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Studies on synthesis of some schiff bases, metal complexes and their antimicrobial activity

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ABSTRACT

Schiff base ligand 2-hydroxy-5-chloro-benzylidene-4-chloro-aniline and 2-hydroxy-5-chloro-benzylidene-4-nitro-aniline and its complexes with Cu (II), Ni (II), Co (II) Zn (II) were prepared and characterized by analytical, spectroscopic (IR, UV-Vis) techniques, electrical conductivity and magnetic measurements. The complexes were further screened for antibacterial and antimicrobial activity. © 2010 Trade Science Inc. - INDIA

KEYWORDS

Schiff-base;
Metal complexes;
Antimicrobial activity.

INTRODUCTION

Metal complexes of Schiff bases have played a central role in the development of coordination chemistry. Various studies have shown that, the azomithine group ($>C=N-$) in Schiff base metal complexes has considerable biological significance^[1] and found to be responsible for biological activity such as fungicidal^[2] anti-cancer^[3] and anti-inflammatory activity^[4]. Application of these metal complexes promoted us to carry out the synthesis, characterization and antimicrobial studies of some transition metal Schiff base complexes.

EXPERIMENTAL

The ligand 2-hydroxy-5-chloro-benzylidene-4-chloro-aniline (L^1) 2-hydroxy-5-chloro-benzylidene-4-nitro-aniline (L^2) has been characterized by infrared spectral studies. The complex characterized by electronic spectral studies, magnetic moment and conductivity measurement. The antimicrobial activity of the investigated

compound was tested by the paper diffusion method.

Synthesis of schiff bases

The Schiff bases were synthesized by reported procedure^[5]. To the solution of 5-chloro-salicylaldehyde 0.157gm (0.001 moles) in 25ml ethanol and 0.127gm (0.001 mol) 4-chloro aniline in 25ml ethanol was added (for L^1) and 0.138gm (0.001 mol) 4-nitro aniline in 25ml ethanol was added (for L^2). The solutions were refluxed for 3 hrs. The solution were monitored on TLC, on completion of reaction the solution were cooled and poured in water the separated solid were filtered, washed with water and recrystallized from ethyl alcohol. The composition and yield of Schiff bases have shown in TABLE 1.

Synthesis of metal complexes

The metal complexes of Cu (II), Ni (II), Co (II) and Zn (II) with ligands were prepared by refluxing the ethanolic solution of metal nitrate and ligand in 1:2 molar ratios. The pH of solution was adjusted by alcoholic ammonia.

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TABLE 1 : Analytical data of ligand and their metal complexes.

Compound	Color	Yield (%)	Formula	Mol Wt.	% Analysis Found(Calculated)			
					C	H	M	N
(L ¹)	Yellow	68	C ₁₃ H ₉ Cl ₂ NO	266.12	58.55 (58.67)	3.49 (3.41)		5.32 (5.26)
(L ²)	Yellow Orange	65	C ₁₃ H ₉ ClN ₂ O ₃	276.68	56.31 (56.43)	3.19 (3.28)		10.03 (10.13)
(L ¹) ₂ Cu(H ₂ O) ₂	Brown	55	C ₂₆ H ₂₂ Cl ₄ CuN ₂ O ₄	631.82	49.51 (49.43)	3.47 (3.51)	9.78 (10.06)	4.36 (4.43)
(L ¹) ₂ Ni(H ₂ O) ₂	Yellow	57	C ₂₆ H ₂₂ Cl ₄ N ₂ NiO ₄	626.97	49.73 (49.81)	3.46 (3.54)	9.05 (9.36)	4.32 (4.47)
(L ¹) ₂ Co(H ₂ O) ₂	Pink	52	C ₂₆ H ₂₂ Cl ₄ CoN ₂ O ₄	627.21	49.66 (49.79)	3.49 (3.54)	9.10 (9.40)	4.32 (4.47)
(L ¹) ₂ Zn(H ₂ O) ₂	Orange	60	C ₂₆ H ₂₂ Cl ₄ N ₂ O ₄ Zn	633.67	49.35 (49.28)	3.43 (3.50)	10.12 (10.32)	4.35 (4.42)
(L ²) ₂ Cu(H ₂ O) ₂	Brown	61	C ₂₆ H ₂₂ Cl ₂ CuN ₄ O ₈	652.93	47.76 (47.83)	3.32 (3.40)	9.54 (9.73)	8.42 (8.58)
(L ²) ₂ Ni(H ₂ O) ₂	Brown	60	C ₂₆ H ₂₂ Cl ₂ N ₄ NiO ₈	648.07	48.11 (48.19)	3.31 (3.42)	8.81 (9.06)	8.52 (8.65)
(L ²) ₂ Co(H ₂ O) ₂	Brown	59	C ₂₆ H ₂₂ Cl ₂ CoN ₄ O ₈	648.31	48.09 (48.17)	3.35 (3.42)	8.79 (9.09)	8.56 (8.64)
(L ²) ₂ Zn(H ₂ O) ₂	Orange	54	C ₂₆ H ₂₂ Cl ₂ N ₄ O ₈ Zn	654.77	47.76 (47.69)	3.25 (3.39)	9.72 (9.99)	8.43 (8.56)

TABLE 2 : IR frequency (cm⁻¹) of ligand and complexes.

Compound	ν H ₂ O	ν O-H	ν C=N	ν C-O	ν M-N	ν M-O
L ¹	---	3019	1618.63	1277	---	---
L ¹ Cu	3200	---	1612.30	1383	533	437
L ²	---	3100	1595	1305	---	---
L ² Ni	3300	---	1600.71	1310	610	500

Ligand (0.02 mole) in slight excess was taken in a round bottomed flask containing 30ml anhydrous ethanol and refluxed for few minutes with constant-stirring to ensure complete dissolution, A solution of the appropriate metal nitrate (0.01 M) in 20ml of anhydrous ethanol was then added drop by drop with constant-stirring in the hot solution of ligand. The content was refluxed two hours. After cooling, the pH of the mixture was maintained by the addition of alcoholic ammonia solution. The resultant precipitate was digested for one hour. The precipitate was filtered, washed with hot ethanol (40°-60°C) and dried in vacuum desiccators over anhydrous granular calcium chloride. The composition and yield of metal complexes have shown in TABLE 1.

RESULT AND DISCUSSION

The metal complexes are colored and stable to air and moisture. They are insoluble in water, sparingly soluble in benzene, chloroform, carbon tetrachloride etc.

TABLE 3 : Antimicrobial activities of Schiff bases and their complexes.

compound	Zone of inhibition	
	Staphylococcus aureus	Escherichia coli
L ¹	11	10
L ¹ Cu	20	16
L ¹ Ni	14	10
L ¹ Co	12	11
L ¹ Zn	13	12
L ²	14	08
L ² Cu	19	18
L ² Ni	14	10
L ² Co	15	14
L ² Zn	16	13
Standard drug	26	30

and completely soluble in dimethyl formamide (DMF) and dimethyl sulfoxide (DMSO). The low conductivity value in DMF (1×10^{-4} M) reveals their non-electrolyte nature^[6,7].

In IR spectrum of ligands, shows band in the region 3100-3000cm⁻¹ assigned to -OH stretching, the lowering of normal free -OH stretching frequency from 3600cm⁻¹-3500cm⁻¹ to above value is expected due to the strong intramolecular hydrogen bonding^[8]. A bond at 1618 and 1627 in L¹ and L² respectively assigned to azomethine (C = N) stretching vibration.

A bond at 1277cm^{-1} and 1305cm^{-1} in L^1 and L^2 respectively assigned to enolic C-O stretching frequency.

In the spectra of complexes the band in region $3200\text{-}2900\text{cm}^{-1}$ disappears. The bands at 1618cm^{-1} and 1627cm^{-1} are shifts to lower frequency (1580 and 1612cm^{-1}), indicating the co-ordination of azomethine nitrogen to metal atom. In spectra of all complexes the broad band at $\approx 3300\text{cm}^{-1}$, indicating the presence of coordinated water. The bands at 1277cm^{-1} and 1305cm^{-1} show upward shift (1310 and 1383cm^{-1}) suggest the banding of enolic oxygen of Schiff base with metal during complexation^[9,10]. The new bands in the region $450\text{-}500\text{cm}^{-1}$ and $550\text{-}610\text{cm}^{-1}$ in the spectra and complexes are assigned to stretching vibration of M-O and M-N bands respectively^[11].

The UV-Visible spectra of copper complexes of L^1 and L^2 respectively exhibits bands at 15064cm^{-1} , 25510cm^{-1} and 15492cm^{-1} , 27777cm^{-1} attributed to ${}^3E_g \rightarrow {}^2T_{2g}$ and charge transfer transition which indicate distorted octahedral geometry of Cu(II) complexes, which further supported by μ_{eff} value 1.80 and 1.98 B.M.^[12] respectively.

The electronic spectra of Ni(II) complexes exhibits bands in the three region as 9661cm^{-1} , 15948cm^{-1} and 24271cm^{-1} and 9523cm^{-1} , 16129cm^{-1} and 24390cm^{-1} for L^1 and L^2 respectively. This pattern of absorption may be assigned to transition ${}^3A_{2g} \rightarrow {}^3T_{2g}$ (F), ${}^3A_{2g} \rightarrow {}^3T_{1g}$ (F) and ${}^3A_{2g} \rightarrow T_{1g}$ (P) respectively. Which may be assigned to characteristic three spin allowed transition of octahedral complexes^[13], further supported by μ_{eff} value 3.1 and 3.2 B.M. respectively.

The electronic spectra of Co(II) complexes exhibits bands at 9276cm^{-1} , 17699cm^{-1} , 21367cm^{-1} and 9389cm^{-1} , 17889cm^{-1} and 22471cm^{-1} for L^1 and L^2 respectively. These bands may be assigned to ${}^4T_{1g}$ (F) \rightarrow ${}^4T_{2g}$, ${}^4T_{1g}$ (F) \rightarrow ${}^4A_{2g}$, (F) and ${}^4T_{1g}$ (F) \rightarrow ${}^4T_{1g}$, (p) respectively suggesting an octahedral geometry around cobalt ion^[14] further supported by μ_{eff} value 4.80 and 4.92 B.M. respectively.

The electronic spectra of Zn(II) complexes exhibits bands at 25510cm^{-1} , 25125cm^{-1} for L^1 and L^2 respectively, attributed to charge transfer transition suggesting an octahedral environment^[15]. These complexes are diamagnetic in nature.

Antimicrobial activity

The ligands L^1 and L^2 and their metal complexes were tested for antibacterial activity Mueller Hinton agar was used for testing the susceptibility of microorganism by paper disc diffusion method, using DMSO as solvent, at a concentration of 0.01M against gram positive (staphylococcus aureus) and gram. negative (Escherichia coli) bacteria.

The zones inhibition against the growth of microorganisms was determined at the end of on incubation period 24 h at 37°C and the results are presented in TABLE 3. It was found that the metal complexes are more active than the free ligand.

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