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Green Synthesis, Characterization and Study of Magnetic Properties of Functionalized CNTs using Manganese and Egg Protein

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ABSTRACT

Nanotechnology is projected as the art **of** developing materials of dimension of 1 to 100nm on molecular scale.. CNTs is one of the important nanomaterials, which due to their remarkable properties and applications are considered as one of the high performance nanomaterial. Considering importance of CNTs as nano material a green route is developed to produce them using egg protein and Manganese salt solution.

Keywords : Amino Acid, IR, NMR , VSM ,XRD, TEM

I. INTRODUCTION

CNTS can be prepared by various methods. Chemical method is one of them. In the preparation of CNTs by chemical method it is the foremost requirement that the concentration of carbon at a particular point should be high. Without satisfying this condition CNTs cannot be prepared by this method. In this method protein is taken as carbon source and Nickel salt is used as catalyst. The choice of protein is because of the presence of two connecting sites (-NH_2 and –COOH) within one single molecule. Due to the presence of two connecting sites and $Mn^{2+,}$ present in Manganese salt different protein chains are clubbed together and hence concentration of carbon at a point is increased.

The egg yolk is used as protein source.

The reaction is carried out in ethyl alcohol. In organic medium, protein is denatured and waste changes are observed in the structure of protein.

In denaturation of proteins disruption and possible destruction of both the secondary and tertiary structures takes place. The primary structure of protein (sequence of amino acids) remains unaffected after a denaturation process, because denaturation reactions are not strong enough to break the strong peptide bonds.

Experimental Details: Preparation of CNTs is a twostep process in first step complex of protein and Ni^{2+} is formed and in second step the complex is decomposed in muffle furnace.

Preparation of Egg Protein $MnSo_4$ (800Alc.)

For the preparation of CNTs using egg protein in organic media, egg yolk is separated from egg white. Now 1N solution of $MnSO_4$ is prepared in ethyl alcohol and 5ml of it is mixed with 2gms of egg protein. The reaction mixture is kept undisturbed for 24 hours for precipitation of complex. After 24 hours jelly like green complex is formed. The complex is then kept in desiccator for around fifteen days. After fifteen days the complex is washed with water and dried again in the folds of filter paper. The weight of dry complex is 2.425gms.The complex is now ready to decompose and for further studies.

Decomposition: The complex formed is decomposed at 800°C in a muffle furnace. For decomposition 2gm of complex is weighed and kept in crucible. The complex is then kept in muffle furnace for ten minutes at 800°C.The weight of decomposed complex is 0.136gms.

Purification of CNTs (Removal of Nickel ions):

CNTs are kept in 12N HCl solution for this purpose 6ml HCl per gm of CNT is taken. The CNTs are dipped in solution for 24 hours. Then they are centrifuged and washed till HCi is completely removed.

Characterization: Characterization is carried out in two steps. In first step we characterize Nickel amino acid complex and in second step CNTs are characterized.

Step I st: Characterization of Amino acid nickel complex: This characterization is carried out using NMR and IR. This characterization is helpful in finding out the structure of amino acid mainly involved in the reaction.

IR Spectroscopy: The graph of IR spectra is shown in the Figure 1.

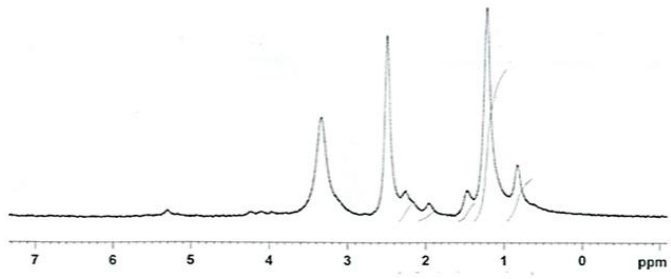

Figure 1. NMR Spectra of Amino acid Nickel complex

Chemical shift ppm	Functional group
3.153-3.334	Amino
2.246-2.486	R-CH_2OH
1.483-1.964	1°Amine
8.36	Amide

Table 2: NMR results of Amino acid Nickel complex

From NMR and IR data it is found that Lucien protein is involved in the formation of complex with nickel.

From the result of NMR and IR it is clear that +I effect plays a vital role in the preparation of protein nickel complex, the amino acid having more no. of alkyl group form compound with nickel. Chemical and physical evidences suggest that alkyl group, relative to hydrogen atoms release electrons to carbon. If an alkyl group is attached to an electron deficient carbon it donates electrons through the carbon-carbon σ bonds[2].

The structure of Lucien with nickel ion is shown in figure 2.

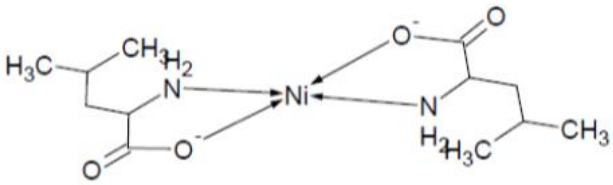

Figure 2. Structure of Lucien Ni^{2+} complex

Characterization of CNTs: CNTS are characterized using XRD &TEM and functionalization is studied using FTIR.

XRD Analysis of CNTs: XRD analysis gives idea about internal structure and crystal spacing within the crystal. XRD graph is shown in the figure 3.

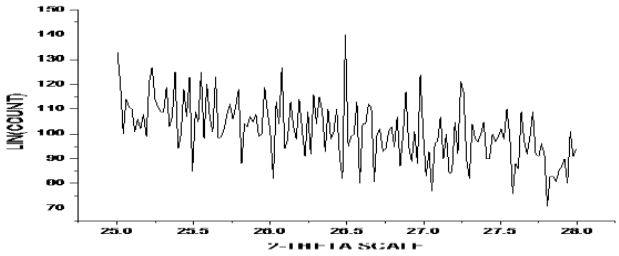

Figure 3. XRD graph of CNTs

The value of 2θ is at 26.5 which show formation of multi walled CNTs.

The results were found to be almost similar to the results reported by for multi walled CNTs prepared by C.R.Bhattacharjij3 by pyrolysis of turpentine oil (2θ=25.6) and with Ioan Stalin etal4 ,who prepared CNTs by catalytic pyrolysis of phenol formaldehyde resin(2θ =26.2).

Characterization using TEM: The TEM images of CNTs are shown in the figure 4

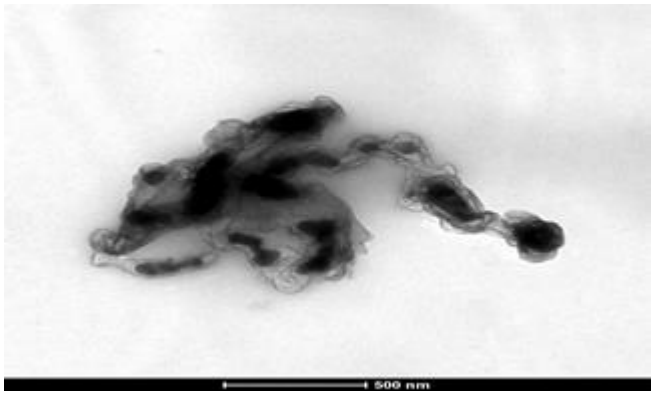

Figure 4. TEM images of CNTs

TEM images show formation of CNTs. The size of CNTs formed is 100nm.

L.A. Dobrzanski and coworkers5 synthesized Au/CNT composites. The morphology was studied by TEM.

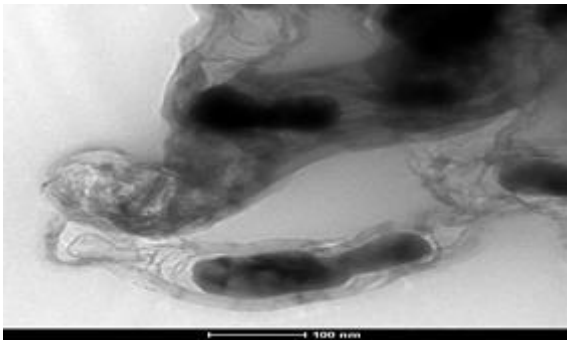

Figure 5

There TEM images showed similarities with my images.

FTIR graph of CNTs: Functionalization of CNTs is studied by FTIR graph. The FTIR graph is shown in fig.6.

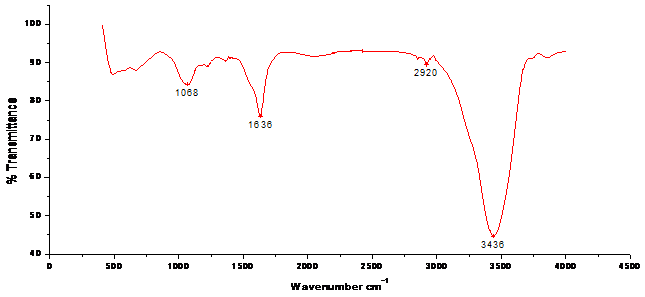

Figure 6. FTIR graph of CNTs

FTIR results are summed up in table 3[1]:

Frequency	Functional group
1068	Aliphatic Amine
1636	1° Amine
2920	Carboxylic Acid
3436	Phenol

Table 3. FTIR results of CNTs

Study of magnetic properties of CNTs: Magnetic properties of CNTs are studied using VSM. The magnetic movement of CNTs is compared with the

blank. The VSM graph of CNTs and blank are shown in figure 7 and 8.

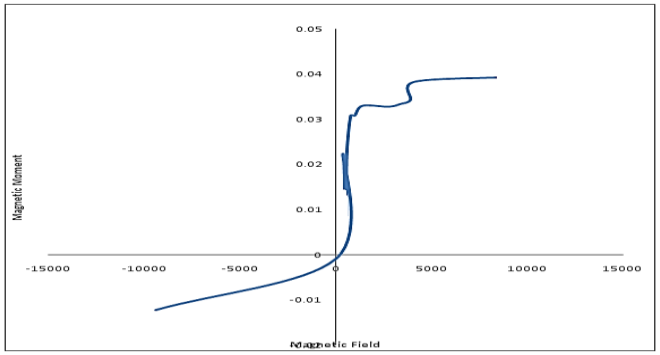

Figure 8. VSM graph of CNTs

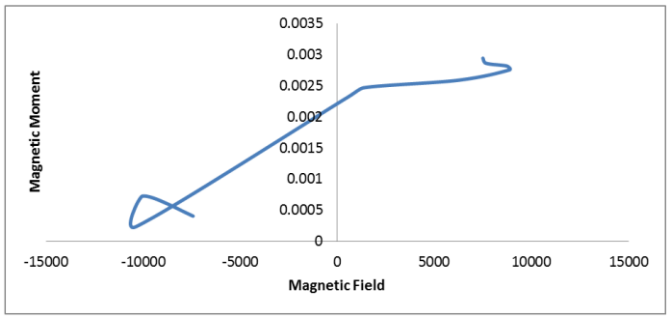

Figure 8. VSM Graph of Blank Reference

Interpretation of Graphs: CNTs are diamagnetic in nature but VSM graphs shows that all the samples are paramagnetic in nature. It is also clear from the graph that samples have higher value of magnetic moment in comparison to the blank reference6. Graphs show increase in magnetic moment with increase in magnetic field strength6 and after achieving maximum value it becomes constant.

CNTS are diamagnetic in nature the paramagnatism of CNTs is due to the metal particles embedded inside the tubes.

K. Atre etal7 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.

LI Wei-Xue etal8 studied the magnetic properties of multi-walled carbon nanotubes encapsulated Fe/Co particles. There graph shows resemblance with the graph of my sample.

II. CONCLUSION

CNTs can be prepared by the reaction of Amino Acid with Manganese salt and by decomposing the compound formed. Chemical compound is prepared in organic medium. This shows that even after denaturation, protein form desired compound with Nickel salt. This indicates that primary peptide structure is involved in compound formation.

The structure of main amino acid involved in complex formation and the structure on amino acid and protein complex is deduced by IR and NMR studies .Protein structure confirm the role of +I effect in the formation of complex.

The formation of CNTs is confirmed by XRD and TEM results. XRD results reveal that MWNT are formed.

The functionalization of CNTs formed is confirmed by FTIR results.

VSM studies confirm the paramagnetic nature of CNTs.

TEM results show that the size of CNTs is 100nm.

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