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Synthesis And Characterization Of Hetero Binuclear Complex Consisting Ni (II) And Cs(I) From Caesium Salt Of Acetyl Salicylic Acid (ASPIRIN) And Nickel (II) Schiff Base N,N'–1,2–Ethylene Bis(Ortho-Hydroxyacetophenoniminato) Nickel (II) As Ligand

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Abstract: In order to synthesize hetero-binuclear complex consisting Ni (II) and Cs(I), dibasic Ni (II) Schiff base N,N'–1,2–ethylenebis(ortho-hydroxyaceto phenoniminato) nickel(II) [NiEohacp] has been prepared from Nickel acetate and Schiff base (Eohacp) derived from Ethylene diamine and ortho-hydroxy acetophenone. This Ni (II) Schiff base [NiEohacp] has been used as ligand for complexation with Caesium salt of Acetyl salicylic acid (ASPIRIN) resulting in synthesis of the novel hetero binuclear complex [NiEohacpCsAp]. This synthesized hetero binuclear complex has been obtained in pure dark goldenrod crystalline solid. The structure elucidation of this synthesized hetero binuclear complex has been carried out using various physico-chemical techniques viz. Solubility, Elemental analysis, Magnetic property, Molar conductance measurement, UV-Visible and FT-IR spectroscopy. From the analysis of the data obtained the formula of the synthesized complex [NiEohacpCsAp] has been proposed as $[M_aL\,M_bL^{'}].3H_2O$ Where $M_a = Ni^{2+}, M_b = Cs^{+}$, L = N,N'-1,2-ethylenebis(ortho-hydroxyacetophenoniminato) dianion and L' = Acetyl salicylate with Square planar geometry about Ni^{2+} in the adduct.

Key Words: Hetero-binuclear complex, ortho-hydroxy acetophenone, Schiff base, Nickel, Caesium, N,N'–1,2–ethylenebis(ortho-hydroxyacetophenoniminato) nickel(II), Acetyl salicylic acid, Square planar geometry Synthesis, Characterization.

1. INTRODUCTION:

In recent years a number of binuclear metal complexes have been synthesized and reported in which Schiff bases of diprimary amine and carbonyl compound have been given much importance [1-3]. Deprotonated salen type Schiff bases act as tetradentate N_2O_2 type ligand and form complexes with transition metals. It is well investigated that donor oxygen atoms of these N_2O_2 type ligands have ability to co-ordinate with two metal ions [4-6]. Due to this feature, complexes containing N_2O_2 type ligand structure have ability to hold two metal centres in close proximity – one of transition metal and other of alkali or alkaline earth metal [7-12]. Synthesis of such hetero-binuclear complexes has been of great attraction for the researchers because of their biological importance [13-18]. Very little research work has been reported for hetero-binuclear complex having one of the metal centres as alkali metal [5,12,19,20]. In the present paper, synthesis of a novel hetero-binuclear complex containing Ni (II) and Cs (I) as metal centres has been reported. The present complex compound has been developed from Caesium salt of Acetyl salicylic acid using N,N'–1,2–ethylene bis(ortho-hydroxyacetophenoniminato) nickel(II) as Ligand. The structure of this synthesized complex has been elucidated using physico-chemical techniques such as Elemental analysis, Solubility, Magnetic moment measurement, Molar conductance measurement, FT-IR and Electronic absorption spectra. Experimental data reveal geometry of this synthesized hetero-binuclear complex as Square planar about Ni^{2+}metal centre.

2. MATERIALS AND METHODS

Chemicals used in the synthesis of metal Schiff base and hetero binuclear complex were of A.R. Grade. ESICO International Auto Melting Point Apparatus 1934 was used for the determination of melting point of the synthesized complex. Elemental Analyser EUROVECTOR EA3000 was used to estimate Carbon, Hydrogen, Nitrogen and Oxygen. Caesium and Nickel were estimated by ICP-MS of Agilent Technologies model no.7800. Magnetic moment of the synthesized complex was measured by the Vibrating Sample Magnetometer (VSM). Electronic absorption spectrum of 10^{-4}M solution of the complex was recorded between 200 -900 nm by Shimadzu UV-Visible Spectrometer Model UV-2450 using UV Probe 2.32 software. FT-IR Spectrum of synthesized complex was recorded on Thermo Nicolet Avatar 370 in the range 4000–400 Cm^{-1} by KBr beam splitter.

2.1 Synthesis of Schiff base N,N'-ethylenebis(orthohydroxy acetophenonimine) (Eohacp) derived from ethylenediamine and ortho-hydroxyacetophenone

0.3mole liquid o-hydroxyacetophenone was added to 50ml ethanol. In this solution 0.15 mole ethylenediamine was added drop by drop with continuous stirring. At once deep yellow coloured solid Schiff base appeared. The precipitate was filtered, washed with little ethanol and dried. The Schiff base so obtained, was recrystallised using ethanol as solvent and finally preserved in a desiccator over anhydrous $CaCl_2$.

2.2 Synthesis of N,N'-1,2-ethylenebis(ortho-hydroxyacetophenoniminato) nickel(II) complex, [NiEohacp]

In order to prepare Nickel (II) Schiff base complex, N,N'-1,2-bis(ortho-hydroxyacetophenonidene) ethylenediamine and nickel acetate tetrahydrate were allowed to react in 1:1 molar ratio. For this, 15.0g of N,N'-1,2-bis(ortho-hydroxyacetophenonidene) ethylenediamine (0.05mole) was dissolved in minimum amount of ethanol. 12.458g nickel acetate tetrahydrate (0.05mole) was dissolved in 20ml ethanol. In this solution Schiff base solution was added slowly with constant stirring. The resulting solution was warmed below 40°C for 20-25 minutes. Bright yellow coloured crystalline solid was precipitated out. Precipitate was filtered, washed with little ethanol, dried and kept in desiccator over anhydrous $CaCl_2$.

2.3 Synthesis of hetero binuclear complex [NiEohacpCsAp] from Caesium salt of acetyl salicylic acid (ASPIRIN) and Nickel(II) Schiff base N,N'–1,2–ethylenebis(ortho-hydroxyacetophenoniminato) nickel(II) [NiEohacp]

3.739g N,N'-1,2-ethylenebis(ortho-hydroxyacetophenoniminato) nickel(II) (0.01mole) was dissolved in minimum amount of ethanol. 3.122g (0.01mole) Caesium acetyl salicylate was dissolved in 20ml ethanol. The two solutions were mixed together and refluxed for 25 minutes at about 50°C and cooled to ice temperature when dark goldenrod complex was separated out. Precipitate was filtered, washed with little ethanol and dried. Finally, the synthesized complex [NiEohacpCsAp] was kept in a desiccator over anhydrous $CaCl_2$.

3. RESULTS AND DISCUSSION:

3.1 Physical Properties

The synthesized hetero binuclear complex is dark goldenrod coloured crystalline solid. The synthesized complex is found to be non-hygroscopic. The yield of the synthesized hetero binuclear complex is good. The melting point of the synthesized complex is 249℃. The synthesized complex [NiEohacpCsAp] is soluble in acetone, DMF, and DMSO but insoluble in water, methanol, ethanol, hexane and benzene. The synthesized complex is stable below 250℃. Magnetic moment of the synthesized complex is measured and found $\mu_{eff} = 0$ at 301 K.
This suggests diamagnetic magnetic property of the synthesized complex.

3.2 Elemental Analysis

Result obtained due to analytical estimation is:
C = 44.89%, H = 4.32%, N = 3.92%, O = 20.43%, Ni = 8.35%, Cs = 18.09%
Empirical Formula of the synthesized hetero binuclear complex is determined on the basis of analytical estimation data and is found to be $\boldsymbol{NiCsC_{27}H_{31}N_2O_9}$.

3.3 Molar Conductance Measurement

Molar conductance of 10^{-3}M solution of the synthesized complex in DMF is measured at $20(\pm 0.5)$℃ and is found to be 10.3 $S.Cm^2.mole^{-1}$. The low value of molar conductivity of the synthesized hetero binuclear complex [NiEohacpCsAp] indicates its non-electrolytic nature.

3.4 FT-IR Spectral Analysis

The prominent I.R. absorption band positions in FT-IR Spectrum of synthesized complex [NiEohacpCsAp] are presented in TABLE – 1. FT-IR spectrum analysis reveals a very strong band at 1595Cm^{-1} which may be assigned for $\upsilon_{C=N}$ stretching [21]. Many workers in various Schiff bases have reported $\upsilon_{C=N}$ stretching band for uncoordinated azomethine (C=N) in the range 1650 – 1690 Cm^{-1} [22]. The $\upsilon_{C=N}$ stretching band in [NiEohacp] FT-IR spectrum appears at 1600 Cm^{-1}. The shift of the absorption band of $\upsilon_{C=N}$ stretching towards lower wave number indicates

coordination of the imine nitrogen to the Ni^{2+}ion during [NiEohacp] complex formation [22, 23]. This is further substantiated by the presence of new bands in the region 566 – 654 Cm^{-1} assignable to $\upsilon_{N=M}$ stretching vibrations. Sharp, medium to weak bands in the range 500 – 700 Cm^{-1} have been reported for nitrogen-metal (υ_{N-M}) stretching vibrations [24]. The absence of sharp characteristic absorption band in the region 3600 – 3700 Cm^{-1} due to phenolic O – H stretching vibrations and also the absence of broad band in the region 3200 – 3300 Cm^{-1} due to intermolecular hydrogen bond, indicates deprotonation during formation of nickel complex of Schiff base [NiEohacp]. A sharp IR absorption band appears at 1292 Cm^{-1} which may be assigned for phenolic C - O stretching vibrations. However, phenolic C – O stretching vibrations absorption band in complex [NiEohacp] appears at 1244 Cm^{-1}. A strong absorption band in region 1200 – 1350 Cm^{-1} has been reported for phenolic C – O stretching mode by research workers [25,26-31]. Shifting of phenolic C – O stretching vibration towards higher wave number indicates coordination of phenolic oxygen atom to Cs^{+} ion during hetero-binuclear complex [NiEohacpCsAp] formation from ligand [NiEohacp]. This fact is further substantiated by the presence of oxygen – metal bond stretching vibration (υ_{O-M}) bands in the region 444 – 524 Cm^{-1}.

TABLE 1: Prominent I.R. absorption band positions in FT-IR Spectrum of synthesized complex [NiEohacpCsAp]

[NiEohacpCsAp]	
Absorption Band Position (in cm^{-1})	Assignment
3454 br	Water of crystallisation
3046 br & w,2793 br & vw	ν(Ar – H) Str.
1659 vs	ν(C = O) Str. ester
1595 vs	ν(C = C) Str. aromatic, ν(C = O) Str. carboxylic, ν(C = N) Str.
1534 s	ν(C = C) Str. aromatic
1484 s	ν(C = C) Str. aromatic
1443 s	ν(C – O) Str. carboxylic, ν(C-H) Bending aromatic
1385 m	ν(C – H) def. in CH_3, ν(C – N) Str.
1336 s	ν(C – O) Str. ester
1292 s	ν(C – O) Str. phenolic
1240 s	ν(C – H) skeletal vib., ν(C – CH_3) rocking vibration
1215 s	ν(C – CH_3) rocking vibration
1167 s	ν(C – N) Bending
1143 w, 1115 w, 1026 w	In plane bending of the ring C – H bonds
872 m, 849 m, 810 m	Out – of – plane bending of the ring C – H bonds
746 s	ν(C – C) def. ortho-disubstituted benzene
705 m	Out of the plane ring bending of substituted benzene
654 m, 592w, 566w	ν(N – M) Str.
524 m, 494 w, 444 w	ν(O – M) Str.

Br = Broad, s = Strong, vs = Very strong, m = Medium, w = Weak

The prominent I.R. absorption band positions of certain chemical bonds of interest in FT-IR Spectrum of Aspirin, Nickel (II) Schiff base [NiEohacp] and synthesized hetero-binuclear complex [NiEohacpCsAp] have been identified and summarised in TABLE – 2.

TABLE 2: I.R. absorption band positions in FT-IR Spectrum

Chemical Bond	Mode of Vibration	Absorption Band Position (in cm^{-1})		
		Aspirin	[NiEohacp]	[NiEohacpCsAp]
O – H Carboxylic	Stretching	2649br, 2821 br	NA	NP
C – O Carboxylic	Stretching	1313 s	NA	1443 s
C = O Carboxylic	Stretching	1591 s	NA	1595 s
C = O Ester	Stretching	1684 s	NA	1659 s

C – O Ester	Stretching	1265 s	NA	1336 s
C – O Phenolic	Stretching	NA	1244 s	1292 s

Where NA = Not Applicable, NP = Not Present, br = broad, s = strong

Absence of absorption band of carboxylic O – H bond (Str) in [NiEohacpCsAp] indicates deprotonation of carboxylic O – H bond during Caesium salt formation, and shift of absorption band of carboxylic C – O (Str) towards higher wave number in [NiEohacpCsAp] as compared to aspirin further substantiates the fact of deprotonation of carboxylic O – H and conversion of it into O – Cs. Comparison of absorption band position of carboxylic C = O (Str) in FT-IR spectra of Aspirin and [NiEohacpCsAp], we find no major shift in wavenumber which indicates non-involvement of oxygen atom lone pair of carboxylic C = O in coordination during complex [NiEohacpCsAp] formation. However, slight shift of absorption band of ester C = O (Str) towards lower wave number from aspirin to [NiEohacpCsAp] indicates presence of hydrogen bonding between ester C = O group and H_2O (water of crystallisation). On comparison of FT-IR spectrum of Aspirin and [NiEohacpCsAp], we find major shift in absorption band of ester C – O (Str) towards higher wave number which indicates coordination of oxygen atom of ester C – O with Caesium during complex [NiEohacpCsAp] formation. This fact is supported by the presence of oxygen-metal bond stretching vibration (υ_{O-M}) bands in the region 444 – 524Cm^{-1}. A broad band at 3454Cm^{-1}indicates presence of H_2O as water of crystallisation in [NiEohacpCsAp] molecule [28, 32].

3.5 Electronic Absorption Spectrum Analysis

Band positions in electronic absorption spectrum of the complex [NiEohacpCsAp] has been summarised in TABLE – 3.

TABLE 3: Band positions in electronic absorption spectrum of the complex [NiEohacpCsAp]

[NiEohacpCsAp]	
Absorption Band Position (in cm^{-1})	Assignment
Broad band ranging 267 – 468 nm with a peak at λ_{max} = 415 nm and a shoulder at 354 nm	(1) Intraligand transitions: (a) Benzene Chromophore $\Pi \rightarrow \Pi^*$ transition (b) Azomethine Chromophore $\Pi \rightarrow \Pi^*$ transition (c) Co-ordinated azomethine $n \rightarrow \Pi^*$ transition (2) Ligand to Metal Charge Transfer (LMCT) (3) d → d transition
Band with a peak at λ_{max} = 557 nm	d → d transition

Appearance of no bands between 500 – 1000 nm excludes possibility of square pyramidal and octahedral geometry around Ni^{2+} ion. Further appearance of no band above 1000 nm excludes possibility of tetrahedral geometry around Ni^{2+} ion. For a square planar geometry around Ni^{2+} ion there must be appearance of two bands in region 420 – 500 nm & 500 – 600 nm assignable to $^1A_{1g} \rightarrow {}^1A_{2g}$ and $^1A_{1g} \rightarrow {}^1B_{1g}$ transitions, respectively. In this case a band appears at 557nm corresponding to $^1A_{1g} \rightarrow {}^1B_{1g}$ d – d transition and another band of $^1A_{1g} \rightarrow {}^1A_{2g}$ d-d transition has been obscured by the CT band at that region [21,33]. Hence, in the complex [NiEohacpCsAp] there is square planar geometry around Ni^{2+} ion. This is further supported by zero magnetic moment of complex [NiEohacpCsAp] which indicates its diamagnetic nature with no unpaired electron, and hence a low spin complex.

4. PROPOSED STRUCTURE:

Investigation results reveal coordination of azomethine nitrogen to Ni^{2+} ion, deprotonation of phenolic O – H of Schiff base, Eohacp and co-ordination of its oxygen to Ni^{2+} during nickel Schiff base, [NiEohacp] formation. This suggests Schiff base as tetradentate ligand acting as N_2O_2 type. Further, investigation results confirm deprotonation of carboxylic O – H of acetyl salicylic acid with formation of O – Cs bond. Again, it indicates coordination of oxygen of ester C – O of Caesium acetyl salicylate to Cs atom of O – Cs during complex [NiEohacpCsAp] formation. There is evidence of coordination of oxygens of phenolic C–O of Nickel (II) Schiff base N, N'-1,2-ethylenebis(ortho-

hydroxyacetophenoniminato) nickel (II) with Cs. This suggests Ni (II) Schiff base acting as bidentate ligand during formation of hetero binuclear complex [NiEohacpCsAp]. There is no evidence of coordinated water molecules. However, analysis of structure developed by these investigation results and empirical formula, suggests three water of crystallisation molecules present in the synthesized complex molecule. Thus, the synthesized complex is suggested as [NiEohacpCsAp].$3H_2O$. The investigation result obtained from electronic absorption spectrum analysis coupled with magnetic moment measurement, suggests Square planar geometry about Ni^{2+} ion in the adduct [NiEohacpCsAp].

5. CONCLUSION:

From the above investigation results and discussion, it may be summarised that nickel Schiff base N,N'-1,2-ethylenebis(ortho-hydroxyacetophenoniminato) nickel(II) acts as "bidentate ligand" in the adduct [NiEohacpCsAp].$3H_2O$. There is no change in geometry about Ni^{2+} ion during adduct formation from nickel Schiff base complex [NiEohacp], and it remains square planar with coordination number 4. The adduct is non-electrolytic and diamagnetic in nature. The suggested formula for the synthesized complex is [Ma L M_b L']$.3H_2O$ where $M_a = Ni^{2+}$, $M_b = Cs^+$, L = N,N'-1,2-ethylenebis(ortho-hydroxyacetophenonimin ato) dianion and L' = Acetyl salicylate. The suggested structure of the synthesized hetero binuclear complex is shown in FIGURE 1.

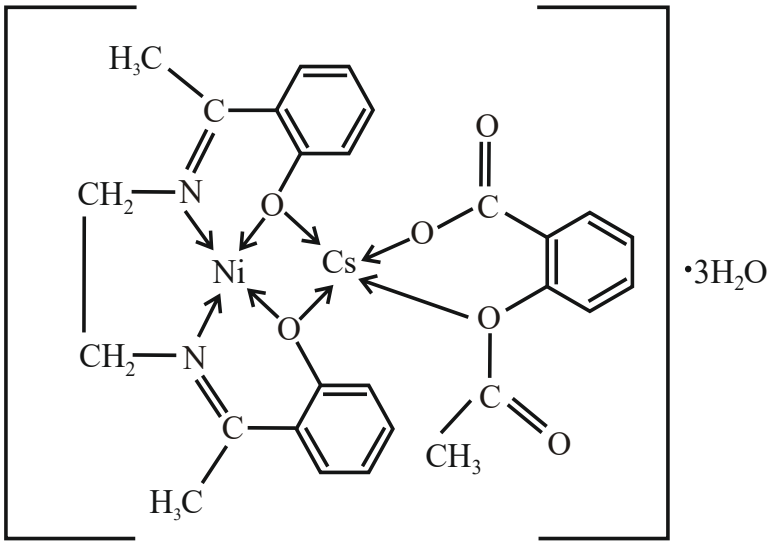


FIGURE 1: Proposed Structure of Synthesized Hetero-binuclear Complex

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7. CONFLICT OF INTEREST

The author declares that there is no conflict of interest.

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