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SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF Fe(III) AND Cr(III) COMPLEXES OF SUBSTITUTED 4,4'- DIMETHOXYBENZOINSEMICARBAZONES

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Abstract

Recently, the synthesis of 4,4'-Dimethoxybenzoinsemicarbazones with semicarbazide hydrochloride in presence of aqueous sodium hydroxide in DMF-water (80%) medium respectively. The synthesis of 4,4'- Dimethoxybenzoinsemicarbazones were carried out by the known literature method. They were characterized by elemental and spectral analysis. The Physico-Chemical data suggested octahedral geometry for Cr(III) & Fe(III) complexes. The synthesized complexes were screened for antimicrobial activity at a concentration of 1000µgm/ml. Which was serially diluted to determine their MIC values.

Keywords:- Metal complexes , 4,4'-Dimethoxybenzoinsemicarbazones,Antimicrobial activity, sodium hydroxide,DMF-Water(80%) medium , spectral analysis

Introduction

The spectroscopic and biochemical studies of chromium and Mn(II) with p-vanillin containing thiosemicarbazone and semicarbazone ligands reported by chandra[1] .Benzoinsemicarbazone are well known for their biological activity Coordination compounds containing ONS as donor atoms are reported to possess antimicrobial activity[2] . Kumar[3] carried out synthesis and characterization of Mn(II), Fe(III) ,Co(II), Ni(II) and Cu(II) complexes of salicyaldehydesemicarbazone . Khan[4] reported synthesis and characterization of Co(II), Ni(II) Cu(II) and Cd(II) complexes with 2-furfuralsemicarbazone (FSC) and 5-methyl-2-furfuralsemicarbazone (MFSC). Physico-chemical and spectral studies of Ni(II) complexes of 2-substituted benzaldehydesemicarbazone, and thiosemicarbazones were carried out by Kumar[5]. O- vanillin semicarbazone have been synthesized and characterized by different physicochemical techniques by Hingorani [6] . Choudhary [7] carried out synthesis and characterization of new series of mixed ligand complex of Co(II) and Cu(II) with thiosemicarbazone/ Semicarbazone and screened their antibacterial and antifungal activities in detailed. Investigation on variety of semicarbazones and Schiff bases and their transition metal complexes was carried out by several workers [8-9] Mohapatra [10] reported the complexes of divalent Mn(II), Co(II), Cu(II) with benzil semicarbazone .In the present work novel transition metal complexes of substituted benzoin semicarbazone are reported. The magnetic moment values of Cr(III),Mn(II) and Fe(III) complexes are in good agreement with the presence of three, four and five unpaired electrons respectively[11].

Experimental Section

The 4,4'-DMBSC was prepared by refluxing substituted benzoine with semicarbazide hydrochloride in presence of alkaline medium for 3-4 hours this reaction mixture was kept

overnight. This solid products formed were isolated and washed several times with water alcohol mixture the purity was checked by TLC paper. Their structural details were confirmed on the basis of elemental and spectral analysis. Synthesis of complexes the equimolar mixture of each of the ligand(0.01M) and metal salts(0.01M) were refluxed on a water bath for 6,8 hours in presence of sodium acetate in ethanol . The reaction mixtures was kept overnight. The product formed were isolated washed several times with cold water ethanol mixture. The characterization of synthesized complex was made with elemental analysis, IR and UV-Vis spectra. The melting point of all synthesized compounds was recorded using hot paraffin bath. The carbon and hydrogen analysis were carried out of carlo-Ebra 1106 elemental analyzer. Nitrogen estimation was carried out colman-N-analyzer-29. IR spectra were recorded on perkin Elmer spectra were recorded on Brucker Ac 300F spectrometer with TMS as internal standard using $CDCl_3$ and DMSO-d_6 as solvent. The purity of compounds was checked on silica Gel-G pellets by TLC with layer thickness of 0.3mm. All chemicals used were of AR grade.

Result and Discussion

TABLE-1

THE METAL COMPLEXES COMPOUND, MOLECULAR WEIGHT, COLOUR AND ELEMENTAL ANALYSIS OF VARIOUS METAL IONS

Complexes	Colour	Molecular wt	Elemental analysis Found/(calculated)%			
			C	H	N	M
4-DMABSC-Cr(III)	Brown	709.99	56.55 (57.46)	4.00 (5.91)	15.77 (15.65)	6.40 (7.32)
4-DMABSC-Fe(III)	Red	619.99	44.65 (45.58)	3.60 (4.51)	13.54 (13.45)	7.46 (8.38)
4,4'-DMABSC-Cr(III)	Brown	779.99	51.37 (52.30)	4.71 (5.64)	10.76 (10.76)	5.74 (6.66)
4,4'-DMABSC-Fe(III)	Red	747.84	53.63 (54.55)	4.42 (5.34)	11.23 (11.23)	6.53 (7.46)

TABLE-2

IR SPECTRAL DATA OF LIGANDS AND ITS METAL COMPLEXES

Ligands and its Complexes	υ(O-H)	υ(C=N)	υ(C-O)	υ(M-O)	υ(M-N)
4-DMABSC	3463	1569	1181	-	-
4-DMABSC-Cr(III)	3411	1549	1169	462	581
4-DMABSC-Fe(III)	3419	1545	1171	469	587
DMABSC	3472	1658	1119	-	-
4,4'-DMABSC-Cr(III)	3351	1498	1107	460	320
4,4'-DMABSC-Fe(III)	3397	1526	1109	462	328

The IR spectra of lignad shows a strong band at 1569cm^{-1} due to (C=N) group broad band around 3463cm^{-1} in the spectra of complexes is assignable of water. DABSC - Fe (III) shows band at 3463(O-H).Which decreases 3411cm^{-1} inducting that attached to oxygen .However 1569 (C=N) significantly decrease to 1545 cm-1 showing linkage through azido nitrogen.The υ(M-O)& υ(M-N) vibration are verified to existing by the appearance of new weak bands in the spectra of complexes at 581&320 respectively

TABLE-3

ELECTRONIC SPECTRAL DATA,MAGNETIC MOMENT AND LIGAND FIELD PARAMETER OF THE METAL COMPLEXES

Complexes	μeff (B.M.)	λ max (cm-1)	Dq	B'	B	CFSE	% Cova	$(\Lambda_M)\Omega^{-1}$ Cm^2 Mol^{-1}
4-DMABSC-Cr(III)	4.07	13476,19590,22620	1476	695	0.757	202	24.03	12.8
4-DMABSC-Fe(III)	4.16	13769,19794,22150	1538	697	0.687	352	31.03	11.4
4,4'-DMABSC-Cr(III)	4.08	13479,19230,22621	1476	695	0.757	202	24.03	12.5
4,4'-DMABSC-Fe(III)	5.83	13422,17833,21957	1465	651	0.641	335	35.07	12.8

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The electronic spectrum of Cr(III) complexes exhibits three bands at 13476,19590 and 22620 cm^{-1} which may be assigned to $^4A_{2g} \dashrightarrow {}^4T_{2g}(F)$,$^4A_{2g} \dashrightarrow {}^4T_1g$ (F) and $^4A_{2g} \dashrightarrow {}^4T_1g(P)$,transition, respectively for an octahedral stereochemistry[11] .The magnetic moment value of 4.07 to 4.08 B.M for Cr(III) complex is consistent with octahedral geometry around metal centre. Fe(III) Complexes three bands are observed in case of Fe(III) complexes at 14077, 8565, 23218 cm^{-1} which may be assigned to $^6A_{1g} \dashrightarrow {}^4T_{1g}(F)$,$^6A_{1g} \dashrightarrow {}^4T_2g(F)$ and $^6A_{1g} \dashrightarrow {}^4Eg$, belongs to transition respectively, indicating octahedral geometry of Fe(III)complexes [12-13]. The value of 5.81and5.83 B.M. would suggest high spin six coordination for Fe(III) complexes. The values of various ligand field parameters 10Dq,B B',λmax, CFSE and conductance respectively favoring in octahedral geometry for this complex.

ANTIMICROBIAL ACTIVITY OF COMPLEXES

The compounds were assayed for their antimicrobial activities[14] against four test organisms E.coli, S. aureus, Ps.aeruginosa, B. subtilis at a concentration of 1000µgm/ml by agar well technique[15]. Further their MIC value against these organisms were determined by serial dilution method using DMF as a solvent. The results obtained are given in table-5

TABLE-5

MIC VALUES IN µgm/ml OF COMPOUNDS

Complexes	E.coli	S.aureus	P. aeruginosa	B.Subtilis
4-DMABSC-Cr(III)	250	125	63	125
4-DMABSC-Fe(III)	125	125	250	250
4,4'-DMABSC-Cr(III)	120	250	125	63
4,4'-DMABSC-Fe(III)	63	63	125	125

On the basis of MIC values, 4-DMABS-Cr(III) is found to be most effective antimicrobial agent followed by 4-DMABSC-Cr(III) and 4-DMABSC-Fe(III).The enhance antimicrobial activity in case of the compounds. 44'DMABS-Cr(III) and Fe(III) showed the lowest MIC values(i.e 63µgm/ml) against maximum number of microorganisms.

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