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Synthesis, Characterization and Biological Activity of Schiff Bases Chromium (III) Metal Complexe

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ABSTRACT

The Schiff base ligands were prepared from the Salicyladehyde and 5-amino-1,3,4-thiadiazole-2-thiol derivatives and its new metal complexes of Cr(III), have been Synthesizes successfully in a alcoholic medium. Metal complexes is analyzed by IR,Uv-Vis,Magnetic susceptibility, Molar Conductance, elemental analysis. The metal complexes antifungal activity against tested against *A. Niger* and *F. Oxysporum* and antibacterial activity against *S. aureus* and *B. subtilis* in order to assess their antimicrobial potential using Kirby-Bauer disc diffusion method.

Keywords : IR, Uv-Vis, Magnetic, the Salicyladehyde, *F. Oxysporum, B. subtilis*

I. INTRODUCTION

Schiff bases and their complexes are considered as privileged class of compounds due to their biochemical synthesis, electrochemical analysis, anti-fungal, antiviral, anti-malarial, anti-inflammatory, as well as, catalytic activities[1,2]. Schiff bases are also of interest in industrial fields and as a corrosion inhibitor, thermostable materials, as well as, powerful ligands in the formation of coordination compounds.[3] Recently, Al Zoubi and co-workers have reported the synthesis and structural characterization of Schiff bases and their complexes derived from different amines.[4] These compounds exhibit antimicrobial activity against several Grampositive and Gram-negative bacteria.

Schiff bases represent an important class of compounds because they are utilized as starting materials in the synthesis of industrial products [5]. Moreover, Schiff base is regarded as a ligand [6]. Due to their capability to form complexes with different transition metals Schiff bases can act as catalysts for different reactions [7-11]. The cross linking agents can also be derived from metal complexes with O-N- or -S ligand. For example, the intra-coordination salt such as salicylates, anthranilates and aliphatic or aromatic amines can form strong five or six membered chelates rings, which are able to produce the metal containing cross linking agents with required properties [12]. It is well known fact that N atom plays a key role in the coordination of metals at the active sites of numerous metallobiomolecules [13]. Metallorganic chemistry is becoming an emerging area of research due to the demand of new metal based antibacterial and antifungal compounds [14-15].

Chromium Schiff base complexes has received much attention because of the +3 oxidation state of the chromium metal ion, since the +3 oxidation state

of chromium ion is more stable with d^3 configuration and less toxic than +6 chromium ion [16-17]. Chromium metal has a vital role for maintaining the glucose tolerance factor (GTF) in the normal carbohydrate and lipid metabolism. Insufficient amount of chromium (III) ion consumption may lead to type II diabetes and cardiovascular diseases [18].

The aim of the present work is to synthesize,characterize metal complexes of Chromium and evaluate their antifungal properties against various pathogenic strains of fungi *A. Niger* and *F. Oxysporum* and antibacterial activity against *S. aureus* and *B.subtilis.*

II. MATERIALS AND METHODS

All the chemical of analytical grade. All salts are metal nitrates i.e. $Cr(NO_3)_3.9H2O$ (Sigma-Aldrich) were purchased from Sigma-Aldrich 3,5-dichloro-2-hydroxybenzaldehyde and 5-amino-1,3,4-thiadiazole-2-thiol from Sigma-Aldrich and Alfa Aesar used with-out further purification. Distilled Ethanol used for synthesis of metal complexes and ligand diethyl ether (Sigma-Aldrich). IR Spectra recorded on Perkin Elmer Spectrometer in range 4000-400 cm^{-1} KBr pellets. 1H and 13CNMR Spectra were recorded on BRUKER AVANCE III HD NMR 500 MHz spectro-photometer.Room Temperature magnetic moments by Guoy's method in B.M.Electronic Spectra using DMSO on Varian Carry 5000 Spectrometer. Molar Conductance measurements in dry DMSO having 1×10^{-3} concentration on Systronics conductivity bridge at room temperature. Elemental analysis (C, H, N) were carried out by using perkin Elmer 2400 elemental analyzer. Mass Spectra were recorded on Bruker IMPACT HD.

BIOLOGICAL ACTIVITY: Metal complexes evaluated in vitro their antibacterial activity against two Gram-Positive bacteria,viz, *B. Subtilis*; *S. aureus,* Two fungal strains *A. niger* and *F. oxysporum* by Kirby-Bauer disc diffusion method.The fungal and bacterial strains sub-cultured on PDA and Nutrient Agar. The stock solution (1 mg mL-1) was pre-pared in DMSO solution. The stock solution again diluted by using sterilized water to dilution in 500 ppm. The bacteria were subculture in agar medium and disc were kept incubated for 37oC at 24 hrs. The standard antibacterial drug Miconazole and Ciprofloxacin was also screen under same condition for comparison. Activity was measure and calculated by zone of inhibition (mm) surrounding discs. The experimental value compare with standard drug value Miconazole for the Antifungal activity and Ciprofloxacin for the antibacterial activity.

SYNTHESIS OF SCHIFF BASE LIGAND: The mixture of 1:1 3,5-dichloro-2-hydroxybenzaldehyde (1.91g,0.01mol) with 5-amino-1,3,4-thiadiazole-2-thiol (1.33g, 0.01 mol) dissolved in ethanol. Then add Few drops of glacial acetic acid was added .The resultant mixture stirred for 3-4 hrs the colored precipitate of Ligands was obtained. Then wash with Ethanol recrystallized with Ethanol and Ether then dried in air. The purity of compound was checked by TLC using Silica Gel method(Reported Method)[19].

SYNTHESIS OF METAL COMPLEXES: The metal complexes were prepared by mixing of $Cr(NO_3)_3.9H2O$ with (30 ml) ethanolic solution of Ligand in (metal: ligand) 1:2 ratio. The resulting mixture refluxed on water bath for 5-6hr.A colored product obtain washed with ethanol, filtered, and recrystalised with ethanol.

III. RESULTS AND DISCUSSION

The ligand and its transition metal complexes of 2,4-dichloro-6- (5-mercapto-1,3,4-thiadiazol-2-yl)imino methyl phenol are stable at room temperature in solid state.The ligand is soluble in organic solvent DMSO,DMF and metal complexes is easily soluble in

DMSO.The syn- thesized complexes having 1:2 metal to ligand stoichiometric ratio.

IR SPETRA: The IR spectra of 2, 4-dichloro-6-(5-mercapto-1, 3,4-thiadiazol-2-yl)imino methyl phenol (HL) Schiff base ligand and its complexes are listed.The Infrared Spectra of the complexes are compared with the free ligand in order to determine the coordination sites that may be involved in a chelation. There are some important peaks in the spectra of the ligand, which is different in metal complexes helps to prove that formation of metal complexes (20)

^{1}H NMR AND ^{13}C NMR SPECTRA: The 1H-NMR spectra of ligand were recorded in Dimethyl Sulphoxide solution using TMS as a standard(21).

MASS SPECTRA: Mass Spectra of ligands shows peak at m/z 305 which is M+H peak at 100% intensity this peak support to the structure formation of ligand

MAGNETIC SUSCEPTIBILITY AND MOLAR CONDUCTANCE: The magnetic susceptibility seen at room temperature.Synthesized metal complexes of Chromium (III) is paramagnetic in nature.Molar conductance of metal complexes was observed at room temperature at 1×10^{-3}M DMSO Solution. The studies show negligible molar conductance value in range 8-12 $ohm^{-1}cm^{2}mol^{-1}$results shows in table 4. it is observed that all metal complexes are non-electrolytic in nature (22).

ELECTRONIC ABSORPTION SPECTRA: The electronic spectral data of the ligands and metal complexes in DMSO sol. are given in Table 4. The geometry and nature of the ligand field around the metal ion has been conclude from the electronic spectral data of metal complxes and ligand. Electronic spectra of Cr (III) shows two peak shows ligand to metal donation with diamagnetic suggest octahedral geometry (23).

ANTIMICROBIAL ACTIVITY: Antimicrobial activity In vitro of the ligand and their corresponding metal Chromium complexes on two gram positive *bacteria S. aureus and B.Subtlis* two fungi *A. niger* and *F. Oxysporum* was carried out. All of the tested compounds showed good to moderate biological activity against test microorganism. The bactericidal and fungicidal investigation data of the ligand and Metal complexes are summarized in Table.The investigation shows that Cr (III) shows more The bactericidal and fungicidal activity ascompared to ligand hence activity of metal complexes increases due to chelation increase in delocalization of π electron on chelating ring and enhance the penetration of complexes in lipid membrane and blocks the binding site enzymes of microorganism there are other factors i.e,solubility,lipophilicity/hydrophilicity,Conductivity and M-L bond length that increases the activity of complexes(24).

IV. CONCLUSION

In the present work the synthesized Schiff base ligand binds metal ions in bidentate manner,with N and O donor site of azomethine-N and deprotonated phenolic-O.the antimicrobial activity data showed that Most of the metal complexes is more biologically active compared to those parent ligand against all pathogenic Bacteria and Fungi.

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