SYNTHESIS, SPECTRAL AND ANTIMICROBIAL STUDY OF CR(III) AND FE(III)COMPLEXES OF SUBSTITUTED 4-DIMETHYLAMINOBENZOINOXIMES

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ABSTRACT

Metal benzoinoxime complexes were synthesized from substituted benzoinoximes they were characterized by elemental and spectral analysis. The physico-chemical data suggest octahedral geometry for Cr (III) and 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, Antimicrobial activity 4-Dimethylaminobenzoinoxime,*

Introduction

Synthesis Fe (III) complexes of O-Vanillin oxime and their characterisation by different physico-chemical techniques was carried out by kurup[1].synthesis of mononuclear and binuclear Cr(III) complexes of α-benzoinoxime and their characterization was studied in detailed[2].synthesis of Fe(III) benzoin complexes and there characterization were carried out by El- agaily[3].

Luo[4] synthesized oximes from different carbonyl compounds in a novel ionic liquid or water biphasic system synthesis of Co (II) , Ni (II) Zn (II), Fe (III), Cu (II) complexes with oxime amido and thioamido groups and their characterization were briefly studied by El-Asmy[5]. complexes of Cr (III) and Mn (II) with oximes such as 2-hydroxyacetatophenone oxime, 2-hyroxynaphthaldehyde oxime and salicylaldehyde oxime were synthesized and characterized by Chandra[6].Benzoinoxime are well known for their biological activity, co-ordination compounds containing O,N,S. as donor atoms are reported to possess antimicrobial activity[7]. synthesis characterization and thermall degradation studies of coordination polymers of ethanone oxime were carried out by wanjari[8].Day[9] ,synthesized a large number of Cr (III) complexes and reported the magnetic moment values in the range 3.78-3.99 .Rahangadale[10].synthesized the Schiff base and its complexes were screened for their antimicrobial activities various against bacteria and fungi.

Experimental

The benzoinoxime were prepared by refluxing substituted benzoin with hydroxylamine hydrochloride in alkaline medium for 3-4 hours, the reaction mixtures were kept overnight the solid products formed were isolated and wash several times with water alcohol mixture. The purity was checked by TLC paper. Their structural detail were confirmed on the basis of elemental and spectral analysis In order to synthesize the complexes, the equimolar mixture of each of the ligand (o.o1M) and metal salts was refluxed on a water bath for 6-8 hours in presence of sodium acetate in ethanol. The reaction mixture was kept overnight. The product formed were isolated ,washed several times with cold water ethanol mixture the characterization of synthesized complexes was made by elemental analysis, IR and UV-VIS spectra.

Results and discussion

IR spectral data of ligands and their complexes are given in table -1

Ligand and its complexes	(O-H)	(C=N)	(C-O)	(M-O)	(M-N)
4-DMABO	3423	1660	1385	-	-
$[Cr(L)_2(H_20)_2]\ H_2O$	3408	1590	1375	460	589
$[Mn(II)(L)_2(H_2O)_2]$	3412	1605	1375	465	585
$[Fe(L)_2(H_2O)_2]\ H_2O$	3410	1605	1375	463	591
$[CO(II)(L)_2(H_20)_2]\ H_2O$	3318	1645	1380	482	587
$[Cu(II)(L)_2(H_2O)_2]\ H_2O$	3420	1640	1382	485	59

In these complexes 4-DMABO-Cr(III), υ(O-H) is observed at 3408 cm^{-1} υ(C=N) at 1590 cm^{-1}. Are indicative of linking of oxygen without loss of Hand linking of N to the metal ion respectively.These lower values of bands in hydroxyl and oximino stretching as compared to ligand clearly indicates that the coordinate bonding through hydroxyl oxygen and oximino nitrogen atom to the metal ion. In the complexes of 4-DMABO-Fe(III), υ(O-H) at 3410 cm^{-1} which shoes linking of metal oxygen atom without loss of proton similarly,(C=N) is observed at 1605cm^{-1} which shows decrease in (C=N) stretching frequency during complexation and hence give clue about linkage.The electronic spectrum of 4-DMABO-Cr(III) complexes exhibits their transition in the range 13870,19194,22892 cm^{-1}.

Magnetic moment and electronic spectral data (cm^{-1}) of the metal complexes as given table-2

Complexes	μeff (BM)	λmax(cm^{-1})	Dq(cm^{-1})	B^1 (cm^{-1})	B	%Covalency
$[Cr(L)_2(H_20)_2]$ H_2O	3.97	13870,19194,22892	1516	687	0.749	25
$[Mn(L)_2(H_2O)_2]$	4.51	13652,19126,23251	1527	796	0.784	21
$[Fe(L)_2(H_2O)_2]H_2O$	5.45	13859,20002,24386	1467	751	0.88	18
$[Co(L_2)(H_2O)]H_2O$	5.15	13476,19002,22620	1476	695	0.716	28
$[Cu(L)_2(H_2O)_2]H_2O$	1.93	13670,19002,22620	1467	695	0.716	28

The electronic spectrum of 4-DMABO-Cr(III) complexes exhibits three transition in the range,13870,19194,22892 cm^{-1}.These spectral bands may be assigned to the following transition $^4A_2g(F) \rightarrow {}^4T_2g(F)$, $^4A_2g(F) \rightarrow {}^4A_2g(F)$, $^4T_1g(F) \rightarrow {}^4T_1g(P)$ characteristic to an octahedral geometry. The magnetic moment of 3.97BM for Cr(III) complexes is consistent with octahedral geometry around central metal ion 4-DMABO-Fe(III) complexes exhibit absorption bands at 13859,20002,24386 cm^{-1} ·Which may be consign to $^6A_1g \rightarrow {}^4T_1g(F),{}^6A_1g \rightarrow {}^4T_2g(F),{}^6A_1g \rightarrow {}^4Eg$ trasition respectively suggesting on octahedral geometry around a Fe(III) ion in the complexes under study, further more the magnetic moment measurement recorded at room temperature lies at 5.45 BM. This values is indicates of an octahedral geometry of these complexes the calculated values is of ligand field splitting energy (10Dq) ,Racah interelectronic repulsion parameter (β) and % Covalency as shown in above table.

On the basic of elemental analysis the complexes were assigned the composition as shown in table-3

Complexes	Colour	M. Wt.	Decomposition Temp°c
$[Cr (L)_2(H_2O)_2] H_2O$	Grey	643.99	300
$[Mn (L)_2(H_2O)_2]$	Brown	628.93	282
$[Fe (L)_2(H_2O)_2] H_2O$	Red	647.84	310
$[Co (L)_2(H_2O)_2] H_2O$	Red	650.93	278
$[Cu (L)_2(H_2O)_2] H_2O$	Dark Brown	637.54	273

Elemental analysis :-Table-4

Complexes	Elemental analysis found/(calculated)%			
	C	H	N	M
$[Cr(L)_2(H_2O)_2]H_2O$	58.74 (59.62)	5.27 (6.21)	8.68 (8.68)	7.18 (8.07)
$[Mn(L)_2(H_2O)_2]$	60.11 (61.05)	5.13 (6.04)	8.89 (8.90)	7.81 (8.73)
$[Fe(L)_2(H_2O)_2]H_2O$	58.35 (59.27)	5.22 (6.17)	8.64 (8.64)	7.73 (8.07)
$[Co(L)_2(H_2O)_2]H_2O$	58.03 (58.99)	5.30 (6.14)	7.71 (8.60)	8.98 (9.05)
$[Cu(L)_2(H_2O)_2]H_2O$	59.07 (60.23)	4.88 (5.96)	8.78 (8.78)	8.90 (9.96)

Thermogravimetric Analysis

An analysis of TG curves of 4-DMABO and its metal complexes as shows that the Cr(III), Co(II) and Fe(III) complexes decompose in three stages,the Cu(II) and Mn(II) complexes decompose in two stages.The complexes of Cr(III) , Co(II) and Fe(III) are stable up to 140°C .The presence of water molecule (lattice or co-ordinated) in Cr(III) ,Co(II) and Fe(III) complexes suggested from IR spectra is confirmed by the weight loss observed in first decomposition step of these complexes the Cr(III) ,Co(II) and Fe(III) complexes lose their weights up to 150°C corresponding to one lattice water molecule [% wt. loss obs./calcd : Cr(III):2.79/2.73, Co(II) : 5.53/5.46,Fe(III) : 2.78 / 2.73] and further up to 220° C corresponding to two coordinated water molecule [% wt. loss obs./ calcd : Cr (III) : 8.39 / 8.33 ,Co(II):8.30 / 8.20 , Fe(III) : 8.33/8.26].In case of Cu(II) and Mn(II) complexes[24] weight loss at 150°C corresponds to two co-ordinated water molecule [% wt loss obs. / calcd. : Cu (II): 5.65 / 5.60, Mn (II): 5.73 / 5.66]. These complexes are stable up to 250°C. The organic moiety decomposes in the temperature range of 550° C – 650° C. At the end of the last step/ final stage, stable metal oxides are formed corresponding to C r_2 O_3, MnO_2 , Fe_2 O_3 , CuO and CoO respectively[33] The half decomposition temperature and the basic parameter calculated for the compounds are comparable thermal stability on the basis of the half decomposition temperature is found to be Mn(II) < Cu(II) <Fe (III) < Cr(III) < 4-DMABO < Co(II).

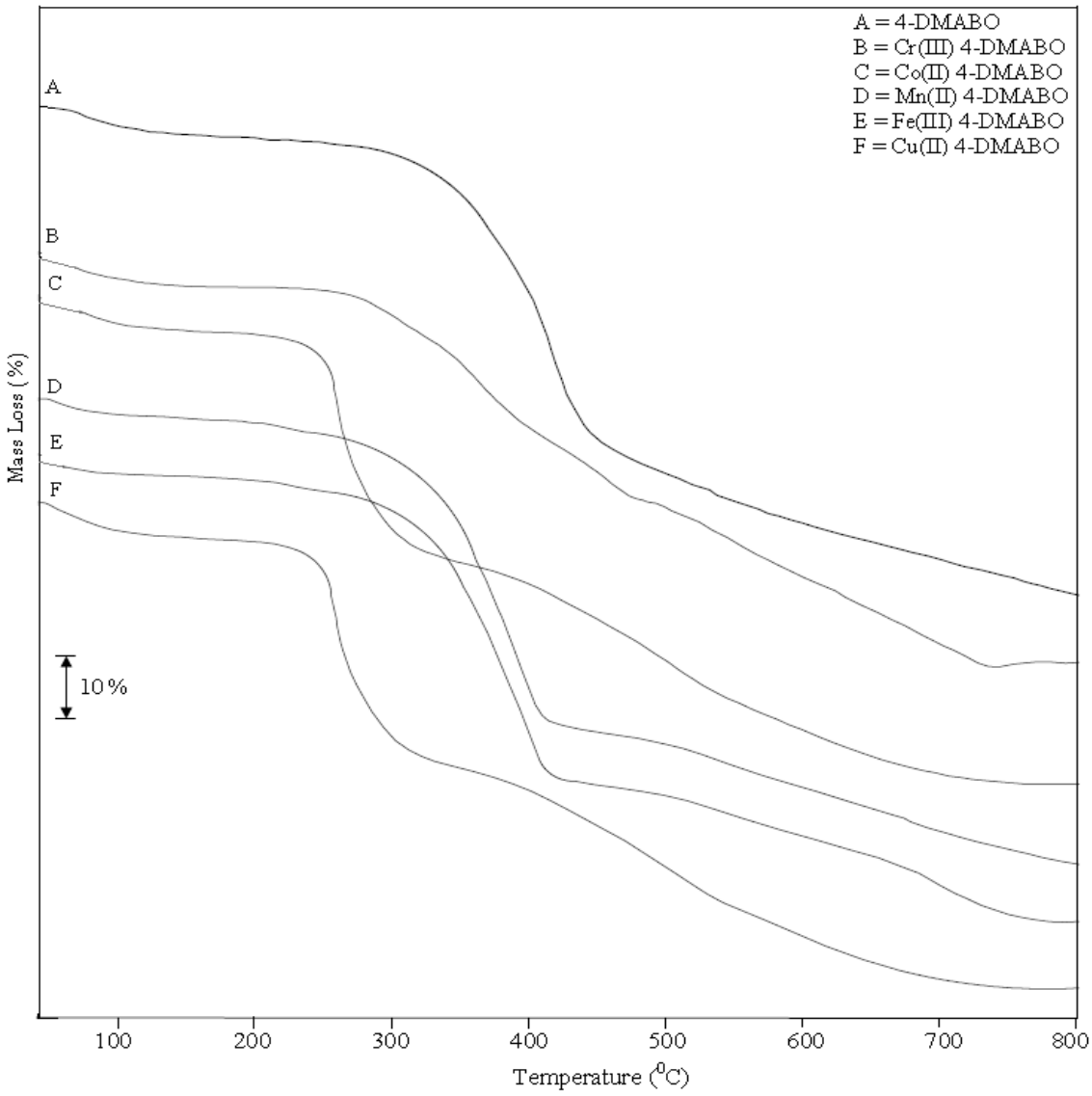

Fig:1.1 Thermograms of 4-DMABO and its complexes

Antimicrobial activity

The compounds were assayed for their antimicrobial activities[12] Against for test organisms. E. Coli S.aureus, P.aeruginosa and B.subtilis, at a concentration of 1000 µgm/ml by agar well technique[13]. Further their MIC value against these. Organisms were determined by serial dilution method using DMF as a solvent, the results obtained are given in the following table.

MIC Values in µgm/ml of compounds

Complex	E. coil	S. aureus	P. aerugenosa	B. subtilis
$[Cr(L)_2(H_2O)_2]H_2O$	63	63	125	63
$[Mn(L)_2(H_2O)_2]$	125	125	125	125
$[Fe(L)_2(H_2O)_2]H_2O$	125	125	63	63
$[Co(L)_2(H_2O)_2]H_2O$	63	125	125	250
$[Cu(L)_2(H_2O)_2]H_2O$	125	125	250	250

On the basis of MIC Values, the complexes 4-DMABO-Cr(III),Fe(III),is found to be most effective antimicrobial agent followed by,Co(II),Mn(II) and Cu(II).The enhance antimicrobial activity in case of the compounds 4-DMABO-Cr(III) may be attributed to the presence of amino group.

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