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Synthesis And Characterization Of Mixed Ligand Complexes Of Transition Metal Chelates Of 1-Nitroso-2-Naphthol And 8-Hydroxyquinoline With Picolinic Acid

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ABSTRACT

Mixed ligand transition metal complexes having general formula ML, HL', where M = Cu(II) or Pd(II); L deprotonated 1-nitroso-2-naphthol or 8-hydroxyquinoline; HL' = picolinic acid have been synthesised and characterised on the basis of Infrared, electronic absorption spectral data, magnetic and conductance measurements. The above study reveals the octahedral geometry of the complexes. Microbiological studies revealed that all prepared mixed ligand complexes are relatively potential inhibitor of the growth of both the test bacteria and one fungi .

Key words: Mixed Ligand Complexes, Picolinic acid, 8-Hydroxyquinoline, 1-Nitroso-2-naphthol.

INTRODUCTION

Picolinic acid has been extensively investigated for their possible roles in analytical determination of metal ions[1-4]. They have replaceable hydrogen atom as well as two donor atoms suitably placed to form a five membered chelating ring with metal ion. The versatile chelating ability of picolinic acid with various metals is well established[5-10]. In the present communication, we report the synthesis and characterization of a number of mixed ligand complexes of Cu(II) or Pd(II) with above mentioned ligands.

EXPERIMENTAL

Picolinic acid(HPicA), 1-nitroso-2-naphthol(1N2N) and 8-hydroxyquinoline(8HQ) of AnalaR grade were used as such.

Preparation of the complexes

To the suspension of 0.01 mole transition metal salt of organic acid(1N2N or 8HQ) in absolute ethanol, 0.01 mole picolinic acid (HPicA) was added. The whole reaction mixture was refluxed with constant stirring on a hot plate of magnetic stirrer for 1-2 hours and cooled to give characteristic colour solid adduct. The adduct was filtered, washed with absolute ethanol and dried in an electric oven at 100°C.

RESULTS & DISCUSSION

Some physical properties and analytical data of the ligand(HPicA) and the new mixed Igand complexes obtained are listed in Table-1.

The mixed ligand complexes are generally coloured. They are appreciably soluble in most polar solvents such as methanol, DMF etc. but are insoluble in non-polar solvents such as benzene, toluene, ether etc. The complexes are found to be stable when stored under dry conditions. Melting/decomposition temperatures of the complexes have been found to be higher than those of the corresponding ligand, indicating thereby their greater stability.

Molar conductance

Molar conductance values of all these complexes were measured in methanol at 23°C at a concentration of 10^{-3}M. The values are given in Table-1. Low values of molar conductance (2.8 - 4.6 $ohm^{-1}cm^2mol^{-1}$) show that these complexes are non-electrolyte in nature.

Infrared spectra

Infrared spectra of the ligand(Picolinic acid) and their mixed ligand Cu(II) or Pd(II) complexes were recorded in KCl phase between 4000-400 cm^{-1} with the help of JASCO FTIR spectrophoto-meter model -5300. Selected IR absorption bands are shown in Table - 2.

IR spectra of picolinic acid & its complexes

The broad band at 3400 cm^{-1} in the spectrum of ligand(HPicA) indicates strong intramolecular hydrogen bonding in it. The spectra of all the mixed ligand complexes of picolinic acid with transition metal derivatives of some organic acids(1N2N & 8HQ) show a number of unexpected features. The shift of the -OH band of the second ligand and its reappearance in the region 3075-3060 cm^{-1} may be assigned to O-H...O/N...H-O absorption and this feature suggests hydrogen bonding to be a dominant factor in stabilizing these complexes

The appreciable shift of 1650 cm^{-1}, 1600 cm^{-1} and 1520 cm^{-1} bands of the ligand (HPicA) in the mixed ligand complexes suggest the coordination of ligand with Cu(II) or Pd(II) through oxygen atom of carboxylate (COO^-) molety

The 1580 cm band of the ligand (HPicA) as shifted by 5 cm^{-1} in the complexes. These features are quite suggestive of the coordination of ligand with Cu(II) or Pd(II) metal through nitrogen atom of pyridine ring.

Electronic absorption spectra

Electronic absorption spectra of the ligand (picolinic acid) and their mixed ligand Cu(II) or Pd(II) complexes were recorded on PERKIN ELMER LAMBDA-15 UV-VIS spectro-photometer in paraffin solvent. The bands observed are given in Table -2

A comparative look of electronic absorption spectral data of the ligand and their complexes indicate that n-p and p-p^* transition of the ligand have shifted to higher frequencies.

The electronic absorption spectra of mixed ligand Cu(II) complexes show broad band at 600 nm with picolinic acid.

The strong absorption in the region 570-580 nm in all the complexes attributed to ligand absorption and charge transfer transition, showing coordination of ligand with metal chelates by L-M interaction. However from the position of spectral bands and their low intensities it is supposed that the complexes are in octahedral geometry.

Magnetic moment

Magnetic moment of mixed ligand transition metal complexes have been measured by Can Faraday magnetic susceptibility balance at 30°C. The magnetic moment values are shown in Table-1.

The spin only value of magnetic moment of Cu(II) octahedral complexes is 1.73 BM corresponding to one unpaired electron. The magnetic moment(μ_{eff}) values for Cu(II) complexes are in the range of 1.75 – 1.86 BM, suggest the octahedral geometry of the complexes. The complexes of Pd(II) are diamagnetic.

Microbiological Studies

Minimum inhibitory concentration(MIC) values ($m\mu gml^{-1}$) of mixed ligand transition metal complexes have been examinaed by Serial dilute method for activity against some bacteria[11], viz., E. coli. S. aureus and fungi[12] viz. C. albicans. Althought a few of the mixed ligand complexes exhibited significant inhibition whereas other did not show any activity. The results are summarized in Table – 3.

The results of antibacterial and antibacterial activity evaluation revealed that four of the oxygen bridged transition metal complexes, $Cu(8HQ)_2$.HPicA, $Cu(1N2N)_2$.HPicA, Pd $(8HQ)_2$.HPicA and $Pd(1N2N)_2$.HPicA were relatively potential inhibitor of the growth of both the test bacteria and one fungi.

The MIC value of $Cu(8HQ)_2$.HPicA & $Pd(1N2N)_2$.HPicA at concentration of 42 μgml^{-1} while $Cu(1N2N)_2$.HPicA & Pd $(8HQ)_2$.HPicA at concentration of 21 μgml^{-1} for E. Coli. The MIC values of $Cu(8HQ)_2$.HPicA, $Cu(1N2N)_2$.HPicA and $Pd(8HQ)_2$.HPicA, $Pd(1N2N)_2$.HPicA at concentration 42 μgml^{-1} and 21 μgml^{-1} respectively for fungi C. albicans. The MIC values of all four mixed lidand transition metal complexes at the same concentration 21 μgml^{-1} for S. aureus.

Structure & bonding

Based on the analytical and spectral(infrared & electronic absorption) studies, the structure and bonding of the newly prepared mixed ligand Cu(II) or Pd(II) complexes involving some chelating organic acids and picolinic acid may tentatively be proposed as shown in Fig. 1.

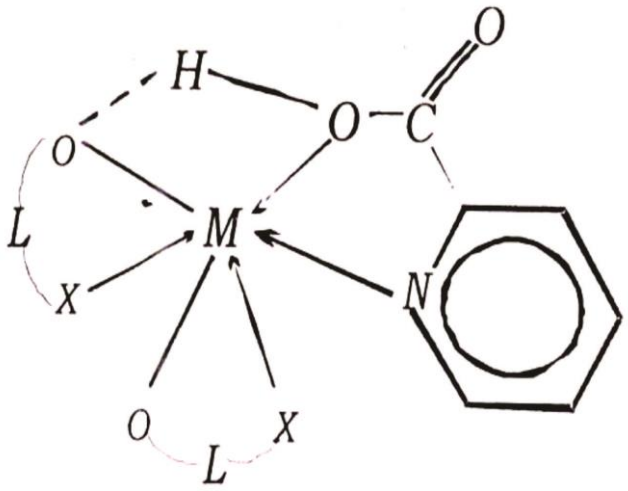

Fig. 1

[Where M = Cu(II) or Pd(II); L = deprotonated 8-hydroxyquinoline or 1-nitroso-2-naphthol; X = O or N].

Table 1:

Compound	Colour	Melting Decomp.	Molar cond.	Magnetic moment	Analysis % found (calcd.) C	H	N	M
Picolinic acid (HPica)	White	135	-	-	-	-	-	
$Cu(8HQ)_2.HPiCA$	Sky blue	280d	2.8	1.75	60.05 (60.15)	3.53 (3.60)	8.86 (8.90)	13.29 (13.36)
$Cu(1N2N)_2.HPiCA$	Light grey	300d	3.1	1.86	58.25 (58.85)	(3.20)	(7.92)	(11.83)
$Pd(8HQ)_2.HPiCA$	Dark yellow	295d	4.5	Diamag.	55.70 (55.80)	3.28 (3.32)	8.12 (8.26)	20.50 (20.72)
$Pd(1N2N)_2.HPiCA$	Greenish black	>300	4.6	Diamag.	54.02 (54.45)	2.54 (2.96)	7.20 (7.32)	18.09 (18.49)

Table 2:

Compound	Selected IR absorption bands (in cm^{-1})					Absoption band (in nm)
Picolinic acid (HPica)	3400br	1650s	1600s	1580s	1520s	283, 235
$Cu(8HQ)_2.HPiCA$	3075w	1640s	1605s	1580w	1500w	600, 570
$Cu(1N2N)_2.HPiCA$	3075w	1640s	1620s	1580w	1500w	600,580
$Pd(8HQ)_2.HPiCA$	3060w	1680m	1600sh	1575s	1500s	645,475
$Pd(1N2N)_2.HPiCA$	3075w	1650w	1600sh	1580s	1500s	645, 480

Br = broad, m = medium, s = strong, sh = shoulder, v = very strong, w = week.

Table 3:

Compound	Minimum inhibitory concentraction (in μgmol^{-1})		
	E. Coli	S. aureus	C. albicans
Cu $(8HQ)_2$.HPCA	42	21	42
Cu$(1N2N)_2$.HPiCA	21	21	42
Pd $(8HQ)_2$.HPCA	21	21	21
Pd$(1N2N)_2$.HPiCA	42	21	21

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