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ANTIMICROBIAL STUDIES OF Cu,Ni,Co METAL COMPLEXES OF 5-(2'-HYDROXYMINO-1'-PHENYL) ETHYLIDINE PHENYL 2,4,-DITHIOBIURETS

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Abstract:- ***The work reported here deals with the synthesis and spectroscopic studies of Cu(II),Ni (ll)and Co(ll) complexes with poly dentate Schiff bases are reported here. These Schiff bases were derived by condensing carbonyl oximes-like isonitrosoacetophenone, HINAP and 2,4-dithiobiurets. The characterization of the complexes was done on the basis of elemental analysis, molar conductivity, spectral IR, electronic thermal analysis spectra. The metal complexes have been screened for their antimicrobial activity against laboratory grown cultures of Escherichia coli, S.aureus, B.cereus, Bacillus subtilis. This provided information of value in guiding and understanding their therapeutic use. The concentration required to inhibit various organisms and the effects of environmental conditions on their activity are of paramount importance. The complexes show moderate activity against (11-22 mm) against the above mentioned bacteria. This shows that besides the complex formation, the nature of the metal ion has important role in the inhibition of the pathogenic activity of the bacteria. The analytical data suggests that the complexes can be formulated as*** $M_2L_2Cl_2.xH_2O$ ***where M=Cu,Ni,Co. The conductivity measurement indicates that they are non-electrolyte in nature. The complexes are thermally stable, insoluble in water but soluble in organic solvent to varying extent. The room temperature magnetic susceptibility measurements indicate their octahedral geometry which is further supported by electronic absorption /diffuse reflectance spectra.***

Keywords: Cu(II),Ni(ll),Co(ll) complexes; Schiff bases; Structural analysis, Antimicrobial studies.

I. INTRODUCTION

Schiff bases and their coordination compounds have gained importance recently because of their application as models in biological, biochemical, and analytical, antimicrobial system, anticancer, antibacterial and antifungal activities. Studies of new kinds of chemotherapeutic Schiff bases [1] are now attracted the attention of biochemists. Schiff bases contain azomethine (>C=N) group as functional group [2] and hence act as an effective ligand. In addition the presence of nitrogen and oxygen donor atoms in the complexes act as stereospecific catalyst for many reactions like oxidation, reduction, hydrolysis and possess antibacterial [6] activity. In this paper, the Synthesis and characterization of Cu,Ni,Co, complexes of 5-(2'-hydroxymino-1'-phenyl) ethylidine phenyl 2,4,-dithiobiurets is studied.

II. MATERIALS AND METHODS

All chemicals used were of A. R. grade purchased from S. D. Fine chemicals (Mumbai) & used without further purification. Distilled solvents were used throughout the experiments. Metal content was determined in the laboratory by the reported methods [7]. C, H and N analysis were performed at the IIT Mumbai. The infra-red spectra of the ligands and of their metal complexes were recorded in KBr pellets in the 4000-400 cm-1 region using a FTIR spectrum one supplied by Perkin Elmer instrument. The electronic spectra were recorded on Beckman Spectrophotometer the [5] diffuse reflectance spectra of solid complexes taken on Carl-Zeiss VSU -2P spectrophotometer. The 1H NMR spectra were recorded on a VXR-300s Varian Super Nuclear Magnetic Resonance spectrophotometer using TMS as an internal standard. TGA analysis was carried out [3] using a Shimadzu DT-30 recording thermal analyzer in an inert atmosphere of nitrogen, from room temperature to 9000c.

Antimicrobial studies: The in-vitro biological screening effects of the investigated compounds were tested against the bacteria Escherichia coli, Staphylococcus, Bacillus cereus, Bacillus subtilis were studied on laboratory grown cultures. Stock solutions were prepared by dissolving the compounds in DMSO and serial dilutions of the compounds were prepared in sterile distilled water to determine the minimum inhibition concentration (MIC). The nutrient agar medium was poured into Petri plates. A suspension of the tested microorganism (0.5 ml) was spread over the solid nutrient agar plates with the help of a

spreader. Different dilutions of the stock solutions were applied on the 10 mm diameter sterile disc. After evaporating the solvent, the discs were placed on the inoculated plates. The Petri plates were placed at low temperature for two hours to allow the diffusion of the chemical and then incubated at a suitable optimum temperature for 30-36 hrs [4]. The diameter of the inhibition zones was measured in millimetres.

III. EXPERIMENTAL

The ligands 5-(2'-hydroxyimino-1'-phenyl) ethylidine 1-phenyl 2, 4-dithiobiurets i.e.

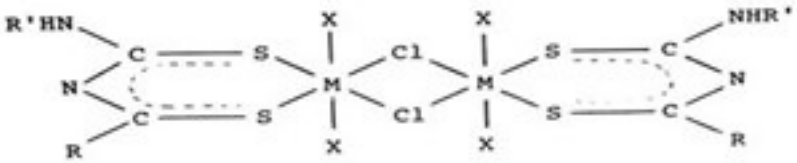
HPEPDTB were synthesised by refluxing a mixture of HINAP and substituted 2,4-dithiobiurets in alcohol for four hours. The mixture was then poured onto crushed ice when yellow coloured ligands separated which were filtered, washed with hot water, dried and recrystallised from alcohol[8]. The metal complexes of these two ligands were prepared by mixing equimolar solutions of ligands in alcohol and the metal chloride in 50% alcohol and raising the pH to 8.0. The mixtures were left overnight. The solid chelates obtained were filtered, washed thoroughly with water and 50% alcohol and dried in vacuum.

IV. RESULTS AND DISCUSSION

All the metal complexes are intensely coloured. They are thermally quite stable, as shown by their high decomposition temperatures, which indicate strong metal to ligand bonding solids. The complexes are insoluble in water, ethanol, methanol, chloroform, carbon tetrachloride etc. but soluble in DMF and DMSO. The complexes dissolve in alkali like sodium hydroxide giving colour, indicating the presence of a free oxime group, suggesting oximino proton is not replaced during complexation. The elemental analysis show 1:1 ligand metal stoichiometry for all the complexes. The analytical data along with some physical properties of the ligand and metal complexes are compared in this paper. The molar conductivities of 1 x 10^{-3} mhos cm^2 mol^{-1} solutions of the complexes in nitro benzene indicate their non-electrolytic nature. The elemental analysis suggest 1:1 (metal: ligand) stoichiometry for all the metal complexes.

They are formulated as $M_2L_2Cl_2$ .xH2O. Here L represents HPEPDTB and M =Co(II),Ni(II) where x=4 except for M=Cu(II) where x=0. On the basis of the elemental analysis and results

of various physicochemical studies, the bonding in the chloro metal complexes of HPEPDTB can be represented as follows:-



R = -N=C(C_6H_5)-CH(=NOH)
R'= phenyl
X = H_2O , when M = Co(II), Ni(II),
X = Nil when M = Cu(II)

Antimicrobial Activity

The result of the preliminary study on antimicrobial activity of complexes screened against B.subtilis, B.cereus, E.coli and S.aureus are tabulated in following Table-I.

Table-I

COMPOUND	B.subtilis(mm)	B.cereus(mm)	S.aureus(mm)	E.coli(mm)
$Cu_2L_2Cl_2$	17	14	17	16
$Ni_2L_2Cl_2.4H_2O$	16	11	22	16
$Co_2L_2Cl_2.4H_2O$	12	13	12	16

The test for complexes carried out in DMF solution using the agar cup method with DMF as blank control. They show resistance to most of the complexes. S.aureus and E.coli are fair to moderately sensitive to Co(II) complexes. They show varying action towards these bacteria. These metal complexes were assayed for their ability to inhibits the growth of micro organisms. The Cu(II) complexes show moderate activity against B. *subtilis.* Result indicate the micro organisms general resistance to most of the complexes of Schiff base ligands derived from 2,4-Dithiobiuret. Maximum antimicrobial activity is shown by Ni(II) complexes against B S.aureus. In conclusion, the studies on antimicrobial activity of metal

complexes indicate that constitution of ligands, its coordination to the metal ion, and the nature of metal ion in the complex have important influences on antimicrobial activity.

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