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Preparation and Studies on Carbon Nanotubes and Zinc Encorporated Carbon Nanotubes

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This study is concerned about the one step water assisted synthesis of high quality carbon nanotubes directly from graphite and the carbon nanotube was encorporated with zinc metal. FTIR technique provide the information about the formation of carbon nanotubes and zinc encorporated carbon nanotubes. XRD analysis was carried out to find out the diameter of the encorporated carbon nanotubes.

Key Words: Carbon nanotubes, Zinc.

INTRODUCTION

The study of carbon nanotubes have, until now been divided largely into two catogories *i.e.*, the study of multi-wall nanotubes (MWNTs)^{1.2} and the study of single-wall nanotubes (SWNTs)^{3,4}. While some experiments show similarities between the two species, others reveal stark differences. For instance, some transport measurements show that MWNTs are diffusive conductors of electric currents, while SWNTs are ballistic⁵. In order to examine the differences in detail, it would be ideal to study samples of nanotubes with precisely controlled number of walls and study the progression of properties from one to two to many walls. Synthesis of exclusively double-wall nanotubes (DWNTs) is the defining step making this possible.

Arc synthesis methods that produce double-wall boron nitride nanotubes have been known for some time⁶⁻⁸. The recent studies have also demonstrated that controlled synthesis of double-wall carbon nanotubes is also possible. Temperature or electron beam induced fusing of C-60 encapsulated inside SWNTs have been used to produce double-wall structures, but in this case the inner 'wall' formed by the C-60 is not continuous for the entire length of the nanotubes⁹. More conventional nanotube synthesis methods have had a disappointingly low yield of carbon DWNTs¹⁰⁻¹² and methods that have high yields require specialized apparatus¹³, exotic ceramic precursors or sulphur-containing precursors^{14,15}. There are simple and reliable method for production of carbon DWNTs that have two walls over their entire length.

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Carbon nanotubes (CNTs) have many unique physical, mechanical and electronic properties. These distinct properties may be exploited such that they can be used for numerous applications ranging from sensors and actuators to composites. As a result, in a very short duration, CNTs appear to have drawn the attention of both the industry and medicinal field. However, there are certain challenges that need proper attention before the CNT based devices can be realized on a large scale in the commercial market. Many studies describes the distinct physical, electronic and mechanical properties of nanotubes. The basis of synthesis and purification of CNTs are reviewed and detailed descriptions have been made.

The present study aims at the synthesis and study of carbon nanotubes *i.e.*, multi-wall nanotubes or single-wall nanotubes (whichever is formed) with encapsulated zinc powder. Zinc metal powder was chosen for encapsulation as zinc acts as a high reducing agent.

EXPERIMENTAL

Preparation of carbon nanotubes: Of the several methods available for the preparation of carbon nanotubes, the technique preferred in this piece of study is the "one-step water assisted synthesis of high quality carbon nanotubes directly from graphite".

Carbon nanotube-1 (CNT-1): A small amount of finely ground pencil lead scraping (impure graphite) was taken in a silica crucible (*i.e.* the crucible used to melt gold). The crucible is closed so that the content of it to the surrounding is cut-off so as avoid further contamination. The crucible is then placed in the preheated furnace until the graphite powder in the crucible becomes red hot (above red hot). As soon as the graphite powder becomes red hot the contents of the crucible are transferred to a beaker containing ice cold water (below 0 °C). The contents of the beaker are then filtered such that the only particles floating over the surface of the water are transferred over to the filter paper. This is due to the fact that the nanotubes formed will be having very low density than the corresponding nanoparticles and other carbon additives formed.

Carbon nanotube-2 (CNT-2): The procedure for the preparation of CNT-2 is similar to that of the preparation of CNT-1 except for the fact that, the graphite powder used for the preparation of CNT-2 is highly pure with a particle size of about $60 \mu m$.

Carbon nanotube-zinc (CNT-Zn): Pure graphite powder and zinc powder are mixed in the ratio of 1:3. The mixture is then taken in a crucible and the experimental procedure is carried out as in the previous cases.

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RESULTS AND DISCUSSION

The FT-IR and XRD datas of three different samples, that is CNT-1 prepared from pencil lead, CNT-2 prepared from pure graphite powder and CNT-Zn-CNT with zinc fillings were analyzed and the following results were discussed.

CNT-1: The FTIR spectrum of CNT-1 is shown in Fig. 1. This spectrum shows the streetching frequency of (C=C) at 1658 cm⁻¹ give an ideal information about the formation of CNTs. The rest of the frequencies may be due to the impurities present along with CNTs.



Fig. 1. FTIR spectra of CNT-1

CNT-2: The FTIR spectrum of CNT-2 is shown in Fig. 2. The following frequencies seemed to be in relation with the study carried out. The band at the frequency of 1642 cm⁻¹ shows the stretching of (C=C) and the band at 638 cm⁻¹ shows the stretching of v(C-H) group. In this case a variety of peaks were observed which may be due to the overtones of the ring stretch in the CNTs.

CNT-Zn: The FTIR spectrum of CNT-Zn is shown in Fig. 3. The following observations were found. The band at the frequency of 1622 cm^{-1} shows the C=C type of stretching and the band at 1572 cm^{-1} is for (C-C) (ring) stretching. The formation of (C-Zn) bond was confirmed by the absorptions at 453, 475 and 505 cm⁻¹. The bands at 735, 675, 645 cm⁻¹ reflects the presence of Zn-Zn bonds and the C-H stretching frequency was at 963 cm⁻¹.





Fig. 3. FTIR spectra of CNT-Zn

In carbon nanotube with zinc intercalation the peaks obtained were numerous and are indicating the intercalation of zinc with that of the carbon nanotubes prepared. The careful observation of the FTIR spectrum of all the three carbon nanotube, it was noted that there were distinct O-H stretching bands at 3444, 3416 and 3436 cm⁻¹, respectively for CNT-1, CNT-2 and CNT-Zn. This may be due to the moisture absorption characteristic of the carbon nanotubes.

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XRD Analysis: The XRD data of CNT-1, CNT-2 and CNT-Zn are summarized in Table-1. Overviewing the XRD data with the help of Debye Scherrer equation, $D = 0.9\lambda/\beta\cos\theta$, the following observations were made.

QUICK VIEW OF XRD DATA						
CTNs	Peak	$\beta \times 10^{-3}$	Left	Left	Right	Right
	number	Radian	20 °	D nm	20 °	D nm
CNT-1	1	7.0127	25.560	20.3435	26.559	20.3150
CNT-2	1	3.9250	26.040	36.3411	26.651	36.3033
	2	3.7157	29.510	38.6630	29.868	38.6202
CNT-Zn	1	3.4366	26.520	41.5120	26.840	41.4766
	2	4.4832	31.590	32.2194	31.986	32.1727
	3	5.7043	33.950	25.5055	34.694	24.8701
	4	4.5530	35.690	32.1137	36.499	31.3169
	5	3.9076	43.050	38.2725	43.475	37.3069
	6	6.2800	56.310	25.1650	56.861	24.5199

TABLE-1 QUICK VIEW OF XRD DATA

In CNT-1, a very sharp peak was obtained around 26°. In the case of CNT-2, sharp peaks were obtained at about 26° and 29° and for CNT-Zn six significant peaks were interpreted. The above table exploits the various peak angles and the particle size of the carbon nanotubes formed. The diameter of the carbon nanotubes formed were calculated by using the above said equation, at different peaks. The broadened peaks corresponds to the smaller diameter.

Conclusion

The work carried out to prepare high quality carbon nanotubes and zinc encapsulated carbon nanotubes. By studying the spectral data it has been noted that the carbon nanotubes formed were preferable multi-walled carbon nanotubes along with some carbonaceous impurities. Carbon in the form of nanotubes have the tendency to absorb moisture content to a large extent. This fact is proved by the occurrence of a broad peak around 3500 cm⁻¹ in all the three carbon nanotube samples. With regard to the O-H peaks it was noted that the peak obtained for CNT-2 is more broadened and peculiar than that for CNT-1 and CNT-Zn. From this behaviour, it may be concluded that MWCNT formed from pure graphite powder have high moisture absorptibity. The diameter of zinc encapsulated MWCNTs is similar to that of the MWCNTs formed with that with pure graphite powder. Hence it may be concluded that the MWCNTs formed are encapsulated with zinc in the interior position of the nanotubes.

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