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Characterisation of Nickel(II) Complexes with Some Polymeric Resins as Ligands

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***Abstract**: The paper describes synthesis of Nickel(II) complexes with some polymeric resins derived by the reaction of urea/ ethylenediamine, formaldehyde and ketooxins of2-hydroxy-4-methylacetophenone and 2-hydroxybenzophenone as ligand. The characterization of complexes were carried out with the help of a I.R, magnetic moment, analytical analysis and UV spectralanalysis. Ligand filled parameters were also calculated.*

Keywords: Ketooxin, Polymeric Resin / Coordination Compounds.

1. Introduction:

The synthesis of coordination compounds with polymeric ligands having unusual electric and magnetic properties have been a subject of study in recent past [1-3]. The synthesis of polymeric resinsobtained by condensingketooxins, (oxins of 2-hydroxy-4-methyl acetophenone and 2-hydroxy benzophenone), urea or ethylene diamine and formaldehyde have been reported earlier. [4-7] The present paper deals with the synthesis and characterization of Ni (II) complexes using such polymeric resins.

The characterization was made by elemental analysis, U.V. and spectral analysis.

2. Experimental:

2.1 Materials and methods

All the chemicals were used of AR gradeTheir purity has been examined by TLC and melting point. The infrared spectrum of the complexes were recorded in KBr pallet on PerkinElmer 1000FTIR spectrophotometer. The magnetic susceptibility of complexes were measured by Guoy method at room temperature

2.2 Synthesis of Nickel (II) Complexes:

2- Hydroxy -4- methylacetophernocoxime-urea-formaldehyde resin and 2-hydroxy benzophenoneurea formaldehyde resins were synthesized by the methodearlier reported [4].

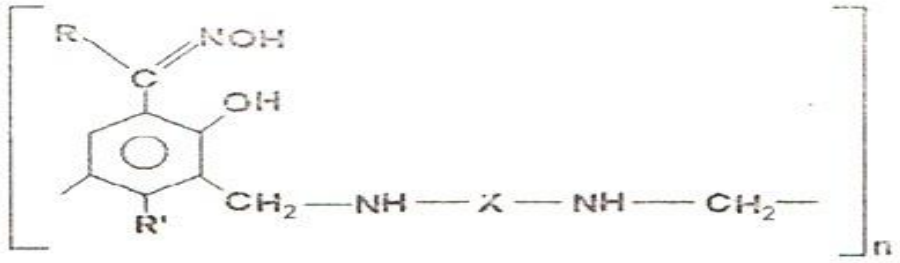
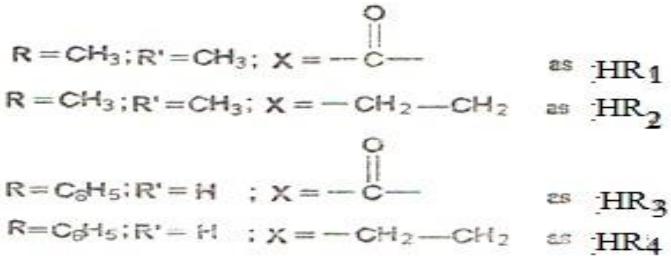


Figure:- 1

Where



The resins(0.01 mole)were dissolved in DMF (100ml) consuming a slight amount of water. To it Ni(II) Nitrate was added slowly with constant stirring. A yellowish green solid was precipitated out at 6.5 pH where an aqueous saturated solution of sodium acetated was added. The product was digested for some time on water both, it was filtered and washed with DMF, hot water and driedat 60°C.

3. Results and Discussions

The physical characteristics, room temperature, magnetic moment values and analytical data for the complexes are presented in table-1

TABLE - 1

Physical constants, magnetic moment and analytical data of Ni(II) complexes

Name of Complexes	μ_{eff} at 298^{0}K in B.M.	Decomposition Temperature ^{0}C	% Yield	Colour	% N	% Ni
$[Ni(R_1)_2(H_2O)_2]$ HR_1	3.10	268^{0}C	75	Purple	15.3 (15.46)	5.0 (5.15)
$[Ni(R_2)_2(H_2O)]$ HR_2	0.01	270^{0}C	80	Black	14.5 (14.98)	4.5 (4.99)
$[Ni(R_3)_2(H_2O)]$ HR_3	3.01	268^{0}C	70	Deep Brownish Red	14.5 (14.98)	4.5 (4.99)
$[Ni(R_4)_2(H_2O)_2]$ HR_4	3.10	270^{0}C	80	Deep Brownish Red	13.00 (13.02)	4.2 (4.34)

The analytical results have indicated that the complexes have composition $[Ni(R_4)_2\ 2H_2O]$ for ligands $(HR_2)$and (HR_4) and $[Ni(R)_2H_2O]$ for ligand (HR_1) and (HR_3). The thermal study of the complexes indicated that the chelates with (HR_1) and (HR_3) correspond to loss of only one water molecule while in the case of chelates with (HR_2) and (HR_4) there were loss of twowater molecules on heating at 120°C. This shows one and two coordinated water molecule respectively in two type of coordination compound.The complexes were highly insoluble in water and common organic solvents.

The magnetic moment data of the complexes were found in the range of 2.38BM indicating that it possess two unpaired electrons and a ^{3}F ground term with spin only magnetic moment 2.38BM.

3.1 Electronic spectral studies:

Electronic absorption spectra of nickel(II) complexes have been recorded in the region 240 to 1000 nm using diffuse reflectance method and are presented in table-2.

TABLE - 2

Electronic spectral bands of Ni(II) complexes (Ignoring the internal ligand transition)

(In nm)

Name of Complexes	U.V.Spectral Data	$^3A_2g \rightarrow {}^3T_2g$ (ν_1)	$^3A_2g \rightarrow {}^3T_1g$ (ν_2)	$^3A_2g \rightarrow {}^3T_1g(P)$ (ν_2)
$[Ni(R_1)_2(H_2O)]$ HR_1	440	990	640	398
$[Ni(R_2)_2(H_2O)_2]$ HR_2	440	995	637	398
$[Ni(R_3)_2(H_2O)]$ HR_3	440	990	635	398
$[Ni(R_4)_2(H_2O)_2]$ HR_4	440	993	637	395

We believe that bands around 397+2nm, 650+5nm and 993+3nm maybe safely assigned to transitions $^3A_2g \rightarrow {}^3T_1g$=v3, $^3A_2g \rightarrow {}^3T_1g(F)=\nu_2$ and $^3A_2g \rightarrow {}^3T_2g(F)=\nu_1$ respectively with an octahedral stereo-chemical arrangement. The results were further substantiated byν_2/ν_1 ratio in the range of 1.55. The reaming bands due to inter ligand transition were found almost interact in complexes. A ligand field parameter have been calculated by the method described in literature[6] and tabulated in table- 3

TABLE - 3

Ligand field parameters for Ni(II) complexes

Name of Complexes	Experimental Data (in cm^{-1})			ν_2/ν_1	B	β	β_0	Calculated Values (in cm^{-1})		
	ν_1	ν_2	ν_3					ν_1	ν_2	ν_3
$[Ni(R_1)_2(H_2O)]$ HR_1	10101	15625	25100	1.54	694.80	0.65	35%	10125	15802	24994
$[Ni(R_2)_2(H_2O)_2]$ HR_2	10050	15698	25100	1.56	709.86	0.67	33%	10050	15763	25034
$[Ni(R_3)_2(H_2O)]$ HR_3	10101	15748	25250	1.55	713.00	0.67	33%	10101	15841	25156
$[Ni(R_4)_2(H_2O)_2]$ HR_4	10070	15698	25300	1.55	739.00	0.69	31%	9971	15710	25188

3.2 I.R Spectral studies:

I.R spectral data for nickel(II) polymeric chelates have been presented in table-4.

TABLE –4

I.R Spectral Bands of Ni (II) complexes (in cm^{-1})

Complexes	ν_{H_2O}	ν_{CH_2} Briding	$\nu_{C=N}$	$\nu - \overset{O}{\overset{\|}{C}} - NH$	ν_{N-O}
$[Ni(R_1)_2(H_2O)]$ HR_1	3250-3400 (B&m)	2800-3100 (w&b)	1590-1600 (S)	1275 (m)	960-1000 (m)
$[Ni(R_2)_2(H_2O)_2]$ HR_2	3248-3400 (B&m)	2799-3100 (w&b)	1589-1600 (S)	--	958-1000 (m)
$[Ni(R_3)_2(H_2O)]$ HR_3	3248-3400 (B&m)	2799-3100 (w&b)	1590-1600 (S)	1274 (m)	960-1000 (m)
$[Ni(R_4)_2(H_2O)_2]$ HR_4	3250-3400 (B&m)	2799-3100 (w&b)	1588-1600 (S)	--	960-1000 (m)

The table revealed that phenolic OH group which observed around 2725cm^{-1} was absent in all polymeric chalets. It is inferred that the phenolic group was deprotonated and involved in coordination. The >C=N group of oxide absorbed around 1630 cm^{-1} in resins but it suffers a lower shift in the polymeric chelates and absorbed around 1590-1600 cm^{-1}.This clearly indicated the involvement of nitrogen loan pair ofoximegroup in coordination.In resins $HR_1$and HR_3 there are carbonyl group which absorbed around 1474 and 1450 cm^{-1}.This peak was almost absent in the corresponding complexes with HR_2 and HR_4 and a new peak around 1275 cm appeared in the complexes We infer that the oxygen atom of carbonyl group was involved in coordination and overlapped with >C=N group. The hump near 3250 to 3400 cm^{-1} again indicated the presence of coordinated water molecules. Such involvement of carbonyl group may not be expected in case of $HR_2$and HR_4.

The -CH_2bridging group remainedintact in polymeric chelates but vN-o absorption registered a shift to higher region which again supported the involvement of nitrogen ofoxime atom and coordination.

4. Conclusion:

Thus a tentative structure for the synthesis of all Nickel(II) chelates may be assigned as-fig-2 and fig-3

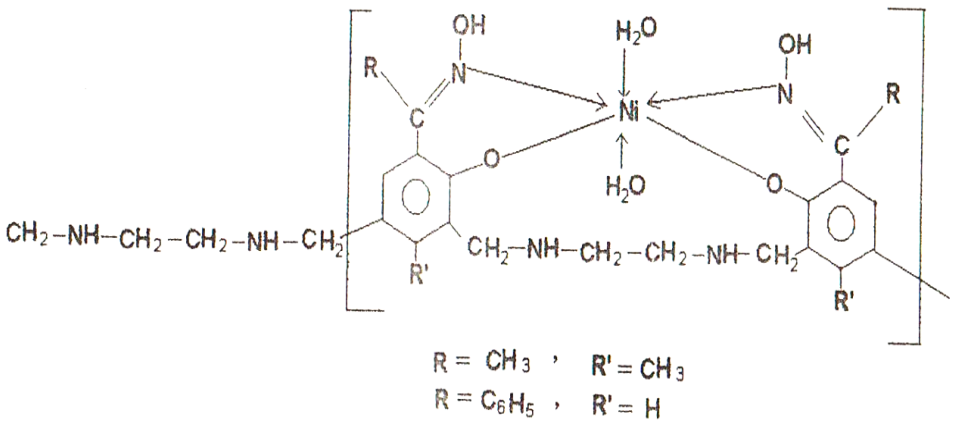


Fig.-2

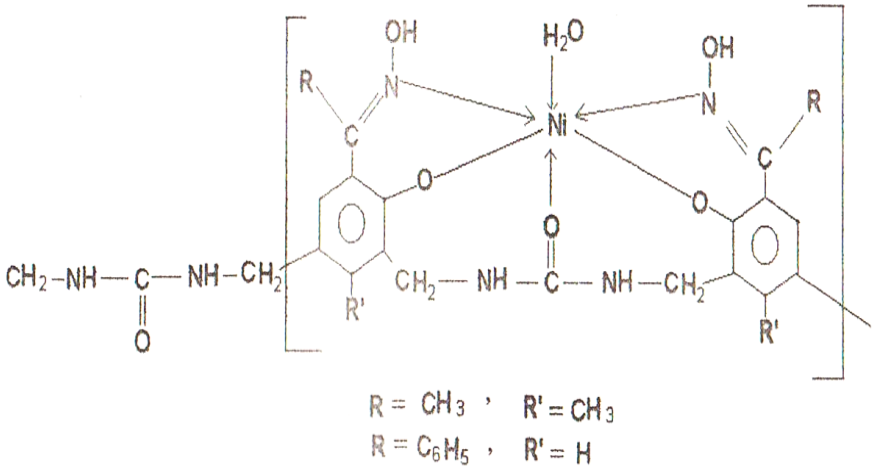


Fig.-3

5. Acknowledgement:

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