ISSN NO. 2320-5407

Journal homepage: http://www.journalijar.com

INTERNATIONAL JOURNAL OF ADVANCED RESEARCH

RESEARCH ARTICLE

Green synthesis characterization and study of magnetic properties of functionalized CNTs using Nickel and Nutrilite protein

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Manuscript Info

Manuscript History:

Received: 12 June 2014
Final Accepted: 19 July 2014
Published Online: August 2014

Key words:
Amino acid, Leucine, IR, NMR ,XRD, VSM

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Abstract

Nanotechnology is the engineering of functional systems at the molecular scale. In its original sense, 'nanotechnology' refers to the projected ability to construct items from the bottom up, using techniques and tools being developed today to make complete, high performance products. CNTs due to their remarkable properties and applications are considered as one of the high performance nano material .Considering importance of CNTs as nano material a green route is developed to produce them using protein and Nickel salt solution.

Introduction

CNTs can be prepared by various methods. Chemical method is one of them. In the preparation of CNTs by chemical method it is essential 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 $Ni^{2+,}$ present in Nickel salt different protein chains are clubbed together and hence concentration of carbon at a point is increased.

The major source of protein is Amway protein which is a mixture of soya and milk protein. It is very rich in nutritive components. Besides very high protein content, it also contains a lot of fiber and is rich in calcium, iron. As per the claims made by the company 100 gms of protein powder contain 85gms of protein and some essential amino acids.

It is claimed to have following amino acids:

Isoleucine, Leucine, Lysine, Methionine cystein, Phenylalanine, Threonine, Tryptohan, Valine, Histidine

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 peptide bonds.

Experimental Details: Preparation of CNTs is a two step 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 SoyaMilk$NiCl_2$ (900Aq)

For the preparation of CNTs using soya and milk protein in organic solution, a blend of soya and milk protein is prepared by mixing them in equal proportion and kept in a beaker. Dry milk powder of Amul Company is used as milk protein source and soya pulses are used as soya source. Now 1N solution of $NiCl_2$ is prepared in ethyl alcohol and 5ml of it is mixed with 2gms of soya and milk protein blend for the reaction. 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. The weight of dry complex is 2.355gms.The complex is now ready to decompose and for further studies.

Decomposition:

The complex formed is decomposed at 900°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 900^{o}C.The weight of decomposed complex is 0.156gms.

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 until HCl 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.

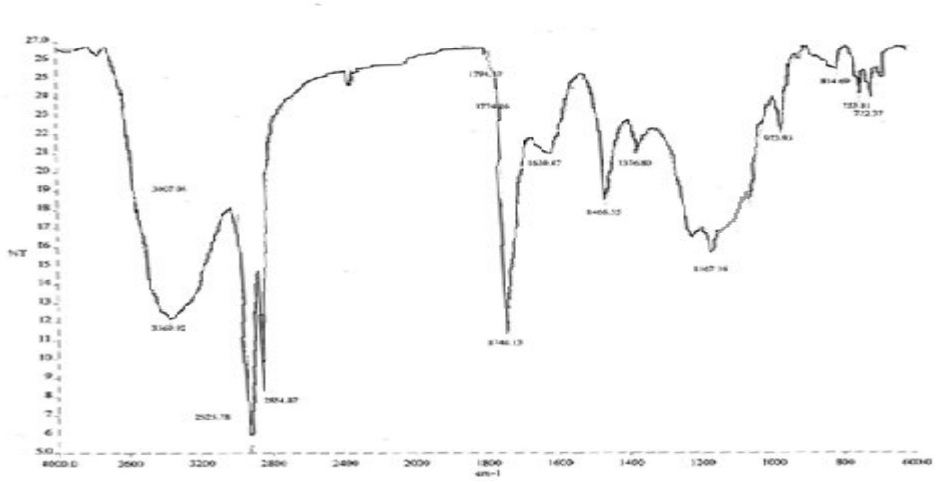

Fig.1. IR Spectra of Amino acid Nickel complex

The results are summed up in the table[1] 1:

Frequency(cm^{-1})	Functional Group
1376	C-O stretching
3369	N-H stretching

2925.78	OH of -COOH group
2854	-OH stretching
1774,1794	C=O linked with carbon attached to $–NH_2$ group
1630	C=O stretching in –COOH
1167	C-N stretching

Table 1: IR results of Amino acid Nickel complex

NMR Spectroscopy: NMR spectrum of protein nickel complex is shown in the figure 2 and the results are Summed up in table[1] 2.

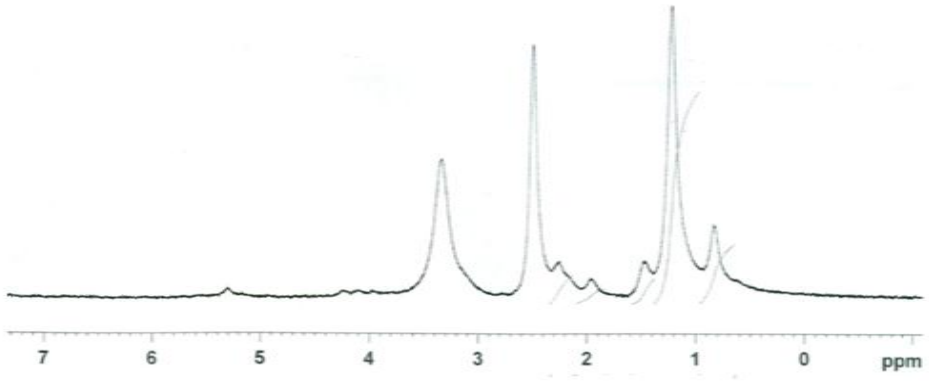

Fig.1. NMR Spectra of Amino acid Nickel complex

Chemical shift ppm	**Functional group**
3.153-3.334	Amino
2.246-2.486	$R\text{-}CH_2OH$
1.483-1.964	1^{o}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 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 3.

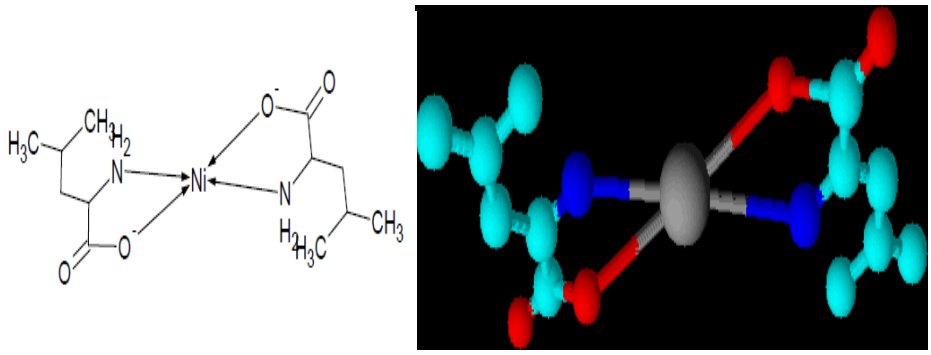

Fig.3.Structure of Lucien Ni^{2+} complex

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 4.

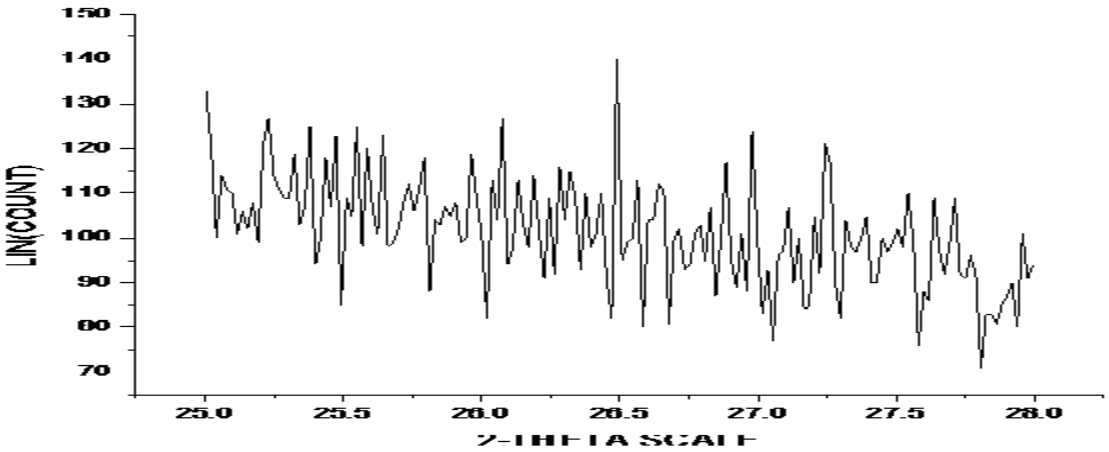

Fig.4.XRD graph of CNTs

The values of 2θ is between 25 to 28 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.Bhattacharjij[3] by pyrolysis of turpentine oil (2θ=25.6) and with Ioan Stalin etal[4] ,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 5.

Fig.5.TEM images of CNTs

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

L.A. Dobrzanski and coworkers[5] synthesized Au/CNT composites. The morphology was studied by TEM. 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.

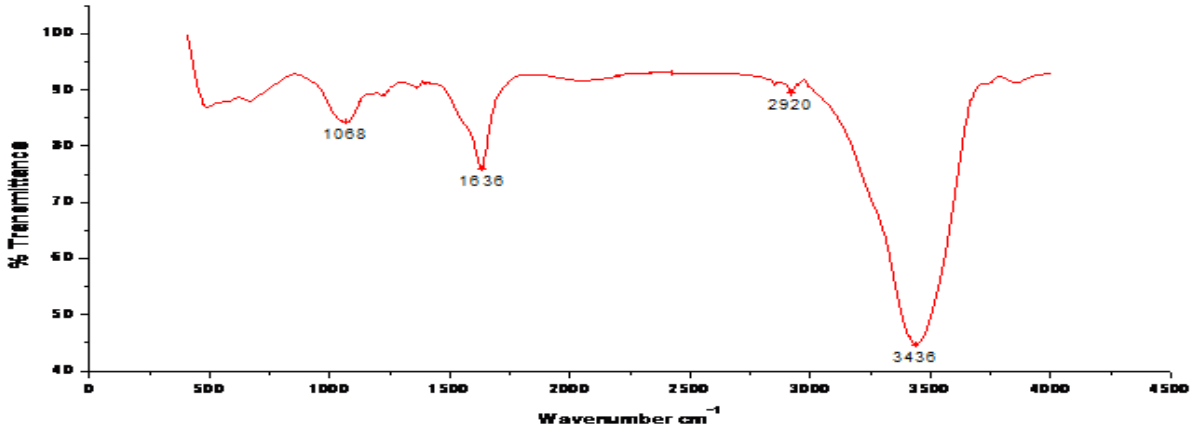

Fig.6.FTIR graph of CNTs

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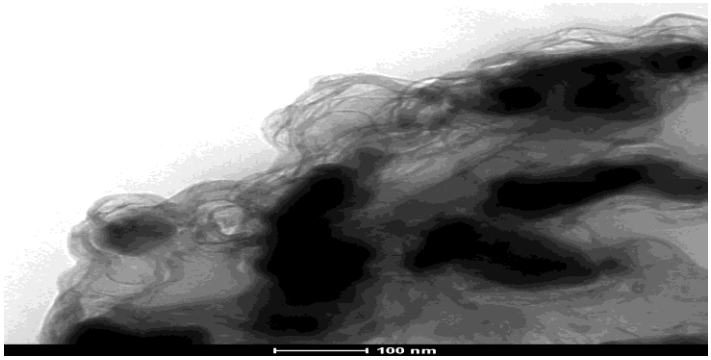

FTIR results are summed up in table 3[1]:

Frequency	**Functional group**
1068	**Aliphatic Amine**
1636	**1^{o} 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.

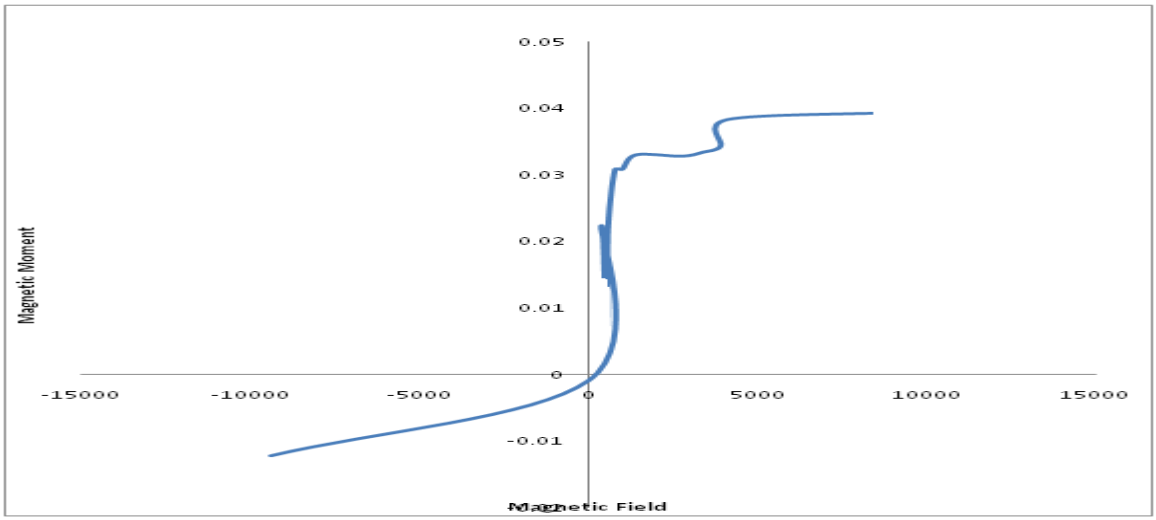

Fig.7.VSM graph of CNTs

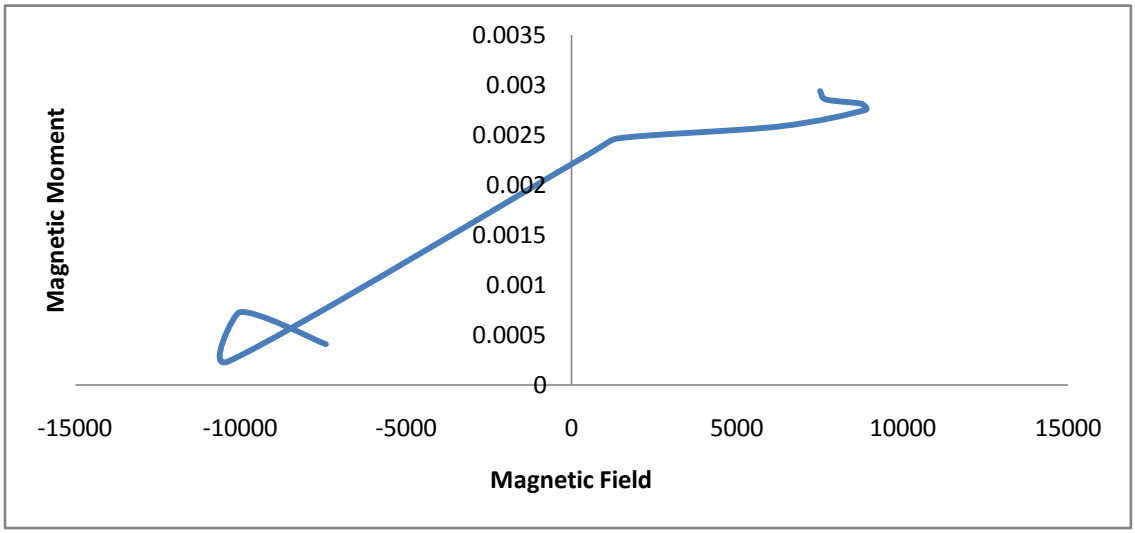

Fig.8.VSM graph of blank reference

CNTs are diamagnetic in nature but 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 reference[6]. Graphs show increase in magnetic moment with increase in magnetic field strength[6].

Actually the paramagnetic nature of CNTs is due to the nickel particles embedded inside the tubes.

. K.Atre etal[7] 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 etal[8] studied the magnetic properties of multi-walled carbon nanotubes encapsulated Fe/Co particles. There graph shows resemblance with the graph of my sample

Conclusion

CNTs can be prepared by the reaction of Amino Acid with Nickel salt and by decomposing the compound formed. Chemical compound is prepared inorganic medium. This shows that even after denaturation protein formed 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.

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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