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Synthesis, Physicochemical and Biological Studies of Chromium (III) Metal Complex

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ABSTRACT

The present work is presented Assessment of the intrinsic structural study inflicted by metal coordination in the Chromium (III) complexes having fundamental importance for proper characterization and understanding of the structure–activity relationships. Efforts are being made here to investigate the coordination properties of metal ligand binding. The metal complex of trivalent chromium is characterized by elemental analyses, FT-IR, UV-Vis, magnetic and molar conductivity measurements. Metal Complexes is non electrolytic and Octahedral in geometry. Metal complexes of Cr (III) exhibit the potent antimicrobial activity.

Keywords: Schiff base, Antimicrobial activity, Coordination, Thiadiazole.

I. INTRODUCTION

The history of coordination complexes metal coordinates with ligand and forms metal complexes[1].In inorganic chemistry most active area is coordination chemistry i.e. chemistry of metal complexes. Now a day's two scientist is important in these chemistry i.e. Alfred Werner and Sorphus Mads Jorgenson.In 1913 Werner got noble prize in chemistry for their valuable contribution in coordination chemistry that leads to increase our understanding about nature of the metal-ligand bond, the structure and stereochemistry of metal complexes, their stability and other properties has been significantly enriched by the advent of sophisticated physicochemical techniques of high precision and capacity[2].Present research is advanced as compare to era of Werner and Jorgenson because development in the field of Coordination Chemistry is beneficial for human beings. In different fields we use coordination chemistry[3].Kossel, Sidgewick, Lewis, Langmuir, Fajan and others of electronic valency theory explain bond between metal ion and ligand called as secondary valencies[4] Primary valencies is oxidation state in complexes. Valence Bond theory is introduced by Pauling in 1931 which focus on electronic structure of central metal ion in ground state, magnetic behavior of complexes, geometry i.e. shape and type of bonding in complexes[5].

In Bio-inorganic chemistry like living system the coordination compounds study is rapidly developed [6].hence due to these reason coordination compounds such as metal-based drugs is very important and innovative area in recent research.In medicine coordination complexes is used as a diagnostic and therapeutic Purpose [7].In Drugs [8]coordination complexes is used also in biomolecules[9] as a ligands is very useful for research which is also depend on metal ligand bond. Different biological process metal plays important role via Chelation [10].but their capacity is affected when form bond to metal ion[11]. Present study the synthesis and characterization of novel Cr(III) Metal Complexes from heterocyclic ligand 2,4-dichloro-6-(((5-mercapto-1,3,4-thiadiazol-2-yl)imino)methyl)phenol.Moreover,the preliminary antimicrobial activities of the ligand and Metal complex are carried out and the results are reported herein.

II. METHODS AND MATERIAL

All the chemical of analytical grade. Salts are metal nitrates i.e. $Cr(NO_3)_3{\cdot}9H_2O$ (Sigma-Aldrich) were purchased from Sigma-Aldrich and used without further purification. 3,5-dichloro-2-hydroxybenzaldehyde and 5-amino-1,3,4-thiadiazole-2-thiol from Sigma-Aldrich and Alfa Aesar used without 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. 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

Antimicrobial Activity

Schiff Base and their 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[12].The experimental value compare with standard drug value Miconazole for the Antifungal activity and Ciprofloxacin for the antibacterial activity.

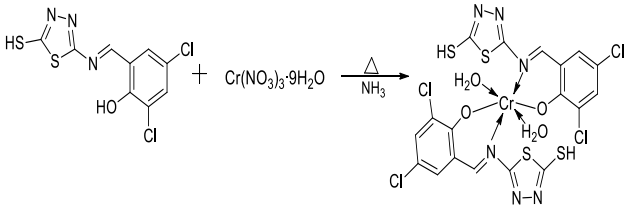
Synthesis of Ligand

The ligand is prepared by reported method[13].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 recrystalized with Ethanol and Ether then dried in air. The purity of compound was checked by TLC using Silica Gel method.

Synthesis of Metal Complex

The metal complexes were prepared by mixing of $Cr(NO_3)_3{\cdot}9H_2O$ with (30 ml) ethanolic solution of Ligand 2,4-dichloro-6-(((5-mercapto-1,3,4-thiadiazol-2-yl)imino)methyl)phenol 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 (Scheme 1).

Scheme 1. Synthesis of metal complexes



2,4-dichloro-6-(((5-mercapto-1,3,4-thiadiazol-2-yl)imino)methyl)phenol Chromium(III) nitrate nonahydrate bis(2,4-dichloro-6-(((5-mercapto-1,3,4-thiadiazol-2-yl)imino)methyl)phenoxy)chromium dihydrate

III. RESULTS AND DISCUSSION

The transition metal complexes (Table.1) Bis(2,4-dichloro-6-(((5-mercapto-1,3,4-thiadiazol-2-yl)imino)methyl)phenoxy)Chromium dihydrate are stable at room temperature in solid state. The ligand is soluble in organic solvent DMSO, DMF and a metal complex is easily soluble in DMSO.The synthesized complexes having 1:2 metal to ligand stoichiometric ratio. The structure data of metal complexes as shown in Table 1.Analytical Data and physical properties shows formation of metal complex (Table.2&3).

Table 1: Proposed Structures of metal complex Cr (III)

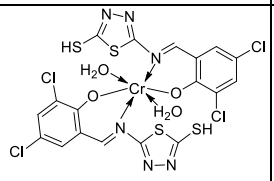
Entry	Products	Time (h)	Yield (%)	M.P (°C)
MC		5-7	70	>300

Table 2: Analytical Data and physical properties of complex.

Comp.	Empirical Formula	Mol. Wt.	Color	M.P (°C)	Yield
Cr MC	$C_{18}H_{12}Cl_4CrN_6O_4S_4$	698	Light Green	>300° C	70

Table 3 : Analytical Data

Elemental Analysis/ Found (Calc.)			
C	**H**	**N**	**S**
30.96 (30.10)	1.73 (1.60)	12.03 (12.20)	18.36 (18.10)

IR Spectra

The IR Spectra of Bis(2,4-dichloro-6-(((5-mercapto-1,3,4-thiadiazol-2-yl)imino)methyl)phenoxy)Chromium dihydrate metal complex (Table 4.) shows 3416 Cm-1 IR stretching frequency for –OH is for coordinated water molecule which is not present in ligand. The water molecule contains oxygen donates electron to metal ion in metal complex. -C=N- Azomethine group frequency for MC is 1624 Cm-1 and Ligand is 1645 Cm-1 these difference is due donation of electron of Azomethine nitrogen of ligands to free metal ion hence confirms formation of metal complex. 1435 Cm-1 is frequency for -C=N-N=C- group Thiadiazole ring of complex.1282 Cm-1 is for C-O Phenolic group,1027 Cm-1 is frequency for N-N Thiadiazole ring,758 Cm-1 for C-S-C group in thiadiazole ring. The newly form metal ligands bond shows IR frequency for shows at 552 Cm-1 for M-O bond and 440 Cm-1 for M-N bond[14].

Table 4 : Infrared Spectra of the Schiff base and Complexes

Comp.	νOH/H_2O	νC-O	νC=N	νC-S-C	ν-C=N-N=C	νN-N
Ligand	3319	1265	1645	756	1467	1028
Cr(III) Complexes	3416	1282	1624	758	1435	1027

Electronic absorption Spectra

The electronic absorption spectral data of the ligands and its metal complexes in DMSO sol. are given in Table 4.Eletronic spectral data shows information about geometry and nature of the ligand field around the metal ion. The band appearing at 221-315 is due to transition of benzene ring of the ligand. The other band due to free ligands 302-371 nm due to transition for phenolic -OH and azomethine moieties(-C=N-).These band shifts longer wavelength due to formation of ligand to metal complexes.The Charge transfer transition from ligands to metal leads to sharp band in between 442 nm and 620 nm it confirms octahedral geometry Cr(III) complex [15].

Magnetic Susceptibility

The magnetic moment value for Chromium (III) (Table 3) complex is 3.85 BM hence it confirms octahedral geometry Cr(III) complex[16].

Molar Conductance

Molar conductance of metal complexes was observed at room temperature at 1×10-3M DMSO Solution (Table 5).Molar conductance value of Chromium (III) complexes shows in between value in range 8-12 ohm-1cm2mol-1.Metal Complexes are non-electrolytic in nature[17]

Table 5. Electronic spectral Magnetic and Molar conductance Data

Compounds	Wavelength in nm	Magnetic moment μeff (BM)	Molar conductance (ohm-1 cm2 mol-1)
Cr(III) Complexes	310,442,620	3.85	9.2

Antimicrobial Activity

The antimicrobial activity in vitro on selected two gram positive bacteria S. aureus and B.Subtlis two fungi A. niger and F.Oxysporum was carried out shown in table.6.The Metal Complexes of Cr(III) is more biologically active as compare to parent ligand because of chelation increase in delocalization of π electron on chelating ring that 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[18]

Table 6. Antimicrobial activity of ligand and its Metal Complexes

Compounds	**Antibacterial Activity**		**Antifungal Activity**	
	S.aureus	***B.subtilis***	***A.niger***	***F.oxysporum***
	Diameter of inhibition Zone in mm	Diameter of inhibition Zone in mm	Diameter of inhibition Zone in mm	Diameter of inhibition Zone in mm
	500 ppm	500 ppm	500 ppm	500 ppm
Ligand	22	21	20	18
Cr Complex	25	23	23	20
Ciprofloxacin (Standard)	34	33	----	----
Miconazole (Standard)	----	----	31	27

IV.CONCLUSION

In the present work Schiff Base metal complex is prepared and Characterized with different analytical technique. The Metal complex is more bioactive as compare to Parent Ligand. This type of research is important for futuristic approach in medicinal chemistry.

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