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Synthesis, characterisation and study of properties of CNTs prepared by egg protein and metal salt

KEYWORDS: Carbon nanotubes, SEM ,VSM, TGA DTA,DSC Albumin

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ABSTRACT *Nanoscience is the study of phenomenon and manipulation of material at atomic or molecular scales, where properties differ significantly from that at a larger scale.*
In nanotechnology we design, characterise and produce materials by controlling their shape and size at nanometre scale1.
CNT is a valuable Nanomaterial and is used frequently because of its remarkable properties.
CNTs are produced by using Egg protein and nickel salt, characterized and their properties are studied.

Introduction
Carbon nanotubes are molecular-scale tubes of graphitic carbon with outstanding properties. They are among the stiffest and strongest fibres known, and have remarkable electronic properties and many other unique characteristics. Due to their unique characteristics development of new techniques to produce CNTs is the area of concern of so many researchers.

For the production of CNTs high amount of carbon is required at a particular place so proteins are taken as key material to produce CNTs. Proteins are bio polymers of amino acids . Amino acids are compounds containing –NH2 and COOH groups within a molecule. With the help of these groups amino acids form complexes with metals. In these complexes different chains of amino acids are combined together. These compounds on decomposition give carbon –metal Nanotubes.

CNTs can form only when the concentration of carbon at a particular point is high so to club various protein molecules together, aqueous solution of Nickel Salt is allowed to react with amino acids present in present in Egg 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 link between two amino acid chains.

5ml of 1N aqueous solution of Nickel Salt is allowed to react with amino acids of present in egg. For that purpose egg yolk is separated from serum. The lone pair present on nitrogen of –NH2 and oxygen of COOH group present in amino acid form complex with Ni (ii). In this way nickel (ii) forms cross link between two amino acid chains. The sample is kept undisturbed in desiccator for some time for self precipitation and drying.

After drying the samples are taken out from desiccator and are ready for drying. The samples formed are decomposed at different temperatures (800°C, 1000°C and 1200°C) in a muffle furnace. At 1200°c only soot is formed.

Characterization by DLS:
1) Results of sample decomposed at 800o C: In this sample the chemical compound is prepared by the reaction of protein and nickel salt solution is decomposed at 800°c.The results are shown in the fig.1

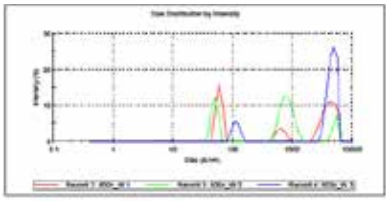
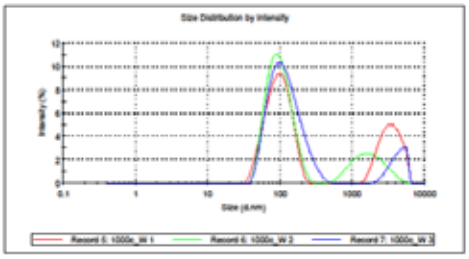


Fig1.DLF Results of sample decomposed at 800°C.

From the above figure it is clear that crystals with particle size 10 to 100 nm are formed but they are very less in number.

2) Results of sample 1000o C: In this sample the chemical compound is prepared by the reaction of protein and nickel salt solution is decomposed at 1000°c.The results are shown in the fig.2

In this figure the particles occupying nano dimension are more in number but degradation is also more.



Due to the above observations it was clear that we can get better results at a temperature of 900o C and 950o C. So the samples were again decomposed at 900 and 950oC

Table 1 DLS results

Record	Type	Sample Name	Measurement Date and Time	T	Z-Ave	PdI	Pk 1 Mean Int	Pk 2 Mean Int	Pk 3 Mean Int	Pk 1 Area Int	Pk 2 Area Int
				°C	d.nm		d.nm	d.nm	d.nm	%	%
1	Size	800c_W 1	Monday, June 07, 2010 11:29	25.0	0.000	0.000	0.000	0.000	0.000	0.0	0.0
2	Size	800c_W 1	Monday, June 07, 2010 11:33	25.0	193.1	1.000	4093	59.56	642.8	53.6	35.0
3	Size	800c_W 2	Monday, June 07, 2010 11:36	25.0	239.6	1.000	811.2	49.66	5179	58.3	29.1
4	Size	800c_W 3	Monday, June 07, 2010 11:36	24.9	813.0	1.000	4615	113.8	0.000	85.4	14.6
5	Size	1000c_W 1	Monday, June 07, 2010 11:43	25.0	130.4	0.627	99.07	3424	0.000	67.3	32.7
6	Size	1000c_W 2	Monday, June 07, 2010 11:45	25.1	115.6	0.377	101.2	1823	0.000	76.2	23.8
7	Size	1000c_W 3	Monday, June 07, 2010 11:47	25.0	123.0	0.420	125.8	4291	0.000	86.9	13.1

Peak 3 Area Intensity	Mean Count Rate	Multimodal Fit Error	Cumulants Fit Error	Number Mean	Volume Mean
%	kcps			d.nm	d.nm
0.0	540.9	0.00	0.00	0.000	0.000
11.4	232.1	0.00579	0.00179	56.20	2405
12.6	257.4	0.00420	0.00575	45.95	1045
0.0	1036.4	0.00604	0.00303	107.4	4546
0.0	333.2	0.00122	3.37e-4	51.62	1661
0.0	313.7	9.85e-4	0.00101	59.14	799.9
0.0	317.0	0.00164	5.76e-4	59.91	1305

DLS gives us idea about the particle size. It is based on the fact larger the particle size more light will be scattered by it. It

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is clear from the graph that when the metal protein complex were decomposed at 800oC, the number of particles having nano dimensions (from 10 to 100nm) is more, as the from the graphs it is clear that the peaks corresponding to particle size 10 to100nm are smaller than the peaks corresponding to 900 to 10000nm. This shows high concentration of smaller particles as small particles scatter less amount of light.

In the graph of CNTs decomposed at 1000oC though sufficient number of particles having particle size 100nm is present in the mixture but particles with size between 10 to 100 nm are very less in number so the peaks are bigger, as there is an increase in particle size. The increase in particle size can be because of the formation of some decomposed matter or association of particles due to melting.

O.Truspike Vych et al2 characterized Carbon Nanotubes Thermo Tropic pneumatic Liquid Crystal Composites, using DLS and reported similar findings. Therefore, DLS result conform the formation of nanosized particles

Characterization by SEM: SEM images of samples decomposed at 900o C and 950o C are shown below:

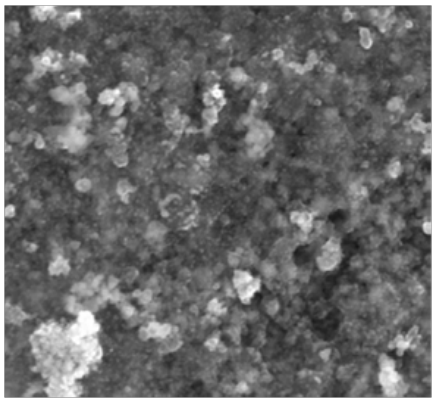

Fig: 3. SEM Pictures of CNTs at 900°C

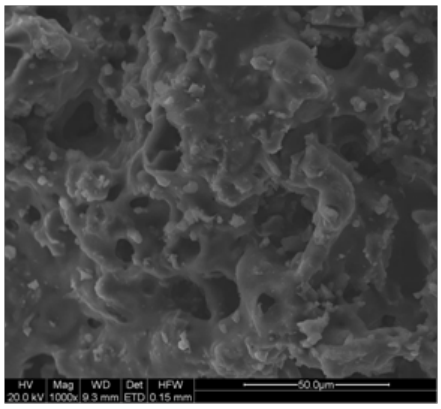

Fig: 4.SEM Pictures of CNTs at 950°C

O.Truspike Vych etal2 characterized Carbon Nanotubes Thermo Tropic pneumatic Liquid Crystal Composites using ESEM the SEM figure resembles with the figure of my CNT samples And conforms the formation of CNTs

K.Atre etal6 of Nanomaterial and nano research laboratory, USA synthesized vertically aligned carbon nanotubes and characterized using SEM. Some of their SEM images showed resemblance with our SEM images. This conform the formation of CNTs.

XRD analysis of CNTs: It is a technique used to examine physiochemical makeup of unknown solids. It is used for phase identification of a crystalline material and provides information on unit cell dimensions.XRD graph is shown in Fig.5.

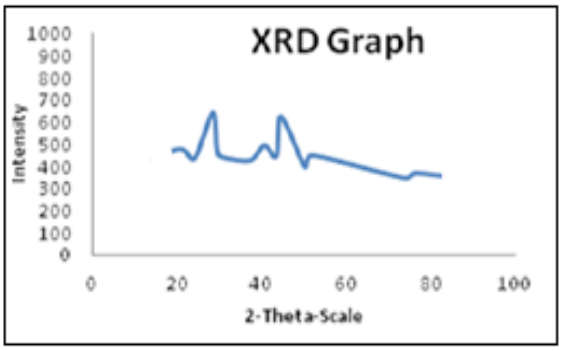

Fig: 5. Graph of Egg protein and Nickel salt in Aqueous Medium

In this graph the value of θ is 23.28 and the value of d is 3.721 this value is slightly less than the standard value.

The results were found to be almost similar to the results reported by for multi walled CNTs prepared by C.R.Bharadwaj7by pyrolysis of turpentine oil(2θ=25.6) and with Ioan Stalin etal8 ,who prepared CNTs by catalytic pyrolysis of phenol formaldehyde resin(2θ =26.2).

This conforms the formation of multiwalled CNTs.

STUDY OF MAGNATIC PROPERTIES: CNTs are expected to show paramagnetic behaviour. This behaviour is due to the presence of unpaired electrons.

The magnetic properties of CNTs are studied using VSM. The magnetic moment of CNTs is compared using a blank reference. In blank sample the magnetic moment of the instrument is studied against magnetic field. Then the experiment is repeated by taking 0.0057 gm of sample. The graphs between magnetic moment and magnetic field sample and blank are shown in fig 6 and 7.

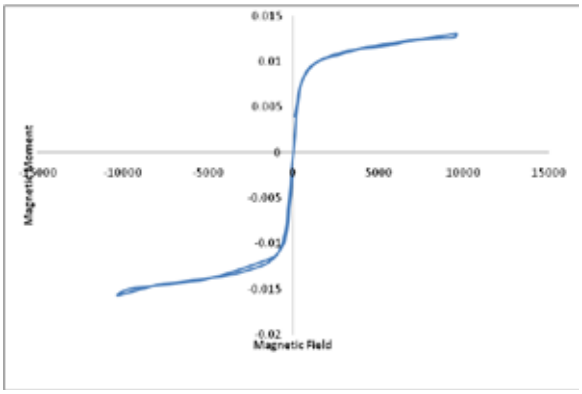

Fig:6 VSM graphs of sample

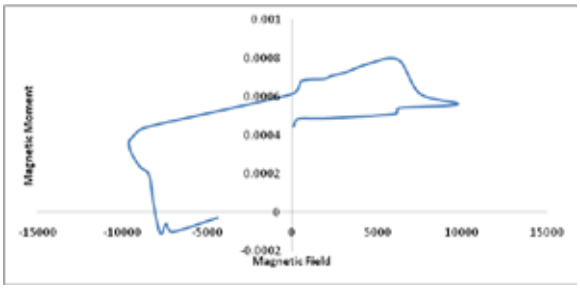

Fig: 7. Graph of blank for refence

In VSM graphs it is found that all the samples are paramagnetic in nature. It is also observed that samples have higher value of magnetic moment in comparison to the blank reference5.

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LI Wei-Xue etal11 studied the magnetic properties of multi-walled carbon nanotubes encapsulated Fe/Co particles. There graph shows resemblance with the graph of my sample. In both the graphs CNTs are paramagnetic in nature. Graphs show increase in magnetic moment with increase in magnetic field strength.

K.Atre etal6 of Nanomaterial and nano research laboratory, USA synthesized vertically aligned carbon nanotubes. They also studied magnetic properties using VSM. There VSM graph also showed similar findings and resembled with the graph of my sample.

Study of thermal properties of CNTs: Thermal properties of CNTs can be Studied by Thermal Gravimetric Analysis (TGA), DTA and DTG. TGA measures the amount and rate of change in weight of a material as a function of temperature and time in a controlled atmosphere. The TGA, DTA, DTG thermo gram of CNTs is shown in Fig .8.

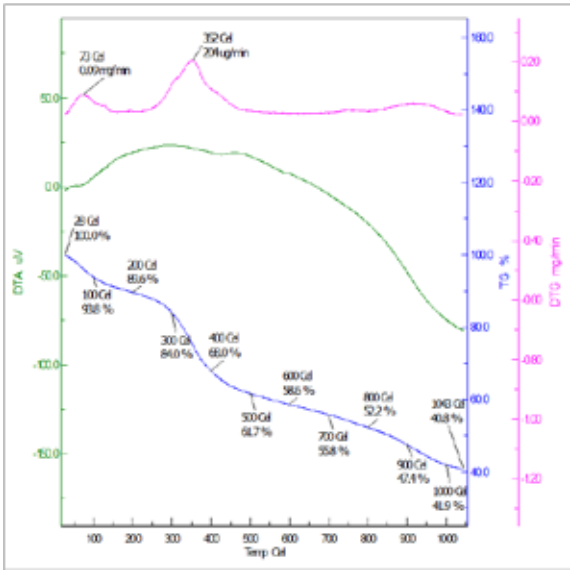

Fig:8. TGA, DTA, DTG thermo gram of CNTs of egg protein and nickel salt in aqueous medium:

The graph shows that the thermal stability and melting point of CNTs is high, the tests were carried out in inert atmosphere of Nitrogen.

At the time of TGA, DTA, DTG analysis the temperature of the furnace was adjusted to 1100°C. From the thermo gram it is clear that dehydration takes place between the temperature range from 300 to 500°C. This is also confirmed by weight losses in DTA, DTG curves. The temperature of the source reached up to 1043°C. At this temperature 40.8% sample was found to be un-decomposed. The melting point was not achieved till this temperature.

Chang etal9 studied thermal properties of multiwalled carbon nanotubes dispersed in water and associated with biological effect using TGA and DTA.

Sumio Iijima etal10 also studied the thermal properties of carbon nanotubes ultrasonicated in monochloro benzene using TGA.

Their thermo grams showed similarities with the thermo grams of our sample but there CNTs decomposed at about 800 to 850oC. The reason might be that they carried out the analysis in air not in nitrogen.

CONCLUSION: In the light of above results it is clear that CNTs can be produced by the reaction of Egg with metal and decomposing the compounds formed .By SEM and DLS result it is clear that decomposition temperature plays a vital role in the formation of CNTs. At 800oC the concentration of nanoparticles is very more but at 1000oC the concentration of nanoparticles was increased .Value of 2θ and d in XRD shows the formation of multi walled CNTs. These CNTs have unpaired electrons and are paramagnetic in nature. Due to the presence of unpaired electrons they are also expected to show thermal conductance. TGA,DTA and DTG thermo gram show that the CNTs are thermally stable.

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