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Chemical Synthesis, Functionalization and Characterization of Multiwalled Carbon Nanotubes

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ABSTRACT

This research work is attempted to synthesize, functionalize and to characterize multi walled carbon nanotubes (MWCNTs). The synthesis of multi walled carbon nanotubes was done by chemical vapor deposition (CVD) method. The characterization of MWCNTs was done by adopting the following techniques such as field emission scanning electron microscope, X-ray diffraction, Fourier-transform infrared spectroscopy. The crystalline quality of MWCNTs was confirmed from the analysis of X-ray diffraction pattern. FE-SEM image obtained for MWCNTs and functionalized MWCNTs and it has been seen that dimeter of most of the MWCNTs lies around 90 nm, where as, functionalized MWCNTs diameter is smaller *i.e.*, around 35 nm only. Fourier-transform infrared spectroscopy study confirmed the presence of –COOH and H- bonded –OH in functionalized MWCNTs.

1. Introduction

Nanotechnology is the innovation and usage of materials, devices and systems by the control of the properties and structure of the matter at the nano scale. Carbon nano tubes (CNTs) are tubular of Buckminster fullerene was discovered by Iijima [1] in 1991. Carbon nanotubes (CNTs) are nano structured carbon allotropes of cylindrical shape, possessing outstanding physical and chemical properties. They are made up of hexagonal units of carbon. CNTs have superior electrical, mechanical and thermal properties. CNTs are divided as single walled carbon nano tubes (SWCNTs) and multi walled carbon nano tubes (MWCNTs). SWCNTs formed by a single graphene sheet and MWCNTs are formed by several graphene sheets wrapped around the tube core [2]. Multi-walled carbon nanotubes (MWCNTs) are the most important class of carbon nanomaterials with the highest production volumes and numerous technical applications. This is especially important when functionalization is required *i.e.*, grafting of chemical functions at the surface of the nanotubes to add new properties to the CNTs.

2. Experimental Methods

2.1 Synthesis of MWCNTs

The chemical vapor deposition (CVD) method, mentioned in Fig. 1, involves catalyst assisted thermal decomposition of hydrocarbons, is the most popular method of producing CNTs which is a low cost and scalable technique for mass production of carbon nanotubes [3,4]. Carbon source came from an organic compound *i.e.* toluene mixed with metal catalyst ferrocene and typical carrier gas was taken as argon likely reported by Vivekchand et al. [5].

2.2 Functionalization of MWCNTs

MWCNTs were treated by a mixture of nitric and sulfuric acid with a molar ratio of 1:3, respectively. 1 g of MWCNTs was added to 40 mL of the acid mixture. Then the oxidation reaction was carried out in a two-necked, round-bottomed glass flask equipped with reflux condenser and stirred by magnetic stirrer. The reaction was continued for 3 hours at 140 °C. Then this mixture was washed with distilled water until the pH of the filtrate was reached at 7 (neutral), and was dried in a vacuum oven at 70 °C for 24 hours [6].

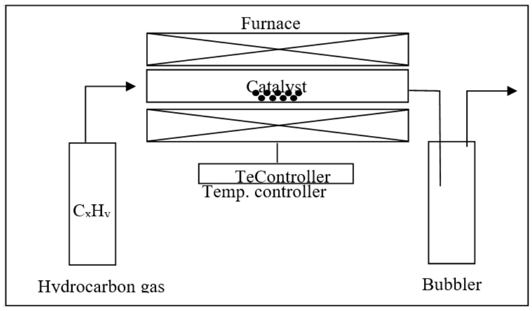

Fig. 1 Schematic diagram of a CVD setup in its simplest form [7]

2.3 Characterization of MWCNTs and Functionalized MWCNTs

Characterization of MWCNTs was made by adopting four given techniques and the results were observed which is presented here viz. X-ray diffraction, field emission scanning electron microscope and Fourier-transform infrared spectroscopy.

2.3.1 X-Ray Diffraction

XRD analysis was studied using BrukerAXSD8 Advance, Germany with CuKα radiation source wavelength 1.54 Å. 2θ scan range of the samples was 10°-80° with scan speed of 2 seconds/step. This study has been done in Department of Chemistry, Dayalbagh Educational Institute, Agra. X-Ray Diffraction pattern can be explained by following Bragg's equation, Sinθ = $\lambda/2d_{hkl}$, where, θ is half of the angle of diffraction beam and incoming beam, λ is wavelength of X-ray, and d_{hkl} the interplanar spacing between the planes.

2.3.2 Field Emission Scanning Electron Microscope

Surface morphology of MWCNT was investigated by field emission scanning electron microscope (FESEM) using MIRA II LMH from TESCAN. This characterization was done at Inter University Accelerator Centre, New Delhi.

2.3.3 Fourier-Transform Infrared Spectroscopy

Structural investigation and functional group were studied by Fourier-transform infrared spectroscopy (FTIR) using Niclot6700 using KBr as a background. This study is done in Heterogeneous Catalysis and Reaction Engineering Laboratory, Department of Chemical Engineering, IIT Delhi. The resulted spectra were found in a range of 650-4000 cm^{-1}.

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3. Results and Discussion

3.1 Synthesis of Multiwalled Carbon Nanotubes

For synthesis of MWCNTs, ferrocene was used as catalyst and toluene as carbon source. 3.2 gram of ferrocene was dissolved in 40 mL of toluene using sonicator. This mixture of toluene and ferrocene was continuously flown in reactor at the rate of 10 mL/hour along with inert gas (Ar) for 4 hours. Reactor temperature was maintained at 600 °C for production of MWCNTs by thermal decomposition of hydrocarbon. By following this process about 3 g of multi walled carbon nanotubes were obtained in one lot. Final removal of the catalysts from the tips of the nanotubes and further purification was done for optimization to yield CNTs of a higher quality [8, 9].

3.2 Functionalization of Multiwalled Carbon Nanotubes

Functionalization of MWCNTs was done with nitric acid and sulfuric acid with a molar ratio of 1:3. MWCNTs were treated with acid mixture and found that –COOH and H-bonded OH functional groups attached with the surface structure of functionalized MWCNTs. This observation was confirmed by Fourier-transform infrared spectroscopy (FTIR).

3.3 Characterization of Multiwalled Carbon Nanotubes

3.3.1 X-Ray Diffraction (XRD)

The crystalline quality of MWCNTs was confirmed from the analysis of X-ray diffraction pattern (XRD) which is presented in Fig. 2. MWCNTs shows highly crystalline structure with 8 characteristic high intensity peaks at 2θ=26.5°, 36.0°, 38.9°, 42.7°, 48.7°, 57.5°, 60.9° and 65.1° corresponding to the (002), (020), (111), (100), (110), (101), (004) and (220) planes of hexagonal system respectively. Functionalized MWCNTs showed only one low intensity peak at 2θ= 26.5° indicating smaller crystallinity. Similar results were also obtained by many researchers [10-16].

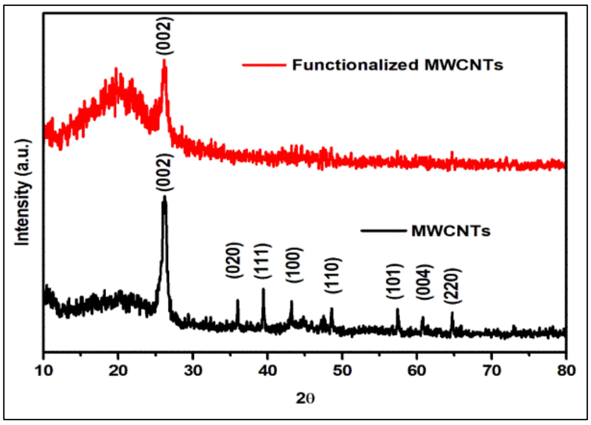

Fig. 2 X-ray diffraction pattern of MWCNT and functionalized MWCNT

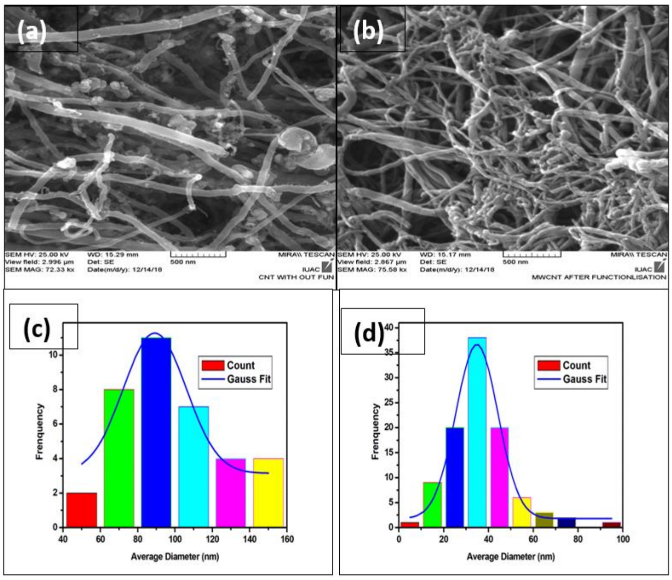

Fig. 3 FE-SEM image of (a) MWCNT, (b) functionalized MWCNT at 500 nm scale; and Gaussian fitting curve of diameter distribution of (c) MWCNT and (d) functionalized MWCNT

3.3.2 Field Emission Scanning Electron Microscope (FE-SEM)

Under FE-SEM study (Fig. 3), the average diameter size of MWCNTs and functionalized MWCNTs was found 89 nm and 34 nm respectively. The average diameter size of functionalized MWCNTs is less than that of the MWCNTs. The reduction in size is caused due to oxidation of MWCNTs with acid mixture ($1HNO_3:3H_2SO_4$).

3.3.3 Fourier-Transform Infrared Spectroscopy (FTIR)

The FTIR spectral study Fig. 4 shows the presence of different functional groups on the surface of multiwalled carbon nanotubes with the corresponding peaks at 1220 cm^{-1}, 1360 cm^{-1} and 1715 cm^{-1}. After the functionalization of MWCNTs with acid mixture (H_2SO_4 and HNO_3), the peaks were appeared at 1501 cm^{-1} [-C-O-H (-O-C)], 1867 cm^{-1} (-C=O med-stretching) 2634-3034 cm^{-1} range (-OH acidic) and 3717 cm^{-1} (-O-H; H-bonded). Thus, present study confirmed the existence of –COOH and H-bonded OH functional groups in the structure of functionalized MWCNTs. [17-20].

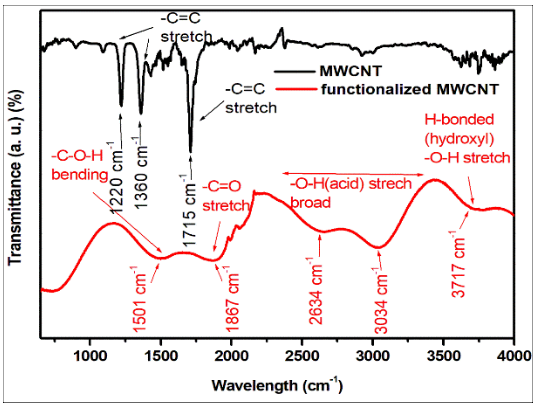

Fig. 4 FTIR spectrum of MWCNT and functionalized MWCNT

4. Conclusion

Many factors have been taken into the consideration to synthesize good quality and better quantity of MWCNTs. In the present study the desired quality MWCNTs were resulted from toluene at the flow rate of 10 mL/hr. These parameters were controlled by simple set-ups which can be manufactured in a common laboratory. X-ray diffraction (XRD) was confirmed the crystalline quality of MWCNTs. Field emission scanning electron microscope (FE-SEM) confirmed the formation of MWCNTs. Average diameter size of MWCNTs and functionalized MWCNTs was found 90 nm and 35 nm respectively. Fourier-transform infrared spectroscopy (FTIR) confirmed the functionalization of MWCNTs with -COOH and -OH functional group.

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