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Synthesis and Characterization of Oxygen Bridged Complexes of Metal Chelates of Isonitrosopropiophenone with Alkali and Alkaline Earth Metal Salts and Study of their Biological Activity

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Abstract: ***In the present study we have synthesized new binuclear complexes of alkali and alkaline earth metal salts with interaction of stable metal chelates of isonitrosopropiophenone of Ni(II) & Cu(II) having general formula*** $[Ma(INPP)_{2.}M_bL]$ ***and*** $[Ma(INPP)_{2.}M_bX]$ ***for alkali metal adducts and*** $[Ma(INPP)_{2.}M_bL_2]$ ***and*** $[Ma(INPP)_{2.}M_bX_2]$ ***for alkaline earth metal adducts where*** M_a***=Ni (II) or Cu(II) and*** $M_b = Li^+$, Na^+ k^+ , Mg^{++} ***or*** Ca^{++}***, L=deprotonated 1-nitroso-2-naphthol and 8-hydroxyquinoline, X=*** ClO_4, SCN^- ***or*** Cl^-***. The IR spectral studies suggests that the Ni(II) & Cu(II) metal chelats act as ligand and coordination towards alkali metal and alkaline earth metal salts takes place through oxygen atom of oximino group. The diffuse reflectance electronic spectra and magnetic moment values of the metal chelates and binuclear complexes with alkali and alkaline earth metal salts indicates the change in geometry during oxygen bridge complex formation. The lower molar conductivities values indicates the covalent nature of the adducts. Some of the complex compounds have shown significant biological activity against the Asperquillus nidulans.***

Keywords: isonitrosopropiophenone, 1-nitroso-2-naphthol, 8-hydroxyqunoline, Biological activity

1. Introduction

In recent years the formation of oxygen bridged binuclear and trinuclear complexes has been well established and extensively investigated [1-23] with the purpose to understand & explore the mechanism of selective absorption of metal ions by plants & animals. The present work deals with oxygen bridged binuclear alkali & alkaline earth metal complexes with "metal complexes as ligand "

Keeping this view in mind , we thought that it would be of interest to study the interaction of Ni(II) & Cu(II) metal chelates with alkali and alkaline earth metal salts .We have selected Ni(II) & Cu(II) metal chelates of isonitrosopropiophenone as "Metal complexes as ligand"

2. Experimental

Materials

Ni(II) & Cu(II) acetate were used of E. Merck quality. The organic acid were used ;
8-hydroxyqunoline and 1-nitroso-2-naphthol of BDH A.R. Quality . isonitrosopropiophenone (HINPP) was prepared by following the procedure of Hurtung and Munch[24-25]

Preparation of Ni (II) & Cu (II) metal chelates of isonitrosopropiophenone (HINPP)

$Ni(INPP)_2$ and $Cu(INPP)_2$ metal chelates were prepared according to the reported method[26-27]

Preparation of alkali/alkaline earth metal salts of organic acids.

95 % ethanolic solution of 0.01 mole/ 0.2 mole of organic acid and 0.01 mole of alkali metal hydroxide /alkaline earth metal hydroxide were mixed. The mixture was refluxed on magnetic hot plate for 1hours with continuous stirring at 80^0 C ,on cooling the resulting solution, a characteristic color precipitate was obtained. It was filtered washed with pure solvent and dried in an electric oven at 100^o C.

Preparation of oxygen bridge complexes(adducts)

Our usual method of synthesis was to take $Ni(INPP)_2$ or $Cu(INPP)_2$ in an absolute ethanol and to add alkali metal/ alkaline earth metal salts to it in 1:1 mole ratio, usually slight excess of alkali metal/alkaline earth metal salts were taken . The reaction mixture was refluxed with constant stirring in hot magnetic plate for about 3 to 4 hours at 60^0 C . The whole substance went in to the solution and subsequently the adducts were precipitated in hot condition during the process of refluxing. They were filtered, washed several times with absolute ethanol and dried in oven at 80^0 C.

3. Results and discussion

The adducts are stable under dry condition , but decompose on exposure to moisture , as such they were kept in a desiccators over anhydrous $CaCl_2$. Some physical properties and analytical data of the metal chelates and their adducts are listed in table-1. From the results, It is evident that the adducts have characteristic color and are different from the metal chelates. All the adducts show high melting/decomposition temperatures which indicate their greater stability.

Molar Conductivities

Molar conductivities (in ohm^{-1} cm^2 mol^{-1}) of compounds were measured in DMSO at 30^o C at a concentration of 10^{-3}

M (table-1). The observed conductance value fall in the range 4-18.5 ohm^{-1} cm^2 mol^{-1} indicate that the complexes are non-electrolytic in nature.

Infrared spectra

Infrared spectra of metal chelates and their adducts were recorded in KBr phase between 4000-200 cm^{-1} with the help of FTIR spectrophotometer. Shimadzu model-8201PC. Selected IR absorption bands of the compounds are given in Table-2.

The band near 1588 ,1555 and. 1245cm^{-1} in Ni(INPP)$_2$ and 1610, 1590 and 1235cm^{-1} in Cu(INPP) had been assigned to the vC=O, or vC=N and vN-O stretching frequency.[26,27] In the alkali /alkaline earth metal adducts of metal chelates of Ni(INPP)$_2$, the C=N frequency shifts to 1560-1575cm^{-1} and the corresponding C=N frequency of Cu(INPP)$_2$ also shows appreciable shift and are observed at 1575-1600cm^{-1} .the slight shift in the C=O frequency is also observed probably due to change in geometry from Trans to Cis form.

The N-O stretching frequency shows significant higher shift to 1250-1280cm^{-1} and 1245-1270 cm^{-1} respectively in the binuclear adducts of Ni(INPP)$_2$ and Cu(INPP)$_2$, suggesting the involvement of oxygen atom of N-O group in coordination with alkali/alkaline earth metal ions.[28]

In the adducts of alkali metal thiocyanate, the thiocyanate peak appears at 2040 cm^{-1}. The shifting of the vC=N to higher frequency(for KSCN it is 2020 cm $^{-1}$)[29], suggest the coordination of alkali metal salts through oximino oxygen of the metal chelates. The medium intensity peaks In the region at 1080-1130 cm^{-1} show that band at region = 1100 cm^{-1} (ionic C1O$^{-}_{4}$) gives only one absorption peak at =1000 cm^{-1}) has split into two peaks. An extra peak at the region 925-958 cm^{-1} has also been spotted. These evidence clearly indicate the presence of coordinated perchlorate [30] in the adducts of alkali/alkaline earth metal perchlorate.

Table 1: Color, Decomposition temperature, molar conductance , magnetic moments & elemental analysis of the complexes

Compound	*Colour*	*Transition (t^{0}C) or decomposition (d^{o}C) temperature*	*Molar Cond. (ohm^{-1} cm^{2}mol^{-1})*	*Magnetic moment values in B.M.*	*%Analysis found/calc.*				
					C	H	N	M$_a$	M$_b$
Ni(INPP)$_2$	Light Green	220d	4.0	3.02	56.23 (56.43)	4.52 (4.18)	7.21 (7.31)	15.09 (15.34)	
Ni(INPP)$_2$.LiIN2N	Brown	>300	----	-----	58.98 (59.81)	4.30 (3.91)	7.36 (7.47)	10.02 (10.45)	
Ni(INPP)$_2$.NaIN2N	Brown	280d	7.3	3.34	57.80 (58.16)	4.20 (3.80)	7.12 (7.27)	9.86 (10.16)	3.78 (3.98)
Ni(INPP)$_2$.KIN2N	Reddish Brown	300d	6.7	3.37	55.82 (56.58)	4.12 (3.70)	6.95 (7.07)	9.68 (9.88)	6.34 (6.56)
Ni(INPP)$_2$.Na8HQ	Light yellow	>320	8.2	3.74	58.03 (58.94)	4.30 (4.01)	7.51 (7.64)	10.38 (10.68)	3.94 (4.18)
Ni(INPP)$_2$.K8HQ	Light yellow	>320	7.8	3.63	56.38 (57.26)	4.11 (3.88)	7.31 (7.42)	10.05 (10.37)	6.58 (6.89)
Ni(INPP)$_2$.NaCLO$_4$	Green	285d	11.2	3.34	42.02 (42.71)	3.37 (3.16)	5.38 (5.53)	11.31 (11.60)	4.29 (4.54)
Ni(INPP)$_2$.KSCN	Dull White	212d	12.6	3.38	46.83 (47.51)	3.51 (3.33)	8.47 (8.75)	12.01 (12.23)	7.86 (8.14)
Ni(INPP)$_2$.KI	Green	265d	----	----	38.59 (39.36)	3.32 (2.91)	4.87 (5.10)	10.46 (10.70)	6.91 (7.12)
Ni(INPP)$_2$.Mg(8HQ)$_2$	Yellow	>320	----	----	61.84 (62.18)	4.31 (4.03)	7.86 (8.06)	8.20 (8.45)	3.31 (3.45)
Ni(INPP)$_2$.Mg(ClO$_4$)$_2$	Light Green	290d	11.7	3.54	34.69 (35.66)	3.02 (2.64)	4.40 (4.62)	9.41 (9.69)	3.79 (3.96)
Ni(INPP)$_2$.CaCl$_2$	Brownish green	292d	----	3.38	42.85 (43.75)	3.51 (3.24)	5.26 (5.67)	11.02 (11.89)	7.61 (8.10)
Cu(INPP)$_2$.NaIN2N	Brown	210d	7.5	2.10	56.87 (57.76)	3.98 (3.77)	6.92 (7.20)	10.08 (10.90)	3.58 (3.94)
Cu(INPP)$_2$. KIN2N	Reddish Brown	205d	7.0	1.99	55.26 (56.12)	3.97 (3.67)	6.69 (7.01)	10.15 (10.61)	6.23 (6.52)
Cu(INPP)$_2$.Li8HQ	Greenish Brown	282d	---	----	59.46 (60.16)	4.39 (4.08)	7.26 (7.79)	11.50 (11.79)	
Cu(INPP)$_2$.Na8HQ	Brown	>300	----	2.26	57.96 (58.42)	4.21 (3.96)	7.21 (7.57)	11.08 (11.45)	3.90 (4.14)
Cu(INPP)$_2$.K8HQ	Brown	225d	7.4	2.18	55.46 (56.77)	4.12 (3.85)	6.96 (7.36)	10.86 (11.13)	6.31 (6.85)
Cu(INPP)$_2$.NaClO$_4$	Sky Blue	185d	11.2	1.86	38.69 (39.83)	2.58 (2.48)	5.36 (5.80)	12.78 (13.18)	4.31 (4.77)
Cu(INPP)$_2$.Mg(ClO$_4$)$_2$	Sky Blue	202d	18.5	1.84	31.98 (32.96)	2.25 (2.05)	4.38 (4.80)	10.31 (10.90)	3.86 (4.11)
Cu(INPP)$_2$.Mg(8HQ)$_2$	Greenish Brown	285d	-----	-----	38.69 (39.83)	2.58 (2.48)	5.36 (5.80)	12.78 (13.18)	4.31 (4.77)

Table 2: IR Absorption Bands in cm^{-1} of metal chelates and their adducts.

Adducts	$vCLO^-_4$/ SCN^-	vC=O	vC=N	vN-O	vM-N	vM-O
Ni(INPP)$_2$		1588s	1555m	1245vs	500m	355br,390s
Ni(INPP)$_2$.NaCLO$_4$	1125m,1103m, 1082s,925s	1595s	1575m	1555vs	502s	375br,390vs,280m
Ni(INPP)$_2$.KSCN	2040vs	1595s	1275m	1250vs	500m	405br,390br,290m
Ni(INPP)$_2$.KI		1595s	1570m	1255vs	502m	360s,275m,280m
Ni(INPP)$_2$.NaIN2N		1595s	1560pr	1260vs	---	---
Ni(INPP)$_2$.Na8HQ		1600m	1570s	1270vs	---	---
Ni(INPP)$_2$.Mg(8HQ)$_2$		1590s	1575sh	1260s	---	---
Ni(INPP)$_2$.CaCl$_2$		1595vs	1570vs	1280vs	---	---
Cu(INPP)$_2$		1610m	1590m	1235s	505s	415s,330s
Cu(INPP)$_2$.NaClO$_4$	1128s,1102m,1080s, 1028sh,955s	1620vs	1600sh	12345s	515vs	420s,365s,335s,260s
Cu(INPP)$_2$.Mg(ClO$_4$)$_2$	1128m, 1102m, 1080s,958m	1620m	1600sh	1245s	515s	421vs,370m,330vs
Cu(INPP)$_2$.K8HQ		1615m	1595s	1250vs	505m	430vr,330s,270m
Cu(INPP)$_2$.Mg(8HQ)$_2$		1600m	1575vs	1270vs	500vs	395s,330s,310vs
Cu(INPP)$_2$.NaIN2N		1610sh	1575s	1255s		
Cu(INPP)$_2$. KIN2N		1600sh	1580s	1250sh		

Further in the low frequency region, the band observed at 490 and 390 cm^{-1} in Ni(INPP)$_2$ and 505 and 415 cm^{-1} in Cu(INPP)$_2$ and assigned due to v(M-N) and v(M-O) mode of vibration. The appearance of the additional bands around 395-310 cm^{-1} are probably due to the bridging oxygen metal bond in adducts of chelates of Ni(INPP)$_2$ and Cu(INPP)$_2$. These evidences show that adducts formation taking place through oxygen atom of oximino group.

Magnetic Measurements

Magnetic measurements were taken on Cahn-Faraday electronic balance at the temperature range 32 to 34.5^{0}C . The result of the magnetic measurements are given in table 1.

The magnetic moment of Ni(INPP)$_2$ has been reported 3.02 B.M. [27] at 298K, Which is slightly greater than that required for two unpaired electronics and is in the range generally observed for the octahedral Ni(II) complexes [31] The binuclear alkali/alkaline earth metal adducts of the Ni(INPP)$_2$ chelates posses magnetic moment values between 3.34 and 3.748. M. which are higher than the reported values for the most of the octahedral Ni(II) complexes, but occur in the range of μ_{eff} value reported for tetrahedral complexes.

In the present investigation of the magnetic moment of Cu(INPP)$_2$ has been found to be 1.21B.M at room temperature which is markedly lower than the spin only value of 1.73B.M Similar subnormal values has been also reported [32] in the Cu(II) , (INAP)$_2$ chelated and suggested that this lower magnetic moment values is due to antiferromagnetic interaction and this may arise through molecular association involving oxobridges.

The binuclear alkali/alkaline earth metal salt adducts od Cu(INPP)$_2$ display magnetic moment values between 1.84 to 2.13 B.M.

Thus it is clear that the binuclear adducts are magnetically dilute and their magnetic moment values fall in the range of planar Cu(II) complexes.

Electronic absorption spectra

All diffuse reflectance electronic spectra were recorded on SHIMADZU UV-VIS 160A spectrophotometer in Nujol mull/paraffin liquid . The bands observed in electronic spectra of Ni(INPP)$_2$ and Cu(INPP)$_2$ & their adducts are given in table-3

Table 3: Major diffuse reflectance bands (nm) of metal chelates and their adducts.

Compound	Diffuse reflectance (nm)
Ni(INPP)$_2$	1000w,605w,367br,342m,300m,260br
Ni(INPP)$_2$.NaCLO$_4$	665w,360br,343s,263m,238m
Ni(INPP)$_2$.KSCN	650w,362br,222s
Ni(INPP)$_2$.KIN2N	655sh,463br,400br,227s
Ni(INPP)$_2$.K8HQ	635sh,400br,260br
Cu(INPP)$_2$	616bw,412m,250s
Cu(INPP)$_2$.NaClO$_4$	1027w,675w, 412s,233s
Cu(INPP)$_2$. KIN2N	1080w,700sh,343m,301m,248br
Cu(INPP)$_2$.Mg(ClO$_4$)$_2$	1050w,685sh,668br,342m,301m,343s

Br=broad, s=strong,m=Medium,w=weak, sh=shoulder and h=hump

The light green complex Ni(INPP)$_2$ in chloroform solution reveals an intense band at 342nm which is ascribed to change-transfer transition. The spectrum also reveals a peak at 1000nm and a broad hump near 605nm which are attributed to spin allowed transitions $3_{A2g} \rightarrow 3_{T2g}$ and $3_{A1g} \rightarrow 3_{T1g}$ (F) (v_2) respectively, in Ni (II) complexes. The frequency of the third transition due to $3_{A1g} \rightarrow 3_{T1g}$ (P) (v_3) is not observed in the spectrum of the complex, probably because it is masked by the tail of high intensity charge-transfer band around 350nm.

The diffuse reflectance spectrum of the solid complex is similar to the solution spectrum except that the position of the charge transfer band is shifted to 367nm.[27]

The spectra of binuclear adducts are different in nature and the band usually observed in 635-665nm.The band in this region may be assigned due to 3_{T1}(F)→ 3_{T1}(P) transition. [33] Lower energy bands due to the transition 3_{T1}(F)→ 3_{A2}(F) and 3_{T2}(F)→ 3_{T2}(F) could not be recorded due to limitations of the instrument.

Hence, the nature of absorption bands of alkali/alkaline earth metal adducts suggests that octahedral geometry of

$Ni(INPP)_2$ has been probably changed into tetrahedral geometry; this fact is also supported by magnetic moment studies. The diffuse reflectance spectra of the $cu(INPP)_2$ complexes in solid state display bands in region 250-412nm due to charge transfer. A weak band also exhibit at 616nm, which is attributed due to d-d transition.

On Comparison with the spectra of alkali/alkaline earth metal adducts, the band at region 616nm has shifted to higher wave length 675-700nm and additional weak band has been observed at region 1027-1050nm.
Similar observation have also been reported [34] by sacconi and Ciampolini. They examined the electronice spectra of a number of Cu(II) complex compound and found that the planar complex have a single band in the range 680-710nm, Where as the pseudo-tetrahedral complexes have bands at 1000-1100nm and 780-800nm, which hey assigned on the basis of their frequencies and relatively low intensities to crystal-field transitions of the Copper(II) ion in pseudo-tetrahedral environment.

Hence, the nature of the absorption bands of alkali/alkaline earth metal adducts of $Cu(INPP)_2$ suggest the adduct information. This fact is also supported by magnetic moment studies.

Antifungal studies

Metal chelates and their oxygen bridged complexes of alkali metal salts were screened for antifungal activity. The tested complex compounds exhibited variable antifungal activity as shown in Table-4 & 5. Among the metal chelate adducts , the $Ni(INPP)_2$. $NaClO_4$ showed efficient antifungal activity against *Aspergillus nidulans* with zone of inhibition 28mM diameter with concentration of 2.00mM.

Table 4: Effect of the metal chelates and oxybridged alkali metal salt adducts on the growth of *Aspergillus nidulans*

Metal chelates and their alkali metal salts adducts	Concentration (mM)	Growth* (Colony diameter in mm)	Percent growth
Control	----	28	100.00
$Cu(INPP)_2$	2 mM	---	00
$Ni(INPP)_2$	2 mM	10	35.70
$Ni(INPP)_2 . NaClO_4$	2 mM	---	00

Table 5: Effect of varying concentrations of $Cu(INPP)_2$ and $Ni(INPP)_2$ $NaClO_4$ and on the growth *Aspergillus nidulans*

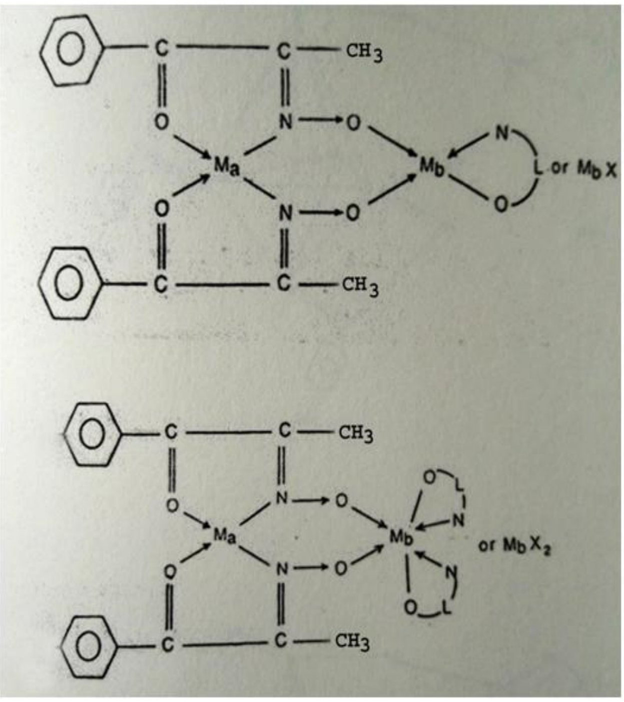
Metal chelates and their alkali metal salts adducts	Concentration (mM)	Growth* (Colony diameter in mm)	Percent growth
Control	----	28	100.00
$Cu(INPP)_2$	0.8 mM	11	40.00
	1.2 mM	07	25.00
	1.8mM	02	7.15
$Ni (INPP)_2 . NaClO_4$	1.0 mM	04	14.30
	1.75mM	02	7.15
	2.0mM	---	---

*Measured after 48 hours 37°C on solid medium containing $NaNO_3$ as the sole source of nitrogen

Structure & Bonding

On the basis of elemental analysis , the molecular formula of binuclear alkali/alkaline earth metal adducts has been suggested as $[M_a (INPP)_2.M_bL]$ and $[M_a (INPP)_2.M_bX]$ for alaki metal adducts and $[M_a (INPP)_2.M_bL_2]$ and $[M_a (INPP)_2.M_bX_2]$ for alkaline earth metal adducts where M_a =Ni (II) or Cu (II)and M_b=Li^+ , Na^+ , K^+ , Mg^{++} or Ca^{++} , L=deprotonated 1-nitroso-2-napthol and 8-hydroxyquinoline, X= ClO^-_4 , SCN^- or Cl^- .

The infrared spectral studies of these adducts, suggest that , complex ligand , i.e. Ni(II) and Cu(II) chelates of isonitrosopropiophenone acts as a Lewis base to the Lewis acid (alkali/alkaline earth metal salts); Coordination is through oxygen atom of oximino group. The probable newly prepared oxygen bridged complexes may be produced schematically as such.



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