

SYNTHESIS AND CHARACTERIZATION OF SOME COMPLEXES OF Ni(II) WITH L-GLYCINE, L-PROLINE, L-LYSINE.

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Article Received on 17/08/2020

Article Revised on 07/09/2020

Article Accepted on 27/09/2020

ABSTRACT

A Series of new Ni(II) complexes with Amino acids (Glycine, Proline, Lysine) were synthesized & characterized by element analysis, molar electrical conductivities, UV, Magnetic Susceptibility, IR and Cyclicvoltametric technique. Electrochemical studies of the complexes reveal that complexes Ni(Gly)₂, [Ni(Pro)₂] and [Ni(Lys)₂] show a simple irreversible reduction wave in the cathodic region. All the complexes were found to be (2:1) electrolyte nature with the general formula [Ni(L₂)] where L is amino acid legend.

KEYWORDS: element analysis, molar electrical conductivities, UV, Magnetic Susceptibility, IR.

1. INTRODUCTION

Coordination complexes are gaining importance in recent years especially in the designing of long acting drugs in metabolism. The metal complexes from bidentate ligands have often been studied recently because of their technical applications^[1,2] and applications in enhancement of drug action.^[3,4] Transition metals are essential for normal functioning of living organism and are, therefore, of great interest as potential drugs.^[5] The coordination chemistry of nitrogen donor ligands is an active area of research.

The modern chemotherapy is promoted on the basis of metals and metal complexes which play a key role in the pharmacological properties of known drugs. There are many metal ions are play very important roles in biological activities in the human body.^[7,8] Ni (II) complexes have attract attention due to their biological applications and coordination modes. When it bound to metals and act as high pharmacological and good chelating agents. Complexes of Ni (II) with amino acids are used as models to study the pharmacological effects of drugs and lowering toxic effects of some metal ions.^[6,7]

Amino acids are basic 'building block' that combine to proteins are chemical species indispensable for performing a huge number of biological function as described by the role of enzyme and important class of bio molecule can act as potential oxygen and donar ligand, it has occurred that helpful their functional groups as fully as possible in metal coordination^[8] Amino acid coordinates to metals it confirms structural liability

and also have applicability in enzyme inhibition.^[9] These complexes have a vast area in pharmacological and toxicological properties that has drawn lot of current attention. Therefore, this attraction we were synthesized the new Ni (II) complexes containing L-Glycine, L-Proline and L-Lysine as ligands. This paper has increased focus on the complexation of Ni(II) with amino acids and characterization by elemental analysis, Molar conductivity measurement CV, IR, UV-Visible.

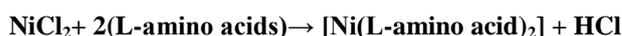
2. EXPERIMENTAL METHOD

2.1 Chemical and reagent

L-Glycine L-Proline, L- Lysine, dinitrosalicylic acid, p-nitrophenyl- α -D-glucopyranoside, Sodium Chloride, Sodium Diphosphate, Disodium Phosphate were purchased from SRL, India. While NiCl₂, rat intestinal acetone powders were procured from Sigma-Aldrich, and DPPH, ABTS was brought from Alfa-Acer. All chemicals other chemicals i.e. ethanol, sodium hydroxide, water were synthetic grade and used without further purification.

2.2 Synthesis of Complexes at 6-7 pH

2 mM of amino acids (Glycine, Proline, Lysine) were dissolve in 30 ml water a transparent solution were obtained. In above solution of 1 mM of NiCl₂ were mixed drop by drop with continuous stirring, a blue/deep blue solution were obtained. The excess solvent was removed by evaporation to facilitate precipitation of the complex on cooling.^[10] The General reaction for the preparation of complexes of nickel is as follows:



2.3 Conductivity measurement

Molar Conductivity of the complexes was measured by using an elico digital conductivity bridge model CM-88 using freshly prepared solution of the complex in methanol.^[11]

2.4 Electronic Spectra

Electronic (UV-Vis) spectra were recorded on a Shimadzu 1800 spectrophotometer using 10-mm quartz cells. All spectra of complexes were recorded in aqueous solution, with same concentration (0.03 mg/ml).^[12]

2.5 Magnetic susceptibility

Magnetic susceptibilities of the nickel (II) complexes were subjected to measure the susceptibility by the modified Gouy method at room temperature using Magnetic Sherwood Balance in MNIT (Allahabad).

2.5 IR Spectroscopy

Infrared (IR) spectra were obtained by the KBr pallet method using a Bruker Alfa-T model Fourier transform (FTIR) spectrometer (Bruker Instrument, Germany). The spectrometer was equipped with a Globar IR source, KBr beam splitter, and detector. For each spectrum, 16 scans were obtained with the resolution of 4 cm⁻¹. The obtained IR spectra were processed by means of the program OPUS 7.0.^[13]

2.5 Cyclic Voltammetric studies

The cyclic voltammetric measurements were carried out with a Metrohm auto lab Instrument having an electrochemical cell with a three-electrode system. The auxiliary electrode was an Ag /AgCl₂. Glassy carbon was used as a working electrode, while a platinum wire electrode used as a reference electrode. The concentration of complexes was taken 0.3 mg/ml, dissolved in supporting electrolyte 10 ml of 0.01 M solution of sodium perchlorate (NaClO₄) solution.^[11]

3 RESULTS AND DISCUSSION

3.1 Molar Conductance

The molar conductance of Ni(II) complexes of amino acids [Ni(Gly)₂Cl₂], [Ni(Pro)₂Cl₂], [Ni(Lys)₂Cl₂] was studied at 3×10⁻³M concentration in room temperature. The values of conductivity are summarized in Table 1. All complexes were dissolved in Methanol. The molar conductance data varies Ni(II) complexes [Ni(Gly)₂Cl₂], [Ni(Lys)₂Cl₂] [Ni(Pro)₂Cl₂], are found 220.03, 165.2 and 234 cm²mol⁻¹ respectively indicating that the complexes were found 2:1 electrolytic nature. The molar conductance data revealed that metal to ligand molar ratio is 1:2 for complexes.^[13]

3.3 UV Visible Spectroscopy & Magnetic Susceptibility

The absorption spectra of nickel (II) chloride and their complexes were recorded in DMSO (according to their solubility) wavelength ranges from 1100 to 200 nm were recorded at room temperature band position of the

absorption maxima and proposed geometry are listed in **Table 1** and **Fig 1.1-1.4**.

Electronic spectra of Ni(II) d-d transition band the in the regions 721, 653 nm. These are ascribed to the spin-allowed transitions 3A_{2g}→3T_{2g}(ν₁), 3A_{2g}→3T_{1g}(ν₂), and 3A_{2g}→3T_{1g}(ν₃) respectively, consistent with their well-defined octahedral configuration[14]. All complex of Ni pure LLCT (ligand to ligand charge transfer) bands are recorded at 275, 385 nm is assigned to π-π* transition. Metal to ligand charge transfer (MLCT) are obtained at 460 and 502 nm is assigned to n-π* transition. The presence of π-π*, n-π* transition in all complexes indicate the functional group of ligand.^[15]

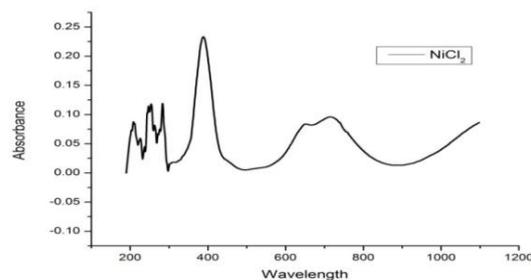


Fig: 1.1 Electronic Spectra of NiCl₂.

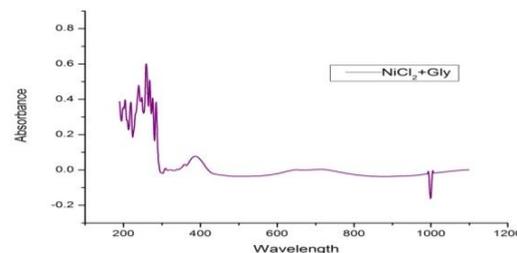


Fig: 1.2 Electronic Spectra of [Ni(Gly)₂Cl₂] Complex.

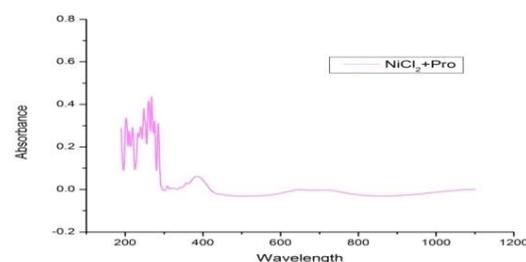


Fig: 1.3 Electronic Spectra of [Ni(Pro)₂Cl₂] Complex.

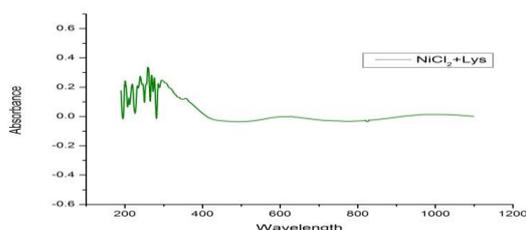


Fig: 1.4 Electronic Spectra of [Ni(Lys)₂Cl₂] Complex.

Table1. Band Assignment of UV-Vis. Spectra, Molar conductance of Ni(II) and its complexes.

Complex	Band assignment	Wave length Found (nm)	Molar Conductance (electrolyte type)
NiCl ₂	V ₁ (³ A _{2g} - ³ T _{2g})	725	280.12
	V ₂ (³ A _{2g} - ³ T _{1g})	654	
	V ₃ (³ A _{2g} - ³ T _{1g})	389	
[Ni(Gly) ₂ Cl ₂]	π - π^* , n- π^*	275, 385	220.03
[Ni(Pro) ₂ Cl ₂]	V ₃ (³ A _{2g} - ³ T _{1g}), n- π^*	460,376	165.01
[Ni(Lys) ₂ Cl ₂]	n- π^* , V ₂ (³ A _{2g} - ³ T _{1g})	392,502	234

3.4 Infrared Spectroscopy

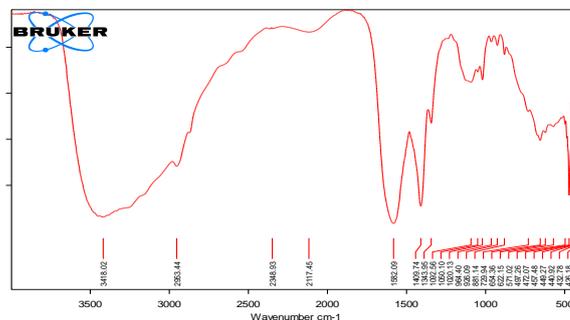
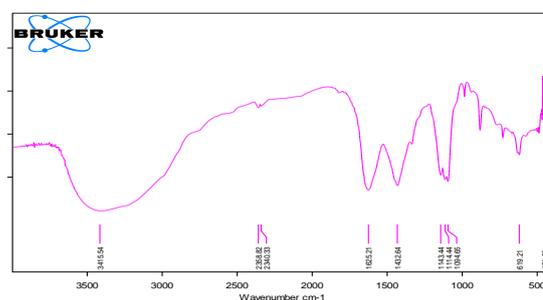
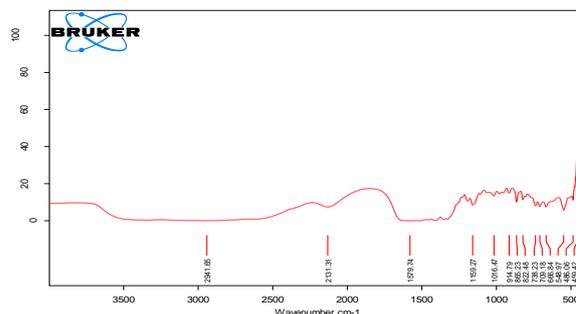
The Infrared (IR) spectroscopy is most widely used spectroscopic techniques employed mainly by inorganic and organic chemists due to its usefulness in determining structures of compounds and identifying them. Chemical compounds have different chemical properties due to the presence of different functional groups.

The important band in [Ni(Gly)₂] complex exhibited a broad band in the region 3419 and 2114 suggesting the presence of (OH) molecule and NH₂ frequencies. The band observed in the,1522, 1408 are attribute to asy COO- and sCOO stretching vibration. The band indicating that 776,753 assigned to be COO- (scissor and CH out of plane deformation) and other frequency found that in 698, 645 assigned to be COO-(wag and NH₂ rock) in complexes. These results agree with reported this studies.^[16]

The important band in [Ni(Pro)₂] complex exhibited a broad band in the region 3415 suggesting the presence of OH molecule. The band observed in the 1597, 1428 are attribute to asy COO- and sCOO stretching vibration.

⁺CH bend. The band indicating that 1066, 947 are assigned to be CH₃ bend and CN stretching. (scissor and CH out of plane deformation) and other frequency found that in 696,644 assigned to be COO-(wag and NH₂ rock) in complexes. These results agree with reported this studies.^[17]

The important band in [Ni(Lys)₂] complex not exhibited a broad band, suggesting absent of (OH) molecule and NH₂ frequencies. The band observed in the 1579, 1409 are attribute to asy COO- and sCOO stretching vibration.

**Fig: 2.1 IR Spectrum of [Ni(Gly)₂Cl₂] complex.****Fig: 2.2 IR Spectra of [Ni(Pro)₂]Cl₂ complex.****Fig: 2.3 IR Spectra of [Ni(Lys)₂]Cl₂ complex.****Table: 3. Most Significant FTIR band Spectra of the Ni(II) and its complex.**

S.N	Complex	Group Assignment	Band (cm-1)
1	Ni(Gly) ₂ Cl ₂	ν(OH)	3419
		CH ₂ sym sterch	1522
		vas COO ⁻ strech	1408
2	Ni(Pro) ₂ Cl ₂	OH	3415
		vasCOO ⁻	1597
		νsym COO	1428
		CH ₂	1143
3	Ni(Lys) ₂ Cl ₂	vasCOO ⁻	1579
		νsym COO ⁻	1409
		COO-Scissor and CHout of plane	1078

3.5 Cyclovoltametry

Cyclic voltammetry is the most flexible electro analytical technique for the study of electro active species. The electrochemical behavior of the three amino acids nickel complex were recorded in 20 Mm KCl Solution at 100 scan rate. The Fig 3.1 of Cyclic voltammogram of $[\text{Ni}(\text{Gly})_2]$ show different reduction peak in cathodic direction which are assigned at $E_{pc} = -0.439$ and the oxidation peak $E_{pa} = -0.351$ at 100 scan rate. The Fig 3.2, 3.3 does not any peak found because voltammogram clearly represent that reduced moiety of Ni(II) complexes does not fully oxidised in further sweep.^[18]

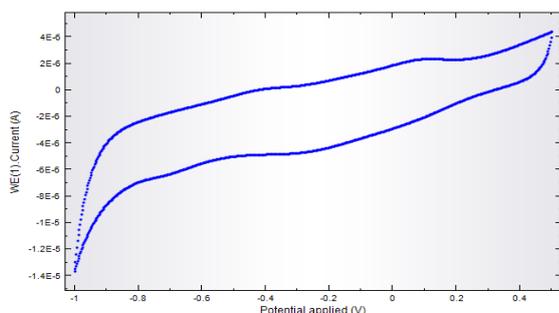


Fig: 3.1 Cyclic Voltammogram of $[\text{Ni}(\text{Gly})_2]\text{Cl}_2$ Complex.

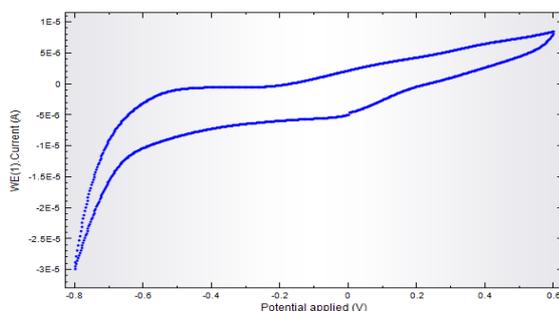


Fig: 3.2 Cyclic Voltammogram of $\text{Ni}(\text{Pro})_2\text{Cl}_2$ Complex.

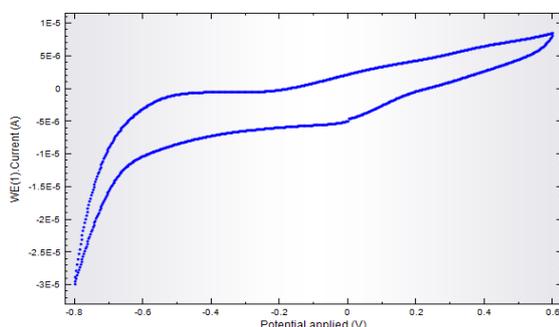


Fig 3.3 Cyclic Voltammogram of $[\text{Ni}(\text{Lys})_2]\text{Cl}_2$.

5. CONCLUSION

Complexes of metal ions with amino acids can be assigned as a perfect models to study the pharmacological active effects of drugs and also lowering toxic effects. The considerable fact is interactions between transitional metal ions and amino acids. A series of complexes of Ni(II) and amino acid i.e.

L-Glycine, L-Proline and L-Lysine with formula $[\text{Ni}(\text{L})_2]+2$ have been synthesized and characterized on the basis of elemental chemical analysis, infrared spectra, UV-Visible and cyclic voltametry measurements. The IR spectra indicated the presence of amino acid coordinated through nitrogen atom and the oxygen from the carboxylic group. The experimental data suggest that the ligands act as bidentate and adopt an octahedral stereochemistry.

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