

**SYNTHESIS, CHARACTERISATION, THERMAL AND ANTIMICROBIAL
STUDIES ON MANGANESE, COBALT, NICKEL, COPPER AND ZINC
COMPLEXES**

R.G Mahale¹, R.B.Mohod²

1. S.S.V.P.S^s.L.K Dr.P.R.Ghogrey Science College Dhule. (Maharashtra).

2. Shri Shivaji Arts, commerce and Science College Akola. (Maharashtra).

ABSTRACT

Metal complexes of Mn(II), Fe(II), Co(II), Ni(II), Cu(II), and Zn(II) with Schiff base ligand derived from 2-hydroxy-5-chloroacetophenone and S-benzyldithiocarbazate have been synthesized. Isolated complexes have been characterized by elemental analysis, IR, Reflectance Spectra, Magnetic measurement, thermal analysis and antimicrobial studies. The copper(II) complex behave a subnormal magnetic moment due to antiferromagnetic exchange interaction while normal magnetic moments at room temperature IR and H¹-NMR spectral studies show that the Schiff base behave as a dibasic and tridentate ligand coordinated through the deprotonated phenolic oxygen, azomethine nitrogen and thioenoilicsulphur. The thermogravimetric data have been analyzed for kinetic parameters using Freeman-Carroll and Sharp-Wentworth method. The antibacterial activities of the ligand and its complexes have been also been screened against various organism.

Keywords: Metal complexes, TGA, Antimicrobial activity

INTRODUCTION

Metal complexes of schiff bases represent an important and interesting class of coordination compound. During the past few years, much attention has been focused on the synthesis of compartmental ligands capable of bringing two or more metal atoms into close proximity. However little attention has been paid to the system in which schiff base derived from 2-hydroxy-5-chloroacetophenone and S-benzylthiocarbamate. Hence it was thought worthwhile to prepare Mn(II), Fe(II), Co(II), Ni(II), Cu(II), and Zn(II) complexes. Their structures have been determined by elemental analysis, magnetic studies, electronic and infra-red spectral data and anti-microbial properties.

MATERIALS AND METHODS

All the chemical and solvents were of AR grade and used as received S-benzylthiocarbamate and 2-hydroxy-5-chloroacetophenone were prepared by literature methods.

Synthesis of schiff base:

An ethanolic solution (10 cm³) of 2-hydroxy-5-chloroacetophenone (5 mmole) was added to a hot ethanol solution (10 cm³) of the S-benzylthiocarbamate (5 mmole). The mixture was heated under reflux on a water bath for 3 hrs and the mixture then cooled in an icebath. The separated dark yellow solid was filtered under reduced pressure recrystallized from ethanol and dried in vacuum yield 62% M.P 237⁰C. The crystallized ligand showed the expected elemental composition infra-red and H¹-NMR spectra.

Preparation of metal complexes:

All the complexes isolated were prepared by the following general method. A methanol solution (20 cm³) of the schiff base (4 mmole) was added to a methanol (20 cm³) solution of appropriate metal acetate (4 mmole) with constant stirring and the mixture was heated under reflux for 2hrs. The complexes were formed immediately after addition of 1 gm of sodium acetate. The product was filtered and washed with hot water and then with hot methanol/ethanol. The compounds were dried under vacuum over silica gel at room temperature. Elemental analyses performed micro analytically. The metals were estimated by

oxide method magnetic susceptibility measured by a Gouy set up at room temperature using mercury tetrathiocyanatecobalate (II) as calibrating agent and calculation were made using computed values of Pascal's constant, IR spectra (KBr) were recorded on a Perkin-Elmer spectrophotometer and electronic spectra on a Karl-Zeiss, VSU-2P spectrophotometer using MgO as calibrant. Thermo gravimetric analysis was recorded on TGS-2 system in air with heated rate of 10^0 min^{-1} . Antimicrobial activity of ligand and complexes were studied by agar disc method using DMF as control.

RESULTS AND DISCUSSION

All the complexes are coloured solid and soluble in water and common non-coordinating solvents but soluble in coordinating solvents like DMF and DMSO. The elemental analysis of the complexes showed that the metal ligand stoichiometry is 1:1 in all the complexes (Table 1).

The infrared spectra of the ligands shows an intense C=S band at 1029 cm^{-1} and absence of band near 2580 cm^{-1} (S,H), which indicates that the ligand remains in thione form at least in the solid state. A band at 3197 cm^{-1} (N,H) in the ligands disappears in the complexes as this secondary amine nitrogen is thioenolization and consequent deprotonation on coordination. Further the ligand exhibits the C=O (Phenolic) stretch at 1287 cm^{-1} . A broad band at 2923 cm^{-1} is assigned to hydrogen bonded phenolic -OH group. A strong absorption band at 1595 cm^{-1} is due to C=N stretching frequency. The band near 956 cm^{-1} is due to N-N (S) and sharp absorption band at 770 cm^{-1} for C=S stretching frequency. The complexes show strong band at 3380 cm^{-1} as sign to uncoordinated fourth position phenolic -OH group.

The Co(II), Ni(II), Fe(II), and Mn(II) complexes exhibit magnetic moments of 4.95, 3.05, 5.20 and 5.83 B.M respectively consistent with high spin and Octahedral structure. The electronic spectra of the Co(II) complexes contains band at 8790, 16660, 19810 cm^{-1} due to the transition ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(F)$, ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$, and ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$ respectively. The Ni(II) complexes shows band at 10869, 46583 and 24815 cm^{-1} due to the transition

${}^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$, ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ and ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ respectively in an octahedral symmetry spectra of Mn(II) complex shows three band 15873, 17857 and 22277 cm^{-1} due to transition ${}^6A_{1g} \rightarrow {}^4T_{1g}(G)$, ${}^6A_{1g} \rightarrow {}^4T_{2g}(G)$ and ${}^6A_{1g} \rightarrow {}^4A_{1g}$, ${}^4A_{1g}(G)$ respectively.

The Fe(II) shows broad band at 140752 cm^{-1} may be assigned to ${}^5T_{2g} \rightarrow {}^5E_g$ transition in octahedral field. The Zn(II) complexes are found to be diamagnetic as expected for d^{10} configuration.

The elimination of water molecules takes place in the first stage. In the present study Co(II), Ni(II), and Cu(II) complexes exhibits the weight loss near $150\text{-}190^\circ\text{C}$ equal to near 7.94, 12.10 and 5.10% respectively which corresponds to two, three, and one coordinated water molecules per atom, there after the non-coordinates part of the ligand in decomposed the followed by the decomposition of actually coordinated chromophore residue. Above 590°C temp. TG curve attains a constant level corresponding to their metal oxides Co_3O_4 , CuO .

The ligand and its metal complexes were screened against the bacteria *E.coli*, *Klebsiella*, *S.aureus* and *B.Subtilis* at a concentration of $50\mu\text{g/ml}$ by disc diffusion technique. The DMF was used as control. Ligand is found to exhibit moderate sensitivity against all the bacterial strains except *Klebsiella*. Ni(II) complex shows moderate zone of inhibition against *E.coil* and *Klebsiella* and bacteriostatic towards all the other bacteria Mn(II) and Zn(II) complex exhibits bacteriostatic behaviors towards all the bacteria strains used Fe(II) complex show less sensitivity towards *E.coil* and B.T while other exhibits bacteriostatic against *Klebsiella* and *S.aureus*. The Ni(II), Cu(II) complexes show moderate sensitivity against all the bacterial strains.

Table 1 Analytical Data of metal Complexes of HASBZ

Proposed composition of metal complexes	Metal % found (Caled.)	C % found (Caled.)	H% found (Caled.)	N% found (Caled.)	S% found (Caled.)	CL% found (Caled.)
$[M_n(\text{HASBZ})_2 \cdot 2\text{H}_2\text{O}]$	13.50 (13.58)	47.28 (35.68)	3.61 (3.70)	6.88 (9.90)	15.72 (15.80)	8.61 (8.64)
$[F_e(\text{HASBZ})_2 \cdot 3\text{H}_2\text{O}]$	13.70 (13.79)	47.19 (47.29)	3.60 (3.69)	6.81 (6.89)	15.63 (15.73)	8.56 (8.66)
$[C_o(\text{HASBZ}) \cdot 2\text{H}_2\text{O}]$	14.31 (14.42)	45.89 (46.90)	3.58 (3.66)	6.73 (6.84)	15.51 (15.64)	8.52 (8.55)
$[N_i(\text{HASBZ}) \cdot 3\text{H}_2\text{O}]$	14.58 (14.63)	46.72 (46.80)	3.60 (3.65)	6.78 (6.82)	15.53 (15.60)	8.50 (8.63)
$[C_u(\text{HASBZ})_2 \cdot 2\text{H}_2\text{O}]$	15.20 (15.25)	44.38 (44.48)	3.51 (3.63)	6.66 (6.77)	15.40 (15.49)	8.39 (8.47)
$[Z_n(\text{HASBZ})_2 \cdot \text{H}_2\text{O}]$	15.52 (15.66)	46.19 (46.26)	3.56 (3.61)	6.70 (6.74)	15.38 (15.42)	8.40 (8.53)



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