



Original research article

Synthesis of nanotubes under carbon environment at low temperature using hydrothermal method



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ABSTRACT

Carbon nanotubes (CNTs) are new birth in nanotechnology which becomes a pillar for scientific research due to its vast application in the field of spintronics, optical and optoelectronics materials. In the present days, CNT are highly synthesized by various popular methods which are carried out under high temperature. So, here in present work CNTs are synthesized by low temperature at 160 °C with familiar hydrothermal method using precursors as Ethanol, Polyethylene Glycol (PEG) and Sodium hydroxide with reaction time of 20 h. Here nanotubes are formed by decomposition of PEG using activating agent NaOH which enhances the decomposition of PEG. From X-ray diffraction (XRD) crystalline nature, crystal structure, average nanotube size, dislocation density are found out. And also, by Williamson hall plot strain is calculated. The presence of different functional groups and the metallic transmittance are confirmed by Fourier Transform Infra Red (FTIR) spectrum. The destination point of absorbance is found out by Ultra-Violet Visible (UV–vis) spectral analysis. And also by Tauc plot direct and indirect band gap are calculated. CNTs are readily emits green radiations which confirmed by Photoluminescence (PL) spectrum. The morphological surveys are done using Scanning Electron Microscope (SEM).

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1. Introduction

The nanomaterials and nanoscience now become a common word not only in scientific research but also in our daily life. Those materials are expected to utilize in many different applications include sensors, optical filters, low threshold laser, controlled drug delivery and biological detection etc. [1,2], Iijima discovered Carbon nanotubes (CNT) in 1991 accidentally under TEM. Due to its potential application in all field researchers focused on synthesis and analysis of CNT's [3]. Various form of CNTs are fullerene, single wall and multi wall carbon nanotubes, carbon nanofibers, carbon nanohorns, carbon nanocapsules, carbon nanonion, carbon nanospheres, ferromagnetic filled CNTs, carbon nanosheets etc. [4–12]. These carbon source materials are prepared by Arc discharge, Laser vaporization, Pyrolysis, high pressure catalytic decomposition of carbon monoxide, flame synthesis, Chemical vapor deposition (CVD), Plasma enhanced CVD, electrophoretic decomposition and high temperature hydrothermal method [13]. The carbon source materials are acetylene, methane, n-butane, propane and ethylene. And hydro carbon source are benzene, toluene, hexane, ethanol, methanol and new carbon source materials as Polyethylene glycol (PEG) and ethylene glycol (EG). Yoshimura and his co-worker synthesis CNT using PEG & EG with other source materials in the presence and absence of catalyst as Fe/Ni/Co under hydrothermal condition at 700–800 °C and

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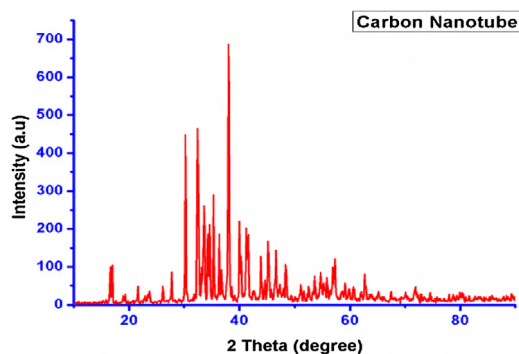


Fig. 1. XRD pattern for Carbon nanotube.

60–100 MPa, this temperature similar to CVD [14,15]. So, here we concentrate to synthesis CNT at low temperature under hydrothermal conditions using carbon source material as PEG which possess high advantage as it dissolve in water and alcohol, having low boiling point and also it have low risk of contaminating in electronics and Sodium hydroxide (NaOH) which act as an activating agent to activate the carbon source materials and enhance its decomposition.

2. Methods and materials

To synthesis carbon nanotube, Ethanol, Polyethylene Glycol (PEG) and sodium hydroxide are used as starting materials. 80 ml of Ethanol, 7 g NaOH and 2 g PEG were dissolved in 10 ml distilled water. This mixture was stirrer for 30 min, after 30 min the solution transfer to pressure vessel. The vessel was sealed and kept in furnace for 20 h at 160°C. After 20 h, obtained precipitation was filter and washed with alcohol and distilled water several time and dried in oven for 3 h. After 3 h the sample to be collected and given for further characterization [16].

3. Results and discussion

In order to investigate various properties of the Carbon nanotube, it goes under a number of characterization techniques. The crystalline nature, lattice parameters hence the structure, particle size, strains and dislocation density of the synthesized Carbon nanotubes are studied using X-ray Diffraction (XRD) technique which use source as Cu-K α radiation with wavelength of 1.54 Å. The different molecular vibrational properties are studied from Fourier Transform Infrared Red (FTIR) spectroscopy using instrument of spectrum Rxl model of Perkin Elmer spectrum range 4000–400 cm⁻¹. The optical properties like absorbance, transmittance, cut-off wavelength hence energy gap are determined from UV-Visible (UV-vis) spectral analysis using instrument Lambda 35 of Perkin Elmer range of 190–1100 nm. Also from the Tauc plot we calculate the energy gap value and confirm whether the material is direct or indirect band gap one. The excitation wavelength and its purity are determined using Photo Luminescence (PL) using instrument model of LS45. The morphological surveys are done from Scanning Electron Microscope (SEM) using instrument of ZEISS model with EHT of 10.00 kV.

3.1. X-ray diffraction

Fig. 1 shows the XRD pattern of synthesised Carbon nanotube. The presence of number peak confirms the crystalline nature of CNT. Indexing the peak using X'Pert high score plus software, the peak match with JCPDS file no: 01-075-1621, it confirms the synthesised Carbon nanotubes are in hexagonal structure whose lattice parameters are $a = b = 2.4700 \text{ \AA}$; $c = 6.7900 \text{ \AA}$ and $\alpha = \beta = 90^\circ$; $\gamma = 120^\circ$. Using Debye Scherrer formula, $D = 0.9 \lambda / \beta \cos \theta$, Where β is Full width half maximum, calculated as 0.2006° using Origin software, λ is the wavelength Cu K α radiation used in XRD as $1.546 \times 10^{-10} \text{ m}$. The average tube size is calculated as 39 nm and its dislocation density as $6.5746 \times 10^{14} \text{ m}^{-1}$.

3.1.1. Williamson hall plot to determine the strain

Williamson hall equation to calculate strain value and also the particle size of the synthesised carbon nanotube, the equation given as, $\beta \cos \theta = (K\lambda/D) + 4 \sin \theta$. From Fig. 2, the plot between $4 \sin \theta$ Vs $\beta \cos \theta$ whose intercept value gives the average particle size as 23 nm and their linear fit slope gives strain value as 0.0106.

3.2. Spectroscopical studies

3.2.1. Fourier transform infrared spectroscopy (FTIR)

FTIR was carried out in the mid IR region ranging from wave number 4000–400 cm⁻¹, the presence of vibrational peak conforms the different functional group in the synthesised carbon nanotube.

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