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SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL STUDIES OF COBALT COMPLEXES WITH BIDENTATE SCHIFF BASES

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ABSTRACT

Ligation properties of Schiff bases derived from 2- aminophenol and substituted aldehydes have been investigated for this purpose, cobalt complexes of these are synthesized and characterized on the basis of elemental analysis, magnetic, thermal and spectral studies. The complexes conform to 1:1 stoichiometry and are non-electrolyte in nature. The coordination of the ligand to the metal ions is found to be through azomethine nitrogen and phonolic oxygen of the Schiff bases in the octahedral environment. All the cobalt complexes show antimicrobial activity.

Key wards: *Schiff base, Cobalt, Spectral studies, Biological activity.*

INTRODUCTION

There is a considerable interest in the coordination chemistry of Schiff bases with various metal ions, partially due to their capability of acting as multidentate N-N and N-O donors with the formation of mono or polynuclear complexes[1,2]. Metal complexes of Schiff bases have been extensively studied due to their synthetic flexibility, selectivity and sensitivity towards the central metal atom. The chemistry of Schiff base complexes continues to attract many researchers because of their applications in various fields like food and dyes industry[3], analytical chemistry[4], catalysis[5] and biological studies[6]. It was therefore, proposed to investigate the ligation properties of Schiff bases derived from 2- aminophenol with substituted aldehydes and their complexes with cobalt ion.

EXPERIMENTAL

All the chemical and solvents used were of A.R. grade. Elemental analysis was done on Perkin Elmer elemental auto analyzer and CHNS thermoquest auto analyzer. IR spectra were recorded on Bomem MB 104 FTIR spectrophotometer. ^{1}HNMR spectra were recorded on Brucker FT 300 at 300 MH$_z$ NMR spectrometer in $CDCL_3$+ $DMSOd_6$ Solvent, using TMS as an internal standard. Magnetic measurements were carried out by the Gouy method at room temperature. The magnetometer was calibrated using Hg (Co $(NCS)_4$). Diamagnetic corrections were made by using Pascal's constants. The TGA, DTA were recorded on 'Mettler Toledo Star System' at RSIC Nagar University, Nagpur. The analytical data are presented in table.

Synthesis of Schiff bases:

The ligands, Schiff bases, were synthesized as a mixture of 2-aminophenol (0.1mole) and substituted benzaldehyde (0.12mole) dissolved in dry ethanol and conc. H_2SO_4 (2-3 drops), was refluxed for 5-6 hours. It was then cooled and diluted with ice cold water and crystallized from ethanol / ethanol water mixture. The purity was checked by M.P. and TLC technique. Further the structures of the Schiff bases were conformed by subjecting them to 1R, NMR and mass spectral studies.

Synthesis of Metal Complexes:

The metal chlorides and Schiff bases in the molar ratio of 1:2 were dissolved in ethanol. The PH of the resulting solution was adjusted just below the value of hydrolysis of the respective metal ion, using alcoholic ammonia. The reaction mixture was refluxed for 3.4 hours while stirring continuously. The precipitate formed was separated by filtration and washed with hot ethanol followed by pet. ether (40-60^{0}c) and dried in vacuo.

RESULTS AND DISCUSSION

The complexes have different colors and seem to be semi crystalline. All the cobalt complexes are stable in air for extended periods of time. The solids do not melt sharply and undergo decomposition. These are insoluble in water and soluble in organic solvents such as DMF, DMSO giving respective colors to the solutions. All compounds gave satisfactory elemental analysis. Values are in close agreement with the values calculated for expected molecular formulae assigned to these complexes, suggesting 1:1 stoichiometry. The molar conductance values of all the complexes in DMSO reveal their non electrolytic nature.

The IR spectra of the free ligands show an – OH absorption band in the region 3325-3419 cm^1. This sharp band has disappeared in the complexes, indicating its involvement in the bond formation process[8]. The strong band at 1618 – 1625 cm^1 due to C=N stretching frequency is lowered by 09-25 cm^1 in the spectra of the complexes, indicating coordination through azomethine nitrogen of the Schiff bases[9,10] .The new bands appearing in the region 400- 418 and 500- 599 cm^1 may probably due to coordinated water molecule and the formation of ν(M-N)and ν(M-0) bonds respectively[11,12]. The broad absorption band observed in all the complexes in the region 3200- 3556cm^1 could be attributed to the lattice water present in the metal complexes.

The ^{1}HNMR spectra of ligands A, B, C, D, E and F have been recorded in $CDCl_3$. In all the ligands – OH group appeared in the range 6.95 to 8.15 δ ppm. While C=N group at 8.65 to 9.10 δ ppm. In ligands E and F, the- OCH_3 and $–CH_3$ protons appear at 3.8 and 2.4 δ ppm . respectively. The rest of the proton appeared in the aromatic region at 6.5 to 8.0 δ ppm. An examination of NMR spectra of the ligand reveals that these are on expected lines according to their structures. A comparison of these spectra with those of cobalt complexes would have given a clear picture of the changes taking place in the ligands.The ^{1}HNMR spectra of the complexes were, therefore, recorded. Unfortunately, however, due to the presence of a metal ion, proton resonance was not effected and one could observed only broad peaks indicating the formation of the complexes.

In the mass spectra of the ligands, probable fragmentation into certain structural units was observed for the ligands under investigations as Ligand **A** M^+197, 180,120(100%) ,104., **B** M^+ 231 (having isotopic peaks), 196, 167, 120, (100%) 93., **C** M^+ 231(100% with isotopic peaks), 196,167,120,93., **D** M^+ 242, 195, 167, 120 (100%), 93., **E** M^+ 210, 120(100%), 91,77., **F** M^+ 227(100%), 205, 183, 120, 77.

The mass spectral data of ligands shows that the theoretical molecular weight calculated which matches with the experimental molecular weight of the ligands.

In the present investigation, the presence of water molecules in the cobalt complexes as indicated in the IR spectra was as certained and verified by the thermal analysis. The thermal studies indicate that complexes decompose after dehydration.

The thermogram showed a weight loss in the range 45-91^{0}c this could be attributed to the loss of absorbed moisture. Further loss up to 490^{0}c could be due to the loss of coordinated water and chloride molecules. The loss thereafter up to 540^{0}c indicated the loss of ligand and finally the complex is converted into its metal oxide[13,14].

The magnetic moments at room temperature for the complexes under study are given in table. The magnetic moments of the cobalt complexes under present investigation are in the range 4.64-5.21 B.M. These values agree with the previously reported experimental values in range 4.70-5.20 B.M. for high spin octahedral cobalt complexes[15] .

The molar conductance of 10^{-3}M solutions in DMSO at room temperature of the complexes has been measured and the values are reported in table. These values were found to be ranging between 25.905 - 30.135ohm^{-1}. cm^2 mol^{-1} and values indicate that all the complexes are nonelectrolytes[16,17].

The ligands and their cobalt complexes were screened for their antibacterial and antifungal activity in vitro against staph. Aureus 209p, E.coli ESS 2231, Aspergillus fumigatus, Candida albicans, Candida albicans ATCC 10231, Candida Krusei GO 3 and Candida glabrata HO5, using standard agar well diffusion assay method[18]. The Co (II) complexes showed moderate antimicrobial activity. From the result one is tempted to conclude that though the ligands are not effective against these microbes their metal complexes seem to have developed a fair antimicrobial activity. Such enhanced antimicrobial activity in the metal complexes was reported earlier[19].

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Table-1 :Analytical data of the ligands and complexes

Sr. No.	**Compound**	**Molecular formula (Molecular weight)**	**Color**	**% Metal Found (Calculated**	**Elemental Analysis.**				**Molar Conductance λm**	**Magnetic Susceptibility χ_g B.M.**
					%C	**%H**	**%N**	**%Cl**		
1.	Ligand A	C_{13} H_{11} NO (197.236)	Pale yellow		79.10 (79.16)	5.58 (5.62)	7.02 (7.10)	--	---	---
	Co (II) – A	Co(II) $C_{13}H_{11}NOCl_2 2H_2O$ (363.197)	Reddish brown	16.125 (16.226)	42.790 (42.991)	2.913 (3.053)	3.702 (3.857)	19.351 (19.552)	26.050	4.642
2.	Ligand – B	C_{13} H_{10} ClNO (231.680)	Greenish yellow	--	67.34 (67.40)	4.32 (4.35)	5.98 (6.05)	14.93 (15.30)	--	--
	Co (II) – B	Co(II) $C_{13}H_{10}ClNOCl_2$ $2H_2O$ (397.547)	Chocolate brown	14.792 (14.824)	39.183 (39.277)	2.485 (2.535)	3.413 (3.523)	17.715 (17.835)	24.435	4.918
3.	Ligand –C	C_{13} H_{10} ClNO (231.680)	Greenish yellow (faint)	--	67.32 (67.40)	4.31 (4.35)	5.98 (6.05)	14.93 (15.30)	--	--
	Co (II) – B	Co(II) $C_{13}H_{10}$ $ClNOCl_2$ $2H_2O$ (397.547)	Reddish gray	14.792 (14.824)	39.183 (39.217)	2.485 (2.535)	3.413 (3.523)	17.715 (17.835)	30.135	4.718

4.	Ligand D	$C_{13}H_{10}N_2O_3$ (242.263)	Light yellow	---	67.38 (67.46)	4.11 (4.16)	11.50 (11.56)	--	---	---
	Co (II) – D	Co(II) $C_{13}H_{10}N_2O_3Cl_2$ $2H_2O$ (408.099)	Reddish brown	14.303 (14.441)	38.123 (38.261)	2.312 (2.470)	6.698 (6.864)	17.212 (17.374)	29.438	4.815
5.	Ligand – E	$C_{14}H_{13}NO$ (211.263)	Yellowish brown	--	79.54 (79.59)	6.15 (6.20)	6.59 (6.63)	---	--	--
	Co (II) – E	Co(II)$C_{14}H_{13}NOCl_2 2H_2O$ (377.130)	Reddish brown(dark)	15.512 (15.627)	44.435 (44.588)	3.312 (3.474)	3.612 (3.714)	18.710 (18.801)	27.530	4.642
6.	Ligand –F	$C_{14}H_{13}NO_2$ (227.264)	Yellow green	--	73.95 (73.99)	5.72 (5.77)	6.11 (6.16)	---	--	--
	Co (II) – F	Co(II)$C_{14}H_{13}NO_2Cl_2 2H_2O$ (393.129)	Reddish brown	14.883 (14.991)	42.682 (42.773)	3.238 (3.333)	3.403 (3.563)	17.968 (18.036)	25.905	5.206

Ligand A: N- (Benzilidine) – 2 Hydroxyaniline

Ligand B: N- (2’ Chlorobenzilidine) – 2 hydroxyaniline

Ligand C: N- (4’ Chlorobenzilidine) – 2 hydroxyaniline

Ligand D: N- (4’ Nitrobenzilidine) – 2 hydroxyanline

Ligand E: N- (4’ Methylbenzilidine) – 2 hydroxyaniline

Ligand F: N- (4’ Methoxybenzilidine) – 2 hydroxyaniline