M. Jaya Brabha et al., IJSRR 2019, 8(1), 1092-1098

Research article **Available online www.ijsrr.org** **ISSN: 2279–0543**

International Journal of Scientific Research and Reviews

Synthesis and Characterisation of Chelating Tris Ethylene Diamine Copper Complex

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ABSTRACT

Chelates are used for the elimination of harmful radioactive, heavy metal toxicity from the body. Some of the chelating agents such as EDTA, Ethylenediamine and CDTA are used in the elimination of harmful radioactive metals from the body. Chelates used in food preservation. Supplementation of essential trace elements is an area of increasing interest in the field of human and veterinary pharmacology. In the present work synthesis of complex of Cu(II) metalwith ligand ethylenediamine and study about their application. The synthesized complex was characterised by EDAX, UV-Visible, FTIR, and TGA& DTA. Application of the complex was studied by its Detoxifying ability with heavy metal lead.

KEYWORDS: Chelate; Poly dentate; Ligand; De-toxicity, Heavy metal.

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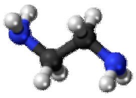
1. INTRODUCTION

The polydentate ligands may be attached to the central metal ion through two kinds of functional group namely acidic and co-ordinating groups to form covalent and co-ordinate ligands respectively. The classification of chelates follows the number and kind of linkages by which the metal ion is attached with the ligands. The covalent bonds are formed by the replacement of one or more hydrogen atoms from the acidic groups present in the ligand by the metal atom. Co-ordinating ligands, without the replacement of hydrogen, are formed by the donation of an electron pair from the ligands. The examples of most common groups of this type are $–NH_2$ (primary, secondary and tertiary amines). Chelate rings are most stable, because of reduced strain, when they have five or six members including the metal ion. The enhanced stability of complexes containing chelated ligands is known as the chelate effect.Chelating metal complexes are of significant interest not only their pharmacological properties as medicinal, antibacterial, antifungal and anticancer agents but also their detoxifying property. Many of the chelating complexes are acting as drugs for specific poisoning.[1-6]

2. EXPERIMENTAL

2.1.Preparation of metal complexes

The complex $[Cu(en)_3]^{2+}$was prepared from copper sulphate and ethylenediamine. 2 mM aqueous solution of metal salt is stirred in a beaker and 6 mM of ethylenediamine was added drop bydrop. With order to get proper mixing continuous strring, 2 ml of ethyl alcohol was added for complete precipitation. Then transferred in a petri dish to remove solvent in hot air oven at 45˚C. After three days a blue coloured complex. [6]

3  + $CuSO_4 \rightarrow [Cu(en)_3]SO_4$

Ethylene diamine

2.2. Techniques used

2.2.1. 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.

2.2.2. FT-IRspectrometer

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.

2.2.3. Thermal Methods

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

2.2.4. 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 were recorded with line type K & M-Series 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**	**Metal Cu**
$CuC_6N_6H_{24}SO_4$	29	20	28	9	12

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

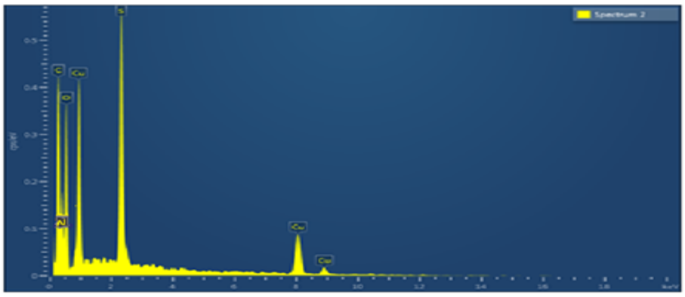


Fig.(1) EDAX of $[Cu(en)_3]^{2+}$ complex

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 (n-electrons) can absorb energy in the form of ultraviolet or visible light..

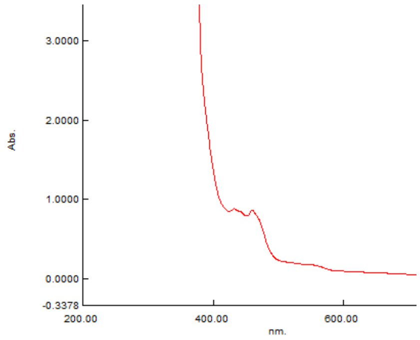
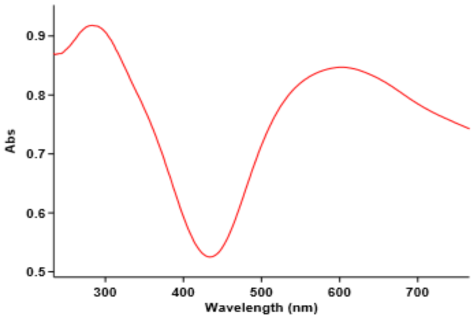


Fig.(2.1) Ligand



Fig(2.2) $[Cu(en)_3]^{2+}$ complex

The ligand ethylene diamine absorbs UV light at 400 nm with a high intensity of n→σ* transition. $[Cu(en)_3]^{2+}$ complex was exhibited a maximum absorption at 300nm,which was assigned to the intramolecular charge transfer transition from the ligand to metal. The band around 600 nm could be attributed to d→d transitions, which corresponds to octahedral geometry. The broadness of band was due toligand field and Jahn-Teller distortion and typical d^9 system. Though three transitions are expected, they are very close in energy and often appear in the form of one broad band envelop.[7].

3.3.FT-IR spectra of chelate complex

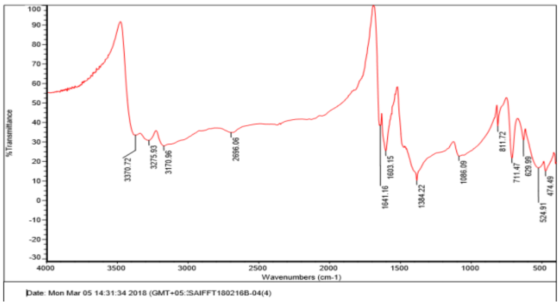


Fig. (3.1) (Ligand)

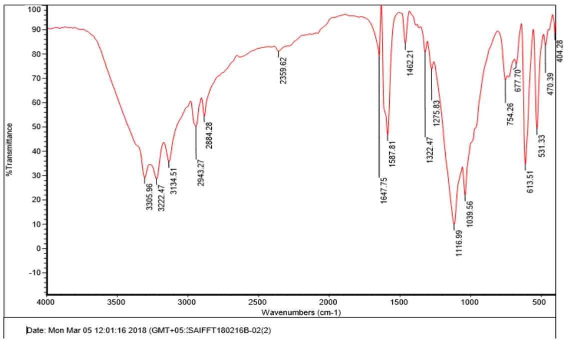


Fig.(3.2) FT-IR Spectrum of $[Cu(en)_3]^{2+}$

FT-IR spectral data

Table.2

Ligand & Complexs	N-H Stretching (Cm^{-1})	C – H Stretching (Cm^{-1})	C – N Stretching (Cm^{-1})
En	3370	3170	1384
$[Cu(en)_3]^{2+}$	3305	3134	1322

From the data of table.2, free ethylenediamine has exhibited N – H stretching frequency at 3370 cm^{-1}. C – H stretching frequency at 3170 cm^{-1} with highly sharp bandand C- N stretching vibration at 1384 Cm^{-1} [8]. [Cu (en)$_3$]$^{2+}$complex showed their own frequencies in the IR region. N – H stretching frequency at 3305 Cm^{-1} [8] C – H and C – N frequencies were exhibited at 3134 and 1322 Cm^{-1} respectively [9].All the absorptions in complex lowered than the corresponding absorption in the free ligand. It was nothing but the flow of electron from the ligand to metal.On the basis of the IR Spectral data ethylenediamine was surely bound with Cu^{2+}metal ions.

3.4. Thermal analysis

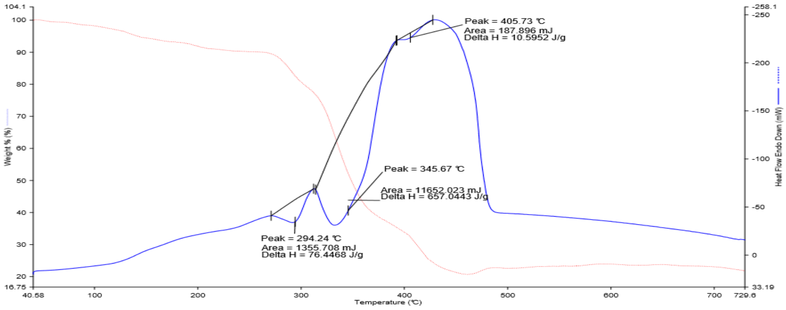


Fig.(4.1) TGA & DTA of $[Cu(en)_3]^{2+}$

Thermal methods mainly used to determine the stability of the complex and intermediate. The [Cu(en)$_3$]$^{2+}$ was stable up to 270°C. This temperature was revealed that, the complex should be anhydrous. At 294°C, 345°C, 405°Cthe volatile sulphate and amine groups might be liberated. At 405 □ the exothermic peak was observed, it was the formation of stable metal oxide. [10]

294□

$[Cu(NH_2CH_2CH_2NH_2)_3]SO_4.$ → $[Cu(NH_2CH_2CH_2NH_2)_3]$
345□ 405□
→ $[Cu(NH_2CH_2CH_2NH_2)]$ → CuO

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 $[Cu(en)_3]^{2+}$ complex , along with 0.01M solution of $Pb(NO_3)_2$ 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.

$$[Cu(en)_3]^{2+} + Pb^{2+} \rightarrow [Pb(en)_3]^{2+}\downarrow + Cu^{2+}$$

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*

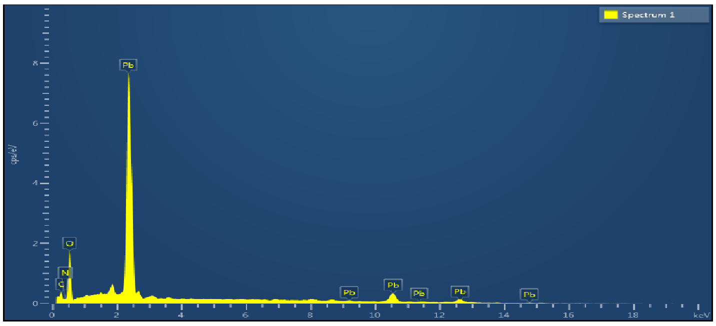


Fig.(6.5) EDAX of Pb in $[Cu(en)_3]^{2+}$ complex

Complexes	**Elements**	**Weight %**	**Atomic %**
Pb in $[Cu(en)_3]^{2+}$	C	5.28	19.4
	N	2	6.31
	O	21.4	59.12
	Pb	71.27	18.17

Detoxifying ability was confirmed by qualitative analysis and EDAX.

4. CONCLUSION

In the present work $[Cu(en)_3]^{2+}$ 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.

ACKNOWLEDGEMENT:

First I thank Almighty God for giving me a chance to do research. Then I express my sincere thanks to my beloved family members and co-others.

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