted six coordinated geometry *i.e.* tetragonal geometry [33]. The complex exhibit the high energy bands in the range 39646 cm^{-1} which was assigned to the L→M charge transfer bands.



(a)



(b)

FIGURE 5: Electronic spectra of (a) $[Mn(L)_2Cl_2]$ (b) $[Cu(L)_2Cl_2]$

TABLE 3: Magnetic moment and electronic spectral data of Mn(II), Co(II), Ni(II) and Cu(II) complexes with Schiff's base ligand

Complex	Magnetic moment μ_{eff} (B.M)	λ_{max} (cm^{-1})			
		υ_1	υ_2	υ_3	υ_4
$[Mn(L)_2Cl_2]$	5.82	18685	22973	24989	28924
$[Co(L)_2Cl_2]$	4.76	9876	20560	26790	37548
$[Ni(L)_2Cl_2]$	2.98	10576	15274	21790	37448
$[Cu(L)_2Cl_2]$	1.97	11211	17322	25851	39646

TABLE 4: Ligand field parameters of Mn(II), Co(II), Ni(II) and Cu(II) complexes with Schiff's base ligand

Complexes	Dq (cm-1)	B (cm-1)	C	β	F2	F4	hx
[Mn(L)2Cl2]	1868	386	3822	0.49	932	109.2	7.27
[Co(L)2Cl2]	987	881	-	0.54	-	-	-
[Ni(L)2Cl2]	1057	355	-	0.34	-	-	-

BIOLOGICAL ACTIVITY

Antifungal and antibacterial activities of ligand and its metal complexes are given in Tables 5-6. The obtained data confirmed that complexes possess high activity as compared to free ligands. Overtone's concept and Chelation Theory explain this activity enhancement of the free ligand after the complexation with metal ion [34]. Basically the metal ion charge is decreased to minimum, increases the lipophilicity of the complexes. This breaks the permeability barrier of the cell and hence retards the cell processes. Principally the increased lipophilic character of the complex favors the access of the complex through the lipid layer of the cell membrane and blocks the metal binding site in the enzymes of microorganisms. As a result of which the respiration process of the cell get disturbs and breaks the chain of protein synthesis [35].

TABLE 5: Antifungal activity data of ligand and its Mn(II), Co(II), Ni(II) and Cu(II) complexes

Compound	Concentration (ppm)	Fungal Inhibition (%)		
		A. niger	*M. phaseolina*	*P.glomerata*
Ligand (L)	1000	35	30	30
	750	25	20	20
	500	15	10	15
	250	NA	NA	NA
$[Mn(L)_2Cl_2]$	1000	90	80	95
	750	85	65	80
	500	50	45	65
	250	20	25	30
$[Co(L)_2Cl_2]$	1000	45	50	45
	750	35	35	35
	500	20	30	20
	250	15	10	15
$[Ni(L)_2Cl_2]$	1000	60	60	40
	750	50	55	25
	500	45	35	10
	250	25	10	NA
$[Cu(L)_2Cl_2]$	1000	75	80	85
	750	60	60	80
	500	45	45	45
	250	20	35	25
Standard Drug	1000	100	100	100
	750	95	95	95
	500	90	90	85
	250	85	75	80

TABLE 6: Antibacterial activity data of ligand and its Mn(II), Co(II), Ni(II) and Cu(II) complexes

Compound	Concentration (ppm)	Inhibition	
		E. coli	*P.aeruginosa*
Ligand (L)	1000	15	05
	500	05	NA
$[Mn(L)_2Cl_2]$	1000	25	30
	500	10	20
$[Co(L)_2Cl_2]$	1000	25	20
	500	20	15
$[Ni(L)_2Cl_2]$	1000	10	20
	500	05	15
$[Cu(L)_2Cl_2]$	1000	15	10
	500	NA	05
Standard Drug	1000	35	35
	500	30	30

CONCLUSION

The newly synthesized ligand acts as bidentate Schiff's base ligand. The analytical, spectral, magnetic, studies confirm the bonding of Schiff base to metal ions. Schiff base was found potentially active towards microbial strains (bacteria and fungi). The observations shows that the Mn(II), Co(II). Ni(II) and Cu(II) complexes have distorted octahedral geometry Figure .

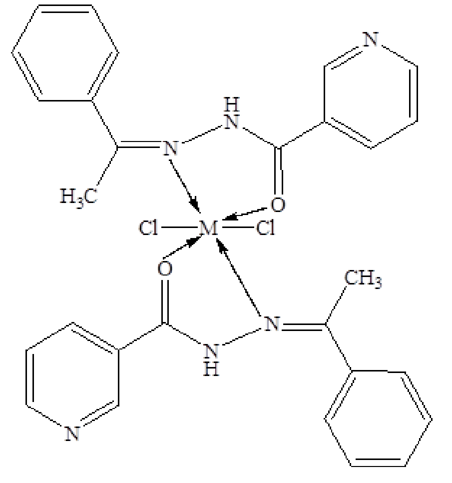


Figure 6: Proposed structure of metal complexes

Where M = Mn(II), Co(II), Ni(II) and Cu(II)

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