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Synthesis and Characterisation of Chelating Tris Ethylenediamine Iron Complex

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ABSTRACT

Polydentate ligand whose structure permit the attachment of their two or more donor atoms to the same metal ion simultaneously and thus produce one or more rings are called chelate. In the present work synthesis of trisethylenediamine iron complex and study about their applications. The synthesized complex was characterised by EDAX, UV-Visible, FTIR, TGA &DTA. Application of the complex was studied by its Detoxifying activity.

KEY WORDS:Chelate; Poly dentate; Ligand; De-toxicity, Heavy Metal

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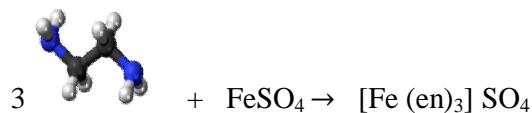
1. INTRODUCTION

For the last two decades, the synthesis of coordination compound with transition metals and their applications have become an attractive field because of the excellent properties such as catalysis, ion exchange, microelectronics, nonlinear optics, porous materials, chelation etc.¹⁻⁷. ring complexes are commonly called as chelating complexes. Many of the chelating complexes are acting as drugs for specific poisoning.⁹ Chelating metal complexes are of significant interest not only their pharmacological properties as medicinal, antibacterial, antifungal and anticancer agents but also their detoxifying property. Bioinorganic Chemistry is acting as a bridge between inorganic chemistry and biochemistry. Polydentate ligands can easily bind with metal to form stable ring complexes. These ring complexes are commonly called as chelating complexes. Many of the chelating complexes are acting as drugs for specific poisoning.⁹

2. EXPERIMENTAL

2.1. Preparation of metal complexes

The complex $[\text{Fe}(\text{en})_3]^{2+}$ was prepared from ferrous sulphate and ethylenediamine. 2 mM aqueous solution of ferrous sulphate was stirred in a beaker and 6 mM of ethylenediamine was added drop by drop. With order to get proper mixing continuous string, 2 ml of ethyl alcohol was added for complete precipitation. Then transferred in a petri dish to remove solvent in incubator at 45°C. After three days a dark brown coloured complex.⁹



Ethylenediamine

2.2. T2.2.1. Energy Dispersive X-ray Spectroscopy:

It provides the elemental curve as output. EDX primarily detects the X-rays emitted from the sample during the process of bombardment by an electron beam for characterizing the elemental composition of the sample. The spectra was recorded with line type K & M-Series at Sophisticated Analytical Instruments Facility STIC, Kochi.

2.2.2. UV-Visible spectrometer

UV-Visible spectra of ligand and synthesised complex was obtained using Perkins Elmer Lambda -25. UV-Visible spectrometer in the range of 200-700nm. The spectra were recorded at Sophisticated Analytical Instruments Facility STIC, Kochi.

2.2.3. FT-IR Spectrometer

IR spectral data is used to determine the structure of the complex. In the present work the FTIR spectra of free Ethylenediamine and synthesised complex was recorded using KBr pellet technique with frequencies in the range of 500 unit to 4000 Cm^{-1} . The spectra were recorded at Sophisticated Analytical Instruments Facility STIC, Kochi.

2.2.4. Thermal Methods

Thermal studies of the complex was recorded using Perkin Elmer Thermal Analyser with heating range is 40° to 73° at 20°/min. Thermogram was recorded at Sophisticated Analytical Instruments Facility STIC, Kochi.

3. RESULTS AND DISCUSSION

3.1. EDAX – Spectroscopy:

Table.1

| Molecular Formula | Element Weight% from EDAX | | | | |
|--|---------------------------|----|----|---|----|
| | C | N | O | S | Fe |
| $\text{FeC}_6\text{N}_6\text{H}_{24}\text{SO}_4$ | 26 | 24 | 26 | 7 | 16 |

From the data of (table.1) EDAX, the formation of complex was confirmed. The elements present in the complex were C, N, O, S, Fe (II). It has been exhibited their characteristics peaks.

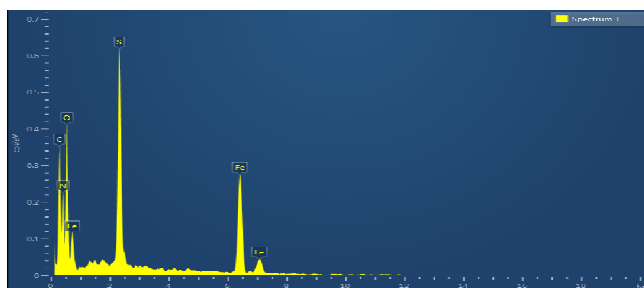


Fig.(1) EDAX of $[\text{Fe}(\text{en})_3]^{2+}$ complex

3.2. UV-Visible absorption spectra

The formation of the chelating metal complex was confirmed by UV-Visible spectra. If the complex having π -electrons or non-bonding electrons can absorb energy ultraviolet or visible light. From the data of UV-Visible spectrum, $[\text{Fe}(\text{en})_3]^{3+}$ complex was exhibited a maximum absorption at

336 nm and a broad band around 500 nm. Iron complex belongs to d^6 system, that has four non bonded electrons, for that exhibited a sharp absorption band corresponds to d-d transition. The broad absorption band corresponds to CTT from ligand to metal.¹¹

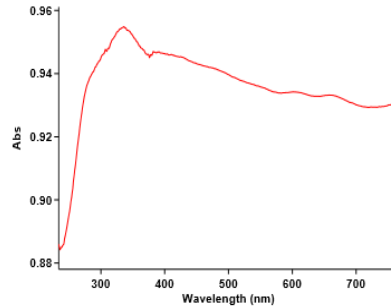


Fig.(2) $[\text{Fe}(\text{en})_3]^{2+}$ complex

3.3.FT-IR spectra of chelate complex

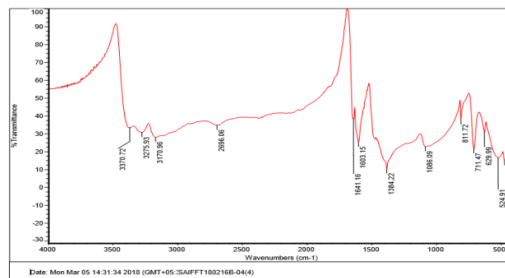


Fig. (3.1) FT-IR Spectrum of Ethylenediamine (Ligand)

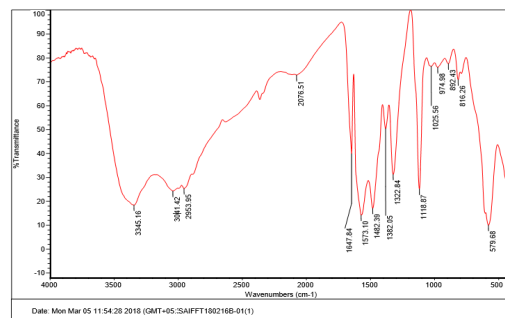


Fig.(3.2) FT-IR Spectrum of $[\text{Fe}(\text{en})_3]$

FT-IR spectral data

Table.2

| Free Ligand Complex | N-H Stretching (Cm^{-1}) | C - H Stretching (Cm^{-1}) | N - H Bending (Cm^{-1}) |
|---------------------------------------|-------------------------------------|---------------------------------------|------------------------------------|
| En $[\text{Fe}(\text{en})_3]^{2+}$ | 3370 3345 | 3170 3042 | 1641 1573 |

From the data of table.2, free ethylenediamine ligand has N – H stretching frequency at 3370 cm^{-1} . C – H stretching frequency at 3170 cm^{-1} N – H bending vibration at 1641 cm^{-1} with highly sharp band C- N stretching vibration 1384 cm^{-1} .

$[\text{Fe}(\text{en})_3]^{2+}$ complex exhibited their frequencies in the IR region with sharp bands. N – H stretching frequency at 3345 cm^{-1} , C - H stretching frequency at 3041 cm^{-1} , C – N exhibit their absorption at 1322 cm^{-1} . The characteristic N – H bending vibration was observed as a strong band around 1584 cm^{-1} . It should be commonly observed fact for chelated ethylenediamine complex. On the basis of that N – H bending vibration bonded with metal at 1573 cm^{-1} with a sharp band.⁹ The absorption frequencies observed in free ligand was greater than the complex frequencies. It was because of, when a ligand Co-ordinated to a metal, the electron could be flow from ligand to metal. So that the stretching frequency around N liked N – H, C – N and C – H were decreased.

3.4. Thermal analysis

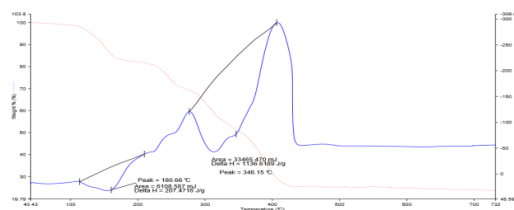
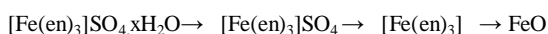


Fig.(4) TGA&DTA of $[\text{Fe}(\text{en})_3]^{2+}$

Thermal methods mainly used to determine the stability of the complex and intermediate. The $[\text{Fe}(\text{en})_3]^{2+}$ was stable up to 100°C . At 100°C water molecules began to evolve, at 160°C anhydrous metal complex was obtained. At 346.15°C the volatile sulphate and amine groups might be liberated. Because of that the peak area was very greater.¹²

100°C 160°C 346°C



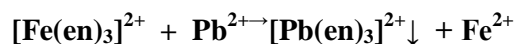
3.5. DETOXIFYING ABILITY

The synthesised chelating metal complex with its detoxifying ability was determined by qualitative and quantitative methods. The heavy metal such as lead mixed with complex; suddenly they produced a new compound. 0.01M solution of $[\text{Fe}(\text{en})_3]^{2+}$ complex, along with 0.01M solution of $\text{Pb}(\text{NO}_3)_2$ was made up to the mark of 100 ml SMF. During the preparation of the solution, product was obtained. Then the solution was filtered by using whatmann Number: 1 filter paper. The

product was taken for qualitative analysis and the filtrate was taken for quantitative analysis. The product might be lead ethylene diamine complex.

3.5.1. Qualitative analysis of lead

The precipitate is treated with KI. If they produce yellow colour, that indicate the presence of lead. Then add a small amount of water and heat the solution, after some time a golden spangle is appeared. It should be confirm the presence of lead.



The precipitate was qualitatively analysed. It was exhibited yellow colour with KI. It was confirmed the presence of lead in the precipitate. The percentage of lead in the precipitate was confirmed by EDAX.

3.5.2. EDAX – SPECTROSCOPY



Fig.(5). EDAX of Pb in $[\text{Fe}(\text{en})_3]^{2+}$ complex

Table.3

| Complexes | Elements | Weight % | Atomic % |
|---------------------------------------|----------|----------|----------|
| Pb in $[\text{Fe}(\text{en})_3]^{2+}$ | C | 5.76 | 22.73 |
| | N | 0.59 | 1.99 |
| | O | 18.65 | 55.23 |
| | Fe | 4.67 | 4.01 |
| | Pb | 70.12 | 16.03 |

Detoxifying ability was confirmed by qualitative analysis and EDAX.

4. CONCLUSION

In the present work $[\text{Fe}(\text{en})_3]^{2+}$ chelating complex was synthesised. The synthesised complex was characterised by EDAX, UV-visible, FT-IR & Thermal techniques. The spectral data showed that magnetism, possible electronic transition, geometry, co-ordination of metal with ligand, thermal stability and its decomposition. An application of synthesised complex was studied by detoxifying ability with heavy metal lead.

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