SYNTHESIS AND CHARACTERIZATION OF CARBON NANOTUBES USING EGG ALBUMIN BY CHEMICAL METHOD

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ABSTRACT

Carbon nanotubes are fascinating electronic material with unique physical properties and many important potential applications. Due to various application of CNTs it is necessary to develop new methods for the synthesis of CNT’s. In this chemical method, complexes of transition metal Co (III) with amino acid present in egg albumin have been synthesized. The complex so formed, is analyzed on the basis of spectroscopic method using UV, IR, NMR spectra.

The amino acid metal complex is decomposed at higher temperature using muffle furnace to get metal carbon nano tubes. These metal carbon nano tubes are characterized using scanning probe instruments like AFM, SEM, TEM, X-RD, TGA and DTG.

KEYWORDS: Co (III), Albumin-Metal-Complex, IR, NMR, AFM, TEM, SEM, XRD

INTRODUCTION

Carbon Nanotubes (CNT’s) are allotropes of Carbon with a Nano structure. Because of their unique physical properties, Carbon Nanotubes are considered as one of the most important material of the 21st century. It consists of Graphite like Carbon, having a diameter of 1-100 nm and a length up to several micrometer or even millimeters. They are among the stiffest and strongest fiber known and have remarkable electronic properties with many other unique characteristics. Carbon Nanotubes have been the focus of considerable study because of their unusual strength along with excellent Mechanical, Electrical, Thermal, Optical and magnetic properties.[1,2,6]

Due to vast application of CNT’s, it is required to develop economical method and technique to prepare Carbon Nanotubes and characterized them. It is also required to inculcate some desired properties in them, so that they can be used in various applications.

SYNTHESIS BY CHEMICAL METHOD

Material and Method

Preparation of CNTs in Alcoholic Medium

It takes place in two steps. First is formation of Cobalt egg albumin complex and second is synthesis of CNTs.

Preparation of Cobalt Egg Albumin Complex

To prepare the carbon nanotubes the solution of cobalt metal salt with concentration of 1N was prepared in ethyl alcohol solution by AR grade techniques. The concentration of Cobalt metal ion was then allowed to react with egg albumin to give Cobalt metal albumin complex. The Cobalt metal albumin complex is taken out and kept in desiccator for
drying. The complex gets dried in forty days. Protein comprising of amino acid present in egg albumin as a monomer, when reacted with alcoholic solution of cobalt salt forms complex with Co$^{3+}$ ions. In this complex lone pair present on the nitrogen of NH$_2$ and oxygen of COO$^-$ of COOH group formed cross link between two amino acid chains.

![Chemical structure of Lysine - Cobalt (III) Complex](image)

Lysine – Cobalt (III) Complex

The complex formed was characterized by IR, NMR and AFM.

**Synthesis of CNTs**

In order to get CNTs the complex was decompose in muffle furnace. The decomposition takes place at 800°, 900° and 1000° c.[3,4,5]

**CHARACTERIZATION**

**Spectral Studies of Cobalt Egg Albumin Complex**

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

**IR Spectra**

IR spectra of Cobalt Amino acid complex is shown in the Fig- I. The information which we get by the IR Spectra is
Figure 1: IR Spectra of Cobalt Egg Albumin Complex

Table 1: Typical Infrared Absorption Hands for Cobalt Amino Acid Complex is

<table>
<thead>
<tr>
<th>Prominent Absorption Hand (CM(^{-1}))</th>
<th>Functional Group</th>
</tr>
</thead>
<tbody>
<tr>
<td>2890</td>
<td>C-H stretching</td>
</tr>
<tr>
<td>3200</td>
<td>N-H stretching</td>
</tr>
<tr>
<td>1750-1755</td>
<td>C=O stretching strong signal in Amino Acid</td>
</tr>
<tr>
<td>1590-1600</td>
<td>NH(_2) or NH(_3^+) asymmetric bending strong signal</td>
</tr>
<tr>
<td>1400</td>
<td>Weak C=O Symmetric stretching in amino acid</td>
</tr>
<tr>
<td>1481-1550</td>
<td>Strong NH(_2^+) or N(^+)H(_3) Symmetric bending</td>
</tr>
<tr>
<td>1100</td>
<td>C-O stretching</td>
</tr>
</tbody>
</table>

In infrared spectroscopy, a weak band around the 3200 Cm\(^{-1}\) assigned to the N-H stretching vibration. C=O stretching strong signal in amino acid occurs at the 1730-1755 Cm\(^{-1}\). The strong intense bands 1600 Cm\(^{-1}\) of metal amino acid complex can be attributed to NH\(_2\) or NH\(_3^+\) asymmetric bending and also a strong intense band 1481-1550 Cm\(^{-1}\) is assigned to the NH\(_2\) or NH\(_3^+\) symmetric bending. The bands at 1400 Cm\(^{-1}\) of metal amino acid complex indicates the presence of weak C=O symmetric stretching in amino acid. The band at 1100 Cm\(^{-1}\) can be assigned to the C-o stretching in cobalt amino acid complex.

**NMR Spectra**

- NMR Spectra of Cobalt Amino acid complex is shown in fig-2. The information which we get by the NMR Spectra is summed up in the table -2

<table>
<thead>
<tr>
<th>Types of Proton</th>
<th>Chemical Shift S (PPM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>H-C-COOH</td>
<td>2.487</td>
</tr>
<tr>
<td>NH(_2)</td>
<td>3.318</td>
</tr>
<tr>
<td>R.CH(_3)</td>
<td>0.84</td>
</tr>
<tr>
<td>R(_2) CH(_2)</td>
<td>1.222</td>
</tr>
</tbody>
</table>
In the "HNMR spectra of the synthesized cobalt Amino acid Complex with chemical shifts within the range 3.318 PPM the NH$_2$ Protons was observed. In the spectra, signal with chemical shift at 2.487 PPM C-H Protons attached to the COOH in the amino acids was observed in the spectra signal with chemical shifts at 1.222 PPM is related to the R$_2$ CH$_2$ (secondary type) Protons and the signal with chemical shift with the 0.840 PPM corresponds to the R.CH$_3$ (Primary type) protons also observed.

CHARACTERIZATION OF CNT’s BY SCANNING PROBEINSTRUMENT

AFM (Atomic Forces Microscopy)

Surface imaging studies were performed using Atomic force microscopy (AFM) to estimates surface morphology and particle size distribution. The samples were characterized at INSTITUTE OF INSTRUMENTATION CENTER, IIT ROORKEE. By this investigation we came to known that the linear dendritic shape of metal ions presents at the surface of Carbon nanotubes. It also predicts that the particle size is predicted 245 nm.
TEM (Transmission Electron Microscopy)

Transmission electron microscopy (TEM) is a microscopy technique. As a result, a specimen having non-uniform density, shape, and thickness can be examined by this technique. The TEM reports given below were analyzed at Institute of instrumentation center, IIT Roorkee. This analysis confirmed that the diameter of the particle is 500 nm. Black spot present on the surface of CNT’s also confirms the presence of metal ions.

![TEM Images of Carbon Nanotubes Synthesized on Decomposition of Cobalt Egg Albumin Complex at 900°C](image)

Figure 4: TEM Images of Carbon Nanotubes Synthesized on Decomposition of Cobalt Egg Albumin Complex at 900°C

SEM (Scanning Electron Microscopy)

Morphology and size of material were investigated using Scanning electron microscope (SEM) with 12 and 15 A accelerating voltages. The sample given in fig.5 was analysed at Institute of instrumentation centre, IIT Roorkee. It predicts the thickness of the CNT’s 1μm and magnification of 50000x. The spherical spots represents CO$_3^+$ metal ion on the surface of CNT’s. Which play a important role in electron transportation.

![SEM Images of CNT’s Synthesized at Decomposition of CO$_3^+$ Egg Albumin Complex at 900°C](image)

Figure 5: SEM Images of CNT’s Synthesized at Decomposition of CO$_3^+$ Egg Albumin Complex at 900°C

X-RD

The X-Ray power diffraction pattern of product were recorded with Cu Kα radiation (λ = A°) in 2θ range from 15° to 80°. The analysis of the X-RD report we find different peaks at different value of 2Theta and d. e.g ((2Theta, d) = (24.662°,3.60692; 48.877°,1.86191;50.208°,1.81560;69.804°,1.34626)), this shows that the analysed sample is multi-walled carbon nanotubes. The report of sample given in fig. was analysed at Institute of instrumentation centre, IIT Roorkee.
Figure 6: The X-RD Image of Carbon Nanotubes Synthesized at Decomposition of Cobalt Egg Albumin Complex at 800°C

DTA, TGA AND DTG (Differential Thermal Analysis, Thermo Gravimetric Analysis, Differential Thermal Gravimetry)

Thermo gravimetric analysis or TGA is a type of testing that is performed on samples to performed on samples to determine changes in weight in relation to change in temperature. Such analysis relies on a high degree of precision in threemeasurements: weight, temperature and temperature change.

The TGA thermogram predicts the 77.6% mass decomposition of metal ion in CNTs from 24-1530 cel.Hence,it predict 22.4% metal purity in CNTs. It proves the thermal stability of CNT’s.

Institute Instrumentation Centre, IITR, Roorkee

Figure 7: TGA, DTG and DTA Image of Carbon Nanotubes Synthesized at the Decomposition of 900°C
RESULTS & DISCUSSIONS

Carbon nanotubes are prepared by the decomposition of Cobalt Egg albumin complex. These Carbon metal nanotubes are formed to exhibit electrical conductivity. This shows the presence of unpaired electron and exhibit Para magnetic behavior. It is also predicted to exhibit thermal conductivity.

CONCLUSIONS

Multi-walled Carbon nanotubes have been prepared by chemical method. The IR and NMR spectra of sample proving the preparation of Cobalt Egg Albumin complex. The AFM image confirms that metal ion present has linear dendritic copolymers on to the surface of CNT’s. The SEM and TEM images predicted the spherical metal ion present on the surface of CNT’s. The X-RD studies confirm the multi-walled structure of carbon nanotubes.

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