SYNTHESIS,CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF SOME METAL COMPLEXES OF SUBSTITUTED 2-HYDROXYBENZOINOXIME

P.M. Dahikar
Department of Chemistry, Shri. R.R. Lahoti Science College , Morshi, Dist. Amravati
pushpadahikar@gmail.com

ABSTRACT

Recently, the synthesis of 2-hydroxybenzoinoxime, 2-hydroxy-benzoinhydrazone, 2-hydroxybenzoinphenylhydrazone and 2-hydroxybenzoin-semicarbazone with hydroxylamine hydrochloride, hydrazine hydrate, phenyl hydrazine and semicarbazide hydrochloride in presence of aqueous sodium hydroxide in DMF-water (80%) medium respectively. 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 sodium hydroxide, DMF-water (80%) 2-Hydroxybenzoinoxime.*

Introduction

Synthesis Fe(III) complexes of O-Vanillin oxime and their characterisation by different physico-chemical techniques was carried out by kurup1.synthesis of mononuclear and binuclear Cr (III) complexes of α-benzoinoxime and their characterization was studied in detailed2.synthesis of Fe(III) benzoin complexes and there characterization were carried out by El- agaily3. Luo4 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-Asmy5. 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 Chandra6.Benzoinoxime are well known for their biological activity, co-ordination compounds containing O,N,S. as donor atoms are reported to possess antimicrobial activity7. synthesis characterization and thermall degradation studies of coordination polymers of ethanone oxime were carried out by wanjari8.Day9 ,synthesized a large number of Cr (III) complexes and reported the magnetic moment values in the range 3.78-3.99 .Rahangadale10.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)
2-HBO	3412	1666	1463	-	-
$[Cr(L)_2(H_2O)_2]$	3275	1598	1452	463	587
$[Fe(L)_2(H_2O)_2]$	3329	1600	1453	464	590
$[Mn(L)_2(H_2O)_2]$	3343	1602	1458	465	583
$[Co(L)_2(H_2O)_2]$	3369	1607	1459	479	589
$[Cu(L)_2(H_2O)_2]$	3386	1610	1462	484	593

In these complexes 2-HBO-Cr(III), υ(O-H) is observed at 3275 cm-1 υ(C=N) at 1598 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 2-HBO-Fe(III), υ(O-H) at 3329 cm-1 which shoes linking of metal oxygen atom without loss of proton similarly,(C=N) is observed at 1600 cm-1 which shows decrease in (C=N) stretching frequency during complexation and hence give clue about linkage.

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
$[Cr(L)_2(H_2O)_2]$	3.96	12667,16815,22354	1396	732	0.77	20
$[Fe(L)_2(H_2O)_2]$	5.45	13859,20132,24386	1527	796	0.74	21
$[Mn(L)_2(H_2O)_2]$	4.49	14075,18867,22892	1535	673	0.70	29
$[Co(L)_2(H_2O)_2]$	5.19	13333,19417,22471	1461	694	0,71	28
$[Cu(L)_2(H_2O)_2]$	1.88	13605,19230,22727	1461	694	0.71	28

The electronic spectrum of 2-HBO-Cr(III) complexes exhibits three transition in the range, 12667,16815,22354 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. 97 BM for Cr(III) complexes is consistent with octahedral geometry around central metal ion 4-DMABO-Fe(III) complexes exhibit absorption bands at 13859,20132 ,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
$[Cr (L)_2(H_2O)_2]$	Red brown	571	295
$[Fe (L)_2(H_2O)_2]$	Grey	575	329
$[Mn(L)_2(H_2O)_2]$	Browinsh red	592	323
$[Co(L)_2(H_2O)_2]$	Yellow	596	286
$[Cu(L)_2(H_2O)_2]$	Dark grey	601	276

Elemental analysis :-Table-4

Complexes	Elemental analysis found/(calculated)%			
	C	H	N	M
$[Cr(L)_2(H_2O)_2]$	57.60 (58.74)	3.91 (4.89)	4.89 (4.89)	8.99 (9.08)
$[Fe(L)_2(H_2O)_2]$	57.44 (58.34)	3.87 (4.86)	4.86 (4.86)	8.60 (9.61)
$[Mn(L)_2(H_2O)_2]$	55.71 (56.66)	4.13 (5.05)	4.72 (4.72)	8.94 (9.26)
$[Co(L)_2(H_2O)_2]$	55.34 (56.28)	4.10 (5.02)	4.69 (4.69)	8.91 (9.93)
$[Cu(L)_2(H_2O)_2]$	54.93 (55.85)	4.00 (4,98)	4.65 (4.65)	9,93 (10.56)

Thermogravimetric Analysis

An analysis of TG curve of 2-HBO and its metal complexes are given in(Fig. 4.5). The decomposition ligand 2-HBO start at comparatively lower temperature of around 120°C where as in most of the complexes decomposition start at higher temperature. Which indicates that the complexes of Co(II) and Mn(II) complexes decompose in three stages,while ligand and its complexes of Cr(III) and Fe(III) in two steps. The Co(II),Mn(II) and Cu(II) complexes are stable upto 130°C .The elimination of one water molecule from Co(II), Mn(II) and two water molecule from Cu(II) complerxes upto 150°C have been observed [% wt. loss obs. /calcd : Co(II) : 3.02 /3.00 ,Mn(II) :5.68 /5.66 ,Cu(II) : 5.98/5.93]. In the some complexes i.e. Co(II) and Mn(II) there is further weight loss upto 230°C indicating the presence of two coordinated water molecule in the each complex[24] [% wt. loss obs. /calcd: Co(II) : 9.06/ 9.00 ,Mn(II) :8.54 /8.53]. There is no weight loss upto 550-650°C indicating the absence of the any other water molecules (lattice / co-ordinated) in all these complexes[33].Finally level beyond 650°C indicates the formation of final decomposition products corresponding to Cr_2O_3, CoO, MnO2, Fe_2O_3 and CuO respectively. The half decomposition temperature and basic parameter calculated for the compounds. The relative thermal stability of half decomposition temperature is found to be

Co(II) < Mn(II) < Cu(II) < Cr(III) < Fe(III) < 2-HBO

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
$[Cr(L)_2(H_2O)_2]$	63	125	63	63
$[Fe(L)_2(H_2O)_2]$	125	63	63	125
$[Mn(L)_2(H_2O)_2]$	63	63	125	125
$[C o(L)_2(H_2O)_2]$	125	125	63	63
$[Cu(L)_2(H_2O)_2]$	125	125	125	125

On the basis of MIC Values, the complexes 2-HBO-Cr(III), is found to be most effective antimicrobial agent followed by Fe(III) , Mn(II) and Co(II). The enhance antimicrobial activity in case of the compounds 2HBO-Cr (III) may be attributed to the presence of amino group.

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