

Research Article**SYNTHESIS AND CHARACTERIZATION OF CNTS BY NATURAL PRODUCT****B.TIWARI¹, I.P.TRIPATHI², SANJAY SAXENA³ HARIBHUSHAN, ⁴A.P.DWIVEDI⁵, RANJANA DUBEY⁶**

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ABSTRACT

Nanotechnology is one of the most emerging technology these days.. Metal carbon nano tubes are having vast applications are used in every sphere of life very frequently. Due to vast applications of CNTs it is necessary to develop new methods to prepare CNTs. One of the new methods to prepare CNTs is chemical method . In this methods Complexes of transition metal Ni(II) with amino acids present in egg albumin have been synthesized. The complex so formed is analyzed by spectroscopic methods like IR, NMR Spectroscopy.

The amino acid metal Complex is decomposed at higher temperature using muffle furnace to obtain metal carbon nano tubes. These metal carbon nano tubes are characterized using scanning probe instruments like DLS and STM.

Keywords: Carbon nano tubes, Natural product, Ni(ii),Albumin, Amino acid, Amino acid-Metal complex, IR,NMR,DLS,STM

INTRODUCTION

Carbon nano tubes (CNTs) are allotropes of carbon with a nanostructure. The length-to-diameter ratio of CNTs is up to 28,000,000:1, which is significantly larger than any other material. As a matter of fact CNTs are molecular-scale tubes of graphitic carbon with outstanding properties. They are among the stiffest and strongest fibers known, and have remarkable electronic properties with many other unique characteristics.

The carbon nano tube structure has already made its appearance because it represents an entirely new form of matter. Single -Walled nano tubes can be either semiconductor or metallic. Nano tubes are also very stiff and very stable. They are built with their length exceeding thickness thousand of times.

Clusters of C-atoms in cylindrical forms (carbon nano tubes) have novel properties that make them useful in many applications in nanotechnology, electronics, optics and other fields of materials science, as well as potential uses in architectural fields.

Due to vast applications of CNTs , it is required to develop new methods and techniques to prepare carbon nano tubes and characterize them. It is also required to inculcate some desired properties in them, so that they can be used in various applications.^{1, 2,6}

MATERIALS AND METHODS

Two normal metal salt solution of Ni²⁺ was prepared by using AR grade chemicals. It is allowed to react with amino acids present in egg albumin to form a complex of amino acids with nickel ion. The metal-albumin complex, so formed was decomposed at higher temperature as reported in the literature.

Preparation of amino acid –metal complex

As we know that proteins are macromolecules comprising of amino acids as monomer. Amino acids are compounds containing –NH₂ and COOH. With the help of these groups amino acids form complexes with metals and different chains of amino acids are combined together. These compounds on decomposition give carbon -metal nano tubes.

When aqueous solution of Nickel Salt is allowed to react with amino acids present in egg albumin the lone pair present on nitrogen of –NH₂ and oxygen of COO⁻ of COOH group present in amino Acid form complex with Ni (ii). In this way nickel (ii) forms cross links between two amino acid chains.

The structure of albumin is very complex and it is very difficult to produce exact structure of amino acid-metal complex. Egg albumins contain a number of different chemical compounds, which form complex with metal to form complex. These metals -amino acid complexes are heated in muffle furnace till decomposition.

These complexes on decomposition give carbon nano tubes.^{3,4,5}

The complex of Amino Acid and metal is expected to contain following groups:

- (i) NH₂ group
- (ii) COOH group
- (iii)CH₂ group
- (iv) Amide group etc.

The compound when analyzed using IR(Infra red) and NMR(Nuclear Magnetic resonance) gave following results. From the results of NMR and IR it is clear that the compound contain all the above said groups.

Characterization

Spectral studies- Though it is very difficult to analyze the complexes of albumin and metal but certain important features can be identified which give valuable information about the structure.

IR spectra-IR Spectra of Nickel Amino Acid Complex is shown is shown fig1. The information, which we get by the IR spectra, is

Table 1: Typical infrared absorption bands for nickel amino acid complex

Functional group	Prominent absorption band (cm ⁻¹)
C-H (alkanes)	1376
Alcohol hydrogen bonded	3369
C-H	2925.78
Carboxylic acid (hydrogen bonded)	2854
Carboxylic acid	1744
Amide	1630
C-O carboxylic acid	1092 & 1167

Table 2: Characteristics proton chemical shift

Types of proton	Chemical shift δ (ppm)
(CH ₂) ₃ CH	1.45
CH ₂ —	4.4
—CH —	5.5
—CH—COOH	6 – 7.8

*Amide group is due to the peptide linkage (—C—NH) between different amino acid chains

NMR spectra: NMR Spectra of Nickel Amino Acid Complex is shown is shown fig.2 The information, which we get by the NMR spectra, is summed up in the table.

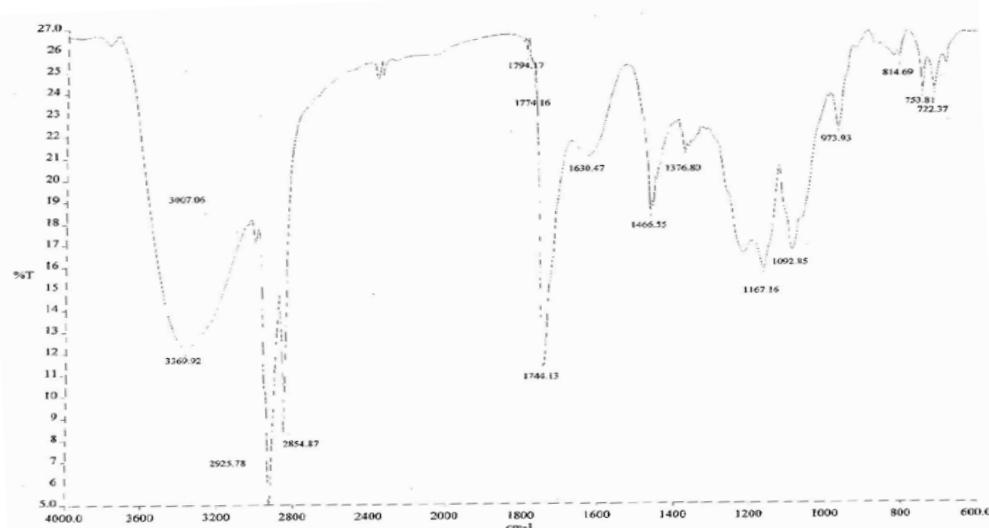


Fig. 1: IR Spectra of nickel amino acid complex summed up in the table given below

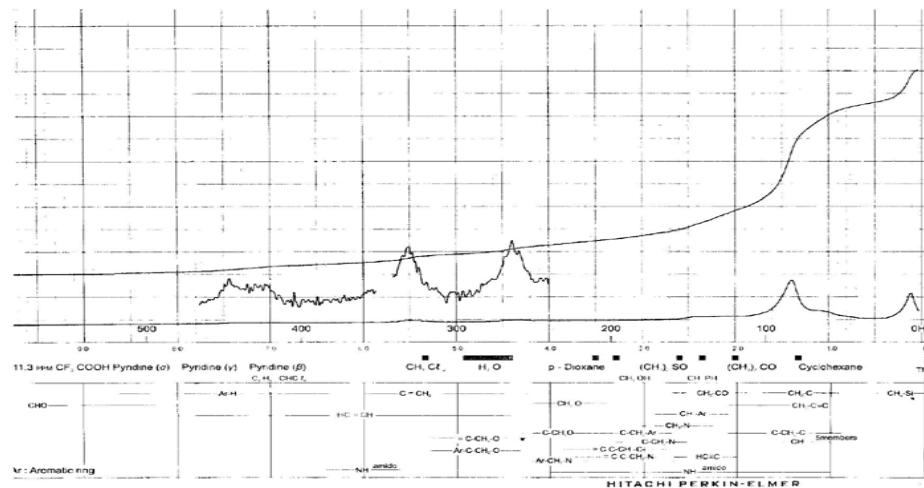


Fig. 2: NMR spectra of nickel amino acid complex

*Peak of hydrogen bonded alcohol is because of ethyl alcohol solvent

The chemical compounds formed are decomposed at different temperatures (800°C, 1000°C, 1200°C) in a muffle furnace. At 1200°C only soot is formed. The samples decomposed at 800°C and 1000°C are characterized by scanning probe instruments.

RESULTS AND DISCUSSION

Characterization by scanning probe instruments

Characterization by DLS: Samples were sent for DLS (Differential Liquid Scanning) analysis and following results are found which gave valuable information about the formation of CNTs. The results are:

Results of sample 800c_S3: In this sample the chemical compound is prepared by taking ethyl alcohol as solvent and is decomposed at 800°C. The results are shown in the fig.3 and are summed up in table 3.

Results of sample 1000c_S3: In this sample the chemical compound is prepared by taking ethyl alcohol as solvent and is decomposed at 1000°C.

The results are shown in the fig.4 and are summed up in table below (fig. 1, fig. 2 and table 3)

Interpretation

Nano material have diverged tunable physical properties as a function of there size and shape due to strong quantum confinement effect and large surface/ volume ratio.

The average intensity reports were reported 44.43% where as average CNTs posses a size distribution report of about 90.15 nm as diameter. Where as some peaks show 83.46 nanometer, 43.02 nanometer etc.

Metal CNTs has diameter of about 30 to 70 nm and length up to 50 micrometer. The CNTs are nearly mono dispersed and average width is 17.40 nm. Many research paper shows the absorption peaks of metal CNTs are from 70 to 130 nm as diameter. It shows one of the most challenging application in electronics, where both the metallic and semi conducting properties of the single wall nano tubes are exploited.

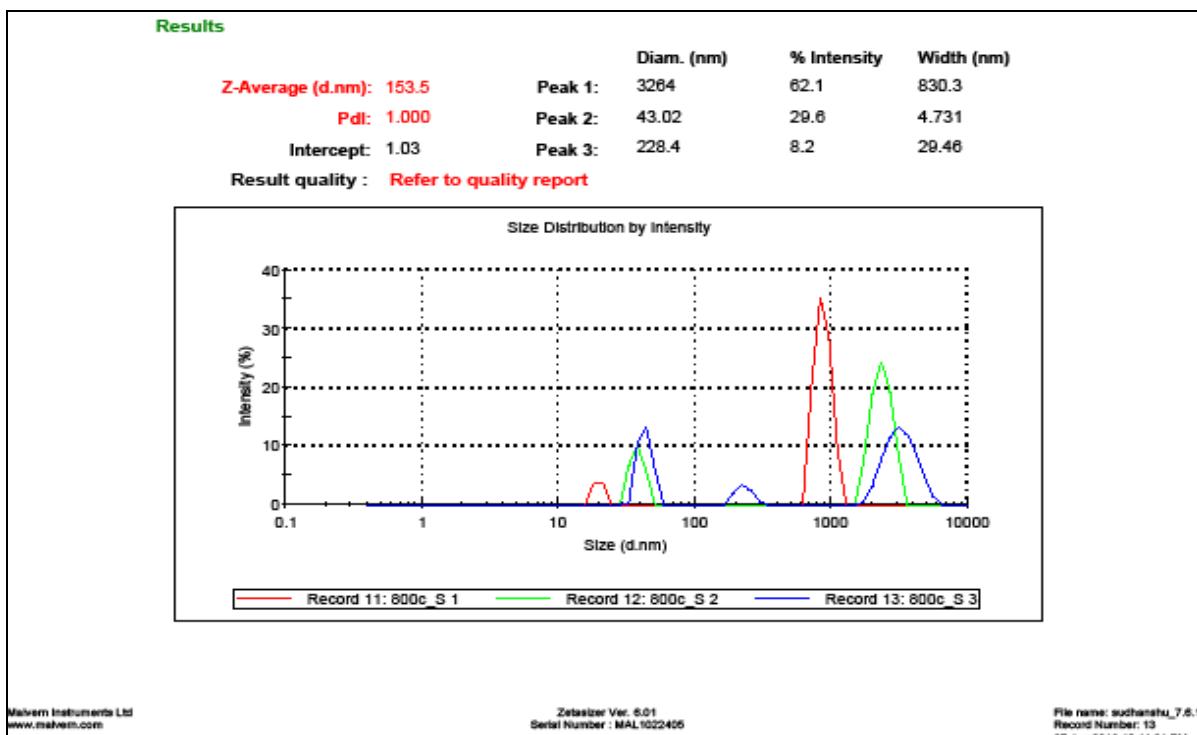


Fig. 3: DLS Results of sample decomposed at 800°C

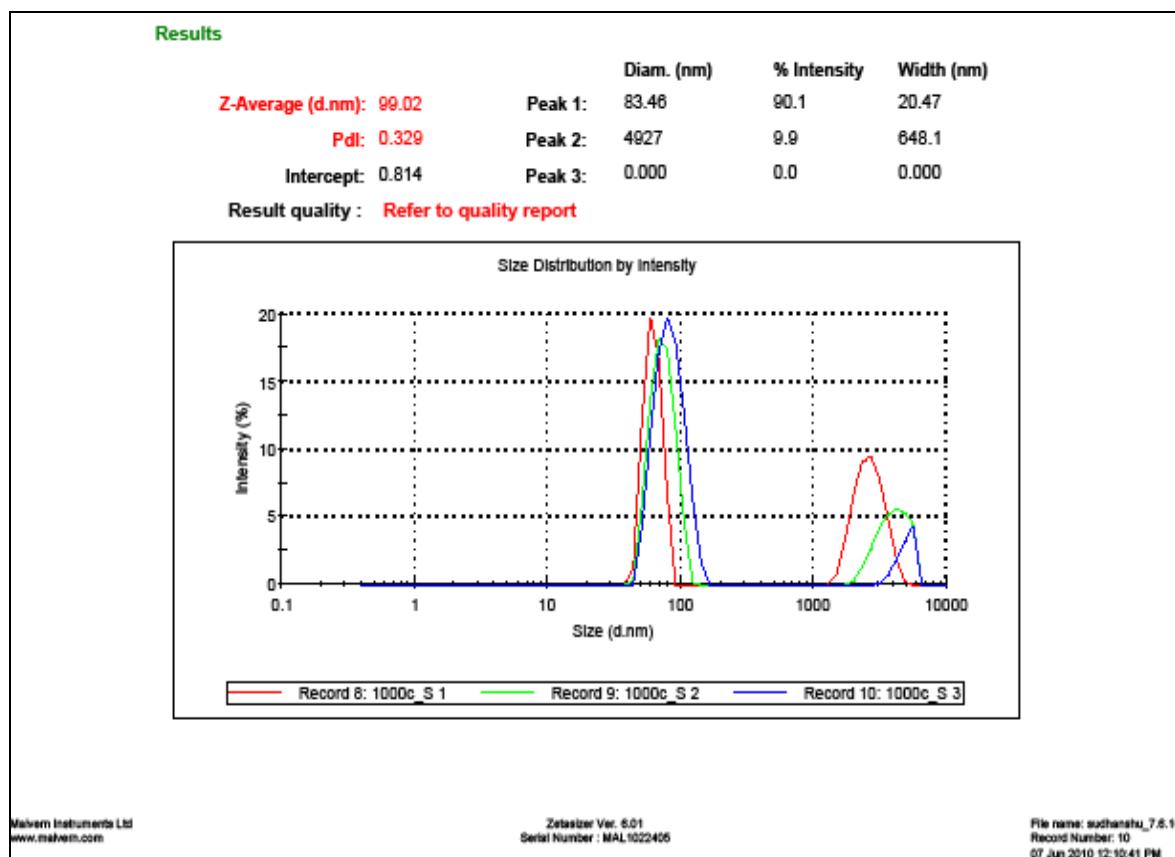


Fig. 4: DLS Results of sample decomposed at 1000°C.

Table 3

Record	Type	Sample Name	Measurement Date and Time	T °C	Z-Ave d.nm	Pdl	Pk 1 Mean Int d.nm	Pk 2 Mean Int d.nm	Pk 3 Mean Int d.nm	Pk 1 Area Int %	Pk 2 Area Int %
1	Size	1000c_S 1	Monday, June 07, 2010 11:52	25.0	108.2	0.660	61.87	2643	0.000	55.9	44.1
2	Size	1000c_S 2	Monday, June 07, 2010 11:55	25.0	105.1	0.515	73.74	3993	0.000	71.3	28.7
3	Size	1000c_S 3	Monday, June 07, 2010 11:57	25.1	99.02	0.329	83.46	4927	0.000	90.1	9.9
4	Size	800c_S 1	Monday, June 07, 2010 12:02	25.0	572.2	0.461	867.5	19.63	0.000	92.5	7.5
5	Size	800c_S 2	Monday, June 07, 2010 12:05	25.0	206.7	1.000	2342	37.79	0.000	77.8	22.2
6	Size	800c_S 3	Monday, June 07, 2010 12:08	25.0	153.5	1.000	3264	43.02	228.4	62.1	29.6

Peak 3 Area Intensity %	Mean Count Rate kcps	Multimodal Fit Error	Cumulants Fit Error	Number Mean d.nm	Volume Mean d.nm
0.0	178.5	0.00369	9.58e-4	55.50	1090
0.0	176.8	0.00157	4.98e-4	60.41	1576
0.0	156.2	0.00208	6.81e-4	63.73	995.5
0.0	200.4	0.00835	0.0238	19.15	388.3
0.0	186.1	0.00697	0.00857	35.63	1006
8.2	171.7	0.00505	0.00705	40.61	1417

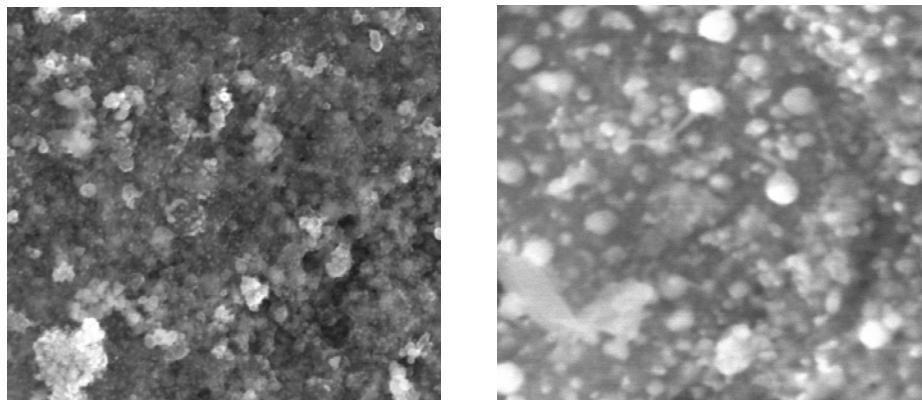


Fig. 5: STM Pictures of CNTs

Characterization by STM:- To confirm the formation of CNTs the compounds were analyzed by using STM(Scanning Tunneling Microscope). STM pictures are shown above

CONCLUSION

Nickel-carbon nano tubes can be formed by the reaction of aqueous solution of nickel salt with amino acids present in egg albumin and decomposing the compound formed.

The results also reveal that the decomposition at 800°C is incomplete and the formation of nano particles is less in the range of 10 to 100 nm. At 1000°C vast increase in the formation of nano particles in the above said range is observed.

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