SYNTHESISCHARACTERIZATION AND STUDY OF COBALTDOPED LYSINE CARBONNANOTUBESINAQUEOUSMEDIUM

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ABSTRACT

Carbon Nano tubes have been considered as one of the most promising material for a wide range of applications, in virtue of their outstanding properties. During the last two decades, both single walled and muti-walled CNT's probably represented the hottest research topic covering materials science. Due to various application of CNT's, it is a very interesting research area for new research to develop feasible, efficient and economical methods for the synthesis of CNT's. The work in this paper deals with the synthesis of multi-walled CNT's from step of number of unit processes and operations. CNT's have been synthesizing from the complexes of transition metal Co (II) with amino acid present in egg albumin in aqueous medium. The complex so formed, is analyzed on the basis of spectroscopic method using IR and NMR Spectra confirmed the cobalt-lysine complexes. The cobalt-lysine complex is decomposed at higher temperature using muffle furnace to get metal carbon nanotubes. These metal carbon nanotubes are characterized using scanning probe instruments like AFM, TEM, X-RD . The properties examined from the analysis shows a very high thermal stability as well as electrical conductivities. This result shows that CNT's made by this process can be further used in various applications of Electrical & Electronics devices.

Keywords:-Co (II), Albumin-Metal-Complex, IR, NMR , AFM, TEM, XRD.

I. INRTODUCTION

Carbon Nanotubes (CNT's) are allotropes of Carbon with a Nano structure. Due to the unique properties of carbon nanotubes, such as high specific surface area, thermal conductivity, chemical stability, optical, mechanical and electrical properties, carbon nanotubes are considered as one of the best area for replacing traditional conventional processes with this new emerging science aimed at miniaturizing of processes with almost efficient uses compared to others. It consists of Graphite like Carbon, having a length of 1-100 nm and a diameter up to several micrometers or even up to millimeters.The transition from micro particles to nanoparticles leads to a number of changes in physical properties. One of the major factors is the increase in the ratio of surface to volume area. As the surface area of a particle increases, the portion of its constituent atoms at or near the surface increases exponentially, creating more sites for bonding, catalysis or reaction with surrounding materials, resulting in improved properties such as increased strength or chemical or heat resistance. Additionally, the fact that nanoparticles have dimensions below the critical wavelength of light can render them transparent, which has implications for part aesthetics. They are among the stiffest and strongest

fiber known and have remarkable electronic properties with many other unique characteristics. Carbon nanotubes have a broad range of potential applications in Nano electronics, gas sensors, cancer therapy and diagnoses. Nano medicine provides a great potential in fighting many dangerous diseases like cancer and also it has great efficiency in drug delivery. [1,2,6] Due to huge application of CNT's, it is required to develop new economical methods and techniques to prepare Carbon Nanotubes and characterized them. For the economical production of carbon nanotubes protein source can be taken as the key material. It is also required to discover some desired properties in them, so that they can be used in various applications.

II. SYNTHESIS OF CARBON NANOTUBES BY CHEMICAL METHOD

2.1 Material and Method

2.1.1 Preparation of CNTs in Aqueous Medium

It takes place in two steps. First is formation of Cobalt egg albumin complex and second is decomposition of complex material to form multi-walled CNT's.

2.1.2 Preparation Of Cobalt Doped Lysine Complex

To prepare the carbon nanotubes the solution of cobalt metal salt with concentration of 1N was prepared in distilled water solution by AR grade techniques. The concentration of Cobalt metal ion was then allowed to react with egg albumin to give Cobalt metal doped lysine complex. The Cobalt doped lysine complex is taken out and kept in desiccator for drying. The complex has to be dried for 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^{2+} 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 as shown below. The complex formed was characterized by **NMR**described in characterization of complex and CNT's later in this paper.

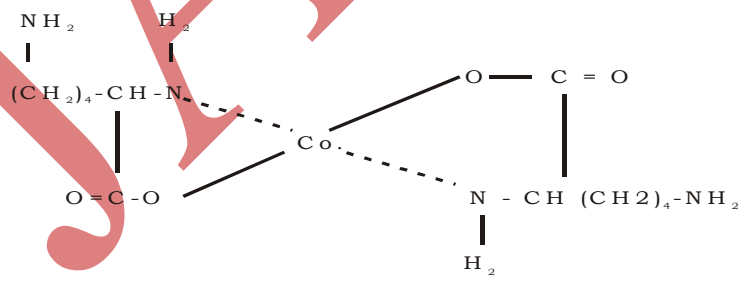


Fig-1: Lysine – Cobalt (II) Complex

2.1.3 Synthesis Of CNT'S.

In order to obtain CNT's the complex was decomposed in muffle furnace. The decomposition takes place at either of temperatures at 800 °, 900° or 1000°c. [3,4,5].After the preparation of cobalt doped lysine complex, the extra metal ion are removed from the surface by washing it from 1N HCl solution. Multi-walled CNT's are prepared mostly at temperature as high as 800-900^{0}C as at high temperature than this range the formation tends to single walled structure. The characterization of multi-walled CNT's are done from AFM, DLS, TEM,andXRD, explained in the next section of this paper.

III. CHARACTERIZATION

3.1 Spectral Studies Of Formation Of Cobalt Doped Lysine 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.

3.1.1NMR (Nuclear Magnetic Rasonance) Spectra

NMR Spectra of Cobalt Amino acid complex is shown in fig-3. The information from the NMR Spectra is summed up in the table -2 shows the chemical shift of the protons.

Table-2: Characteristics Proton Chemical Shift:

Types of proton	Chemical shift S(PPM)
$R.CH_3$	0.839
$R_2.CH_2$	1.220
H-C-COOH	2.486
NH_2	3.310

Fig-2: NMR Spectra Of The Synthesized Cobalt Doped Lysine Complex In Aqueous Medium

In the "H NMR spectra of the synthesized cobalt Amino acid Complex with chemical shifts within the range 3.310 PPM the NH_2 Protons was observed. In the spectra, signal with chemical shift at 2.486 PPM C-H Protons attached to the COOH in the amino acids was observed in the spectra signal with chemical shifts at 1.220 PPM is related to the R_2 -CH_2 (secondary type) Protons and the signal with chemical shift with the 0.839 PPM corresponds to the $R.CH_3$ (Primary type) protons also observed.

The above examination of the material by IR and NMR spectroscopy shows the presence of Cobalt doped lysine complex.

3.2 Characterization Of Cnt's By Scanning Probe Instrument

3.2.1 AFM (Atomic Forces Microscopy)

Surface imaging studies were performed using Atomic force microscopy (AFM) to estimates surface morphology and particle size distribution. By this investigation we came to know that the linear dendritic shape of metal ions presents at the surface of Carbon nanotubes. The white spots in figure 5 show the presence of Co metal ions in rare linear dendritic form with mutli-walled structures .The particle size predicted is 90 nm in . The maximum peak height obtained is of 179.25 nm with no peaks in between[8,9,10]. The distance between peak to peak in y-direction is 179 nm and in z-direction is 89.78 nm shown in figure 8-. Average sizes of peaks are 101.38 nm or Defined line distanced from the mean line (z_i) with roughness approximately 20.93 nm can be calculated by $R_a = \frac{1}{L}\int_0^L \lceil z(x) \rceil dx$ [11,12,13,14,15]

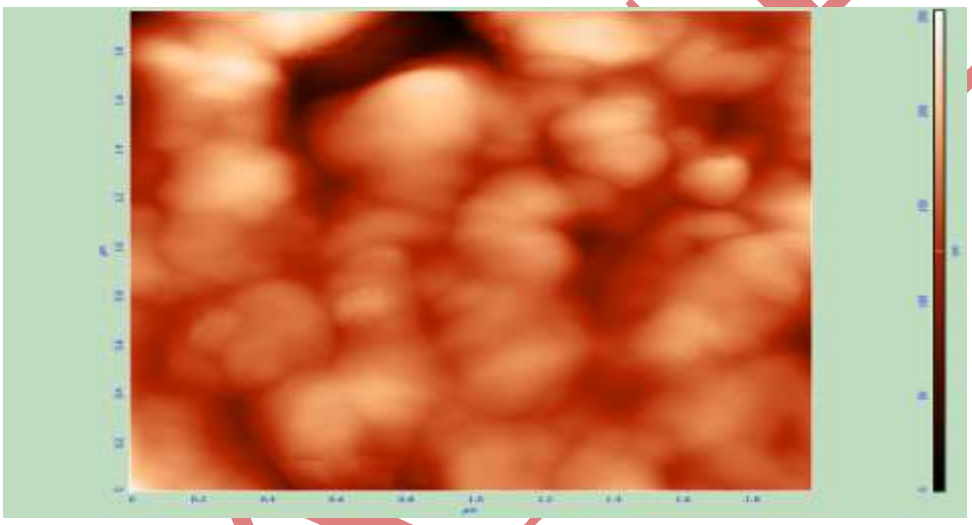
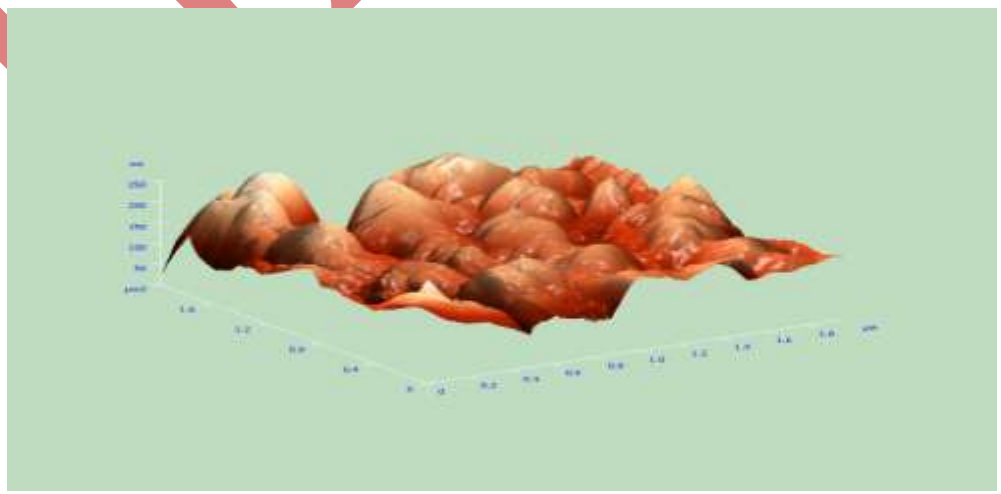


Fig 3 (a): The white spots in fig show the presence of Co metal ions in rare linear dendritic form with mutli-walled structures.



3 (b) : shows 3D morphology of Co metallic ion made CNT'sFig

3.2.2 TEM (Transmission Electron Microscopy)

As a result, a specimen having non-uniform density, shape and thickness can be examined by this technique.Figure 4 shows a TEM micrograph of cobalt nanoclusters, where almost all of the particles stick together as one can see better in the picture taken with 500 K magnification. In such cases, reliable particle counting is impossible with or without touching particles being ignored. This is not due to any inherent inability of Image; instead, it is due to the inherent tendency of thenanoparticales to aggregate[16]. This analysis confirmed that the diameter of the particle is in between 10- 20 nm.It also predicted the zig-zagdendrimer shape of carbon nano tubes [17,18,19].

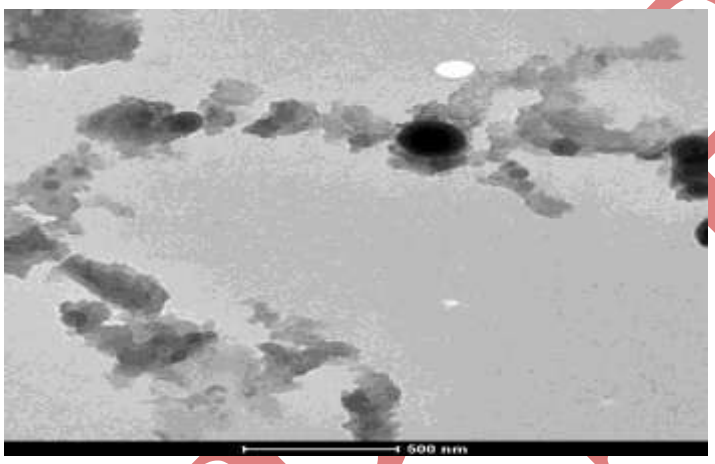


Fig 4:- TEM Images Of Carbon Nanotubes Synthesized On Decomposition Of Cobalt Egg Albumin Complex At 900

3.2.3 X-Ray Diffraction

The X-Ray power diffraction pattern of product were recorded with Cu K*a*radiation (1.54056 λ = A°) in 2 θ range from 15° to 80°.The analysis of the X-RD report we find different peaks at different value of 2ɵ and degree. e.g ((2Theta, d) = (24.662^0,3.60692; 48.877^0,1.86191;50.208^0,1.81560;69.804^0,1.34626)), this shows that the analysed sample is multi-walled carbon nanotubes. The report of sample given in figure 5

Dynamic light scattering (DLS) method has been employed for obtaining the microstructure zetapotential value, average cumulate diameter and the characteristics of the microstructure. The Malvern instrument has been used with Parameter Value **of** Sizing range 0.6 nm to 6 µmDiam, Concentration range 0.1 mg/mL ,Lys to 30w% ,Min sizing sample volume 12 µL ,Min zeta sample volume 0.75 mL, Temperature control 2 to 90 oC, Conductivity range 0 to 200 mS/cm , Laser 3 mW 633 nm He-Ne. the dispersant used is ethylene glycol with RI 1.452 the CNT has RI value of 1.59.

Dynamic light scattering techniques will give an intensity weighted distribution, where the contribution of each particle in the distribution relates to the intensity of light scattered by the particle. It also measures the time dependent fluctuation in the scattering intensity to determine the translational diffusion coefficient(D_T) and subsequently hydrodynamic radius(R_H).

Z-analysis or comulant mean from the dynamic light scattering comes out to be 94.03 nm. The Bimodal curves for each colored lines shows presence of impurities of Co ion complex and the polydespersity index value is 1 which shows broad size distribution of Co ion composites. The y-intercept can be used to evaluate the signal-to-

noise ratio from a measured sample and thus is often used to judge data quality , the intercept value is 0.939 which shows a very good sytem as an ideal system gives value of 1.0 .the three curves in the fig shows number, volume and intensity distribution respectively.

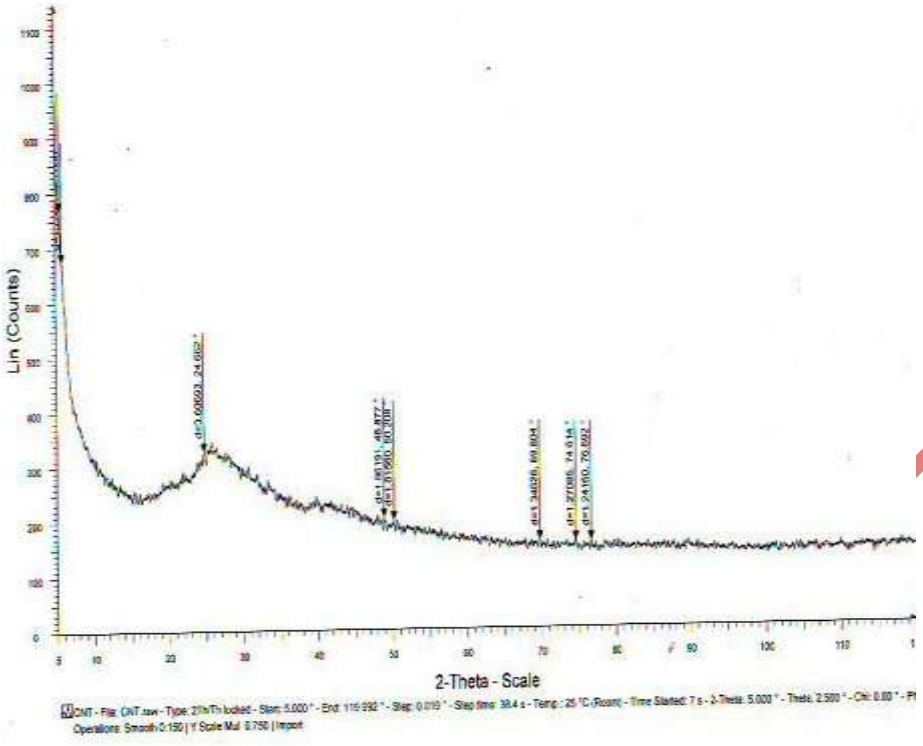


Fig-5: The X-RD Image Of Carbon Nanotubes Synthesized At Decomposition Of Cobalt Egg Albumin Complex At 800^{0}C

3.2.4 Dynamic Light Scattering

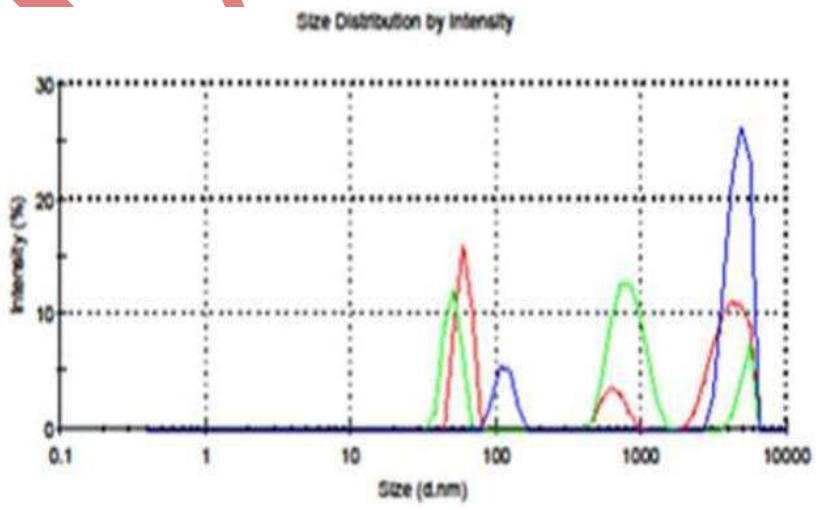


Fig.-6.Dynamic Light Scattering Of Carbon Nano Tubes Synthesized At The Decomposition Of Cobalt Doped Lysine Complex

IV. RESULT & DISCUSSION

Carbon nanotubes are prepared by the decomposition of Cobalt doped lysine complex. These Carbon metal nanotubes are formed to exhibit electrical conductivity proves by dynamic light scattering. This shows the presence of unpaired electron and exhibit Para magnetic behavior. It is also predicted to exhibit thermal conductivity.The TEM analysis confirmed that the diameter of the particle is in between 10- 20 nm.It also predicted the zig-zagdendrimer shape.

V. CONCLUSION

Multi-walled Carbon nanotubes have been prepared by AR grade technique using egg albumin and CO metal ion in aqueous medium. The NMR spectra of sample proves the preparation of Cobalt doped lysine complex. The AFM, TEM image confirms that metal ion present has a linear dendritic copolymers on to the surface of CNT's which can show a great advantages for drug delivering or chemical catalytic reactions in controlled and efficient manner. The large surface to volume ratio can be used in replacing silicon with CNT's for maximizing energy production in solar cells and electrical conductivity. The X-RD studies of the CNT confirm the multi-walled structure of carbon nanotubes. The DLS analysis proves the nano range of the carbon nanotubes and its electrical conductivity.

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