



Research Article

The Systematic Synthesis of Carbon Nanotubes from Aliphatic-Aromatic Compound Mixture Resolves Growth Uniformity and Production Complexity

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Received: 12 December 2019; **Accepted:** 07 January 2020; **Published:** 11 January 2020

Citation: Sydur Rahman Md. The Systematic Synthesis of Carbon Nanotubes from Aliphatic-Aromatic Compound Mixture Resolves Growth Uniformity and Production Complexity. Journal of Nanotechnology Research 2 (2020): 001-009.

Abstract

Nanomaterials exhibit interesting physical properties distinct from both the molecular and broad scales, presenting new opportunities for physico-chemical as well as biomedical researches and applications in various areas of chemistry, biology and medicine. The unique chemical, physical and mechanical properties of carbon nanotubes have stimulated extensive investigation since their discovery in early 1990s by Iijima. Although there have been tremendous advances in the fabrication of CNTs, the integration of these nanostructures into successful applications and large-scale production processes are yet not very smooth. The present research interest focuses on the development of

simple and effective way for the synthesis of carbon nanotubes and proposes the new idea of the organic bulk method. Aliphatic-aromatic compound mixture was used as carbon sources. The carbon nanotubes thus produced were very uniform in size and shape.

Keywords: Carbon Nanotubes; CNTs; MWCNTs; CNT Synthesis; Organic Bulk Method

1. Introduction

Nanotechnology and Nanoscience are about controlling and understanding matters on submicrometer and atomic scale. By definition they are exciting

multidisciplinary fields which involve the design and engineering of objects or tools, characterization, production, and application of structures, devices, and systems by controlled manipulation of size and shape at nanometer (shortly nm) scale. Nanomaterials have sizes ranging from about 1nm up to several hundred nanometers, comparably to many biological macromolecules such as enzymes, antibodies, and DNA plasmids [1]. Materials in this size range exhibit interesting physical properties, distinct from both the molecular and bulk scales presenting new opportunities for biomedical research and applications in various fields including biology and medicine. Among the large variety of nanostructures in the fullerene family, recently one particular member has become the focus of

a great deal of scientific and technological attention: the carbon nanotube (shortly CNT). The basic structural unit of CNT is a graphitic sheet (Figure 1) rolled into a cylindrical shape. In practice, we can roughly divide CNTs into two different classes, either by considering their structure or synthesis method, these are single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) (Figure 1). The first class includes cylinders formed by a single graphitic layer where the typical diameter is 1-1.5nm [2]. Sometimes the diameter reaches to 3-4nm [3]. The second class of tube includes structures formed by the coaxial arrangement of several (2-50) SWCNTs; their external diameter is of the order of 2-100nm [4].

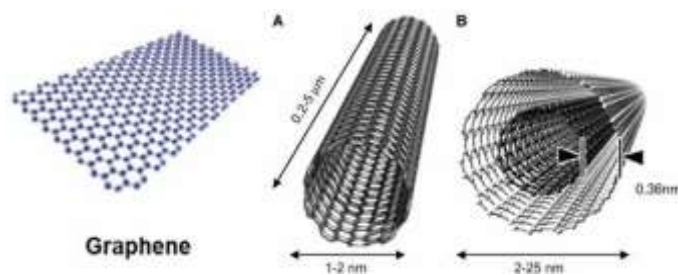


Figure 1: Graphene sheet (left) rolled into cylindrical structure for both SWCNT (A) & MWCNT (B) (schematic diagram).

Since their discovery of CNTs by Iijima [5, 6] there has been extensively studied by researchers in various fields such as chemical, physical, materials and bio-chemical sciences. CNTs have unique nanostructures with remarkably mechanical, thermal and electrical properties, which made them highly attractive for the use as reinforcement in nanotube based composite materials [7, 8]. Another fascinating aspect of CNTs is their cavities, which can be used to incorporate atoms or molecules in order to generate novel compounds or nanostructured materials [9]. Within the family of nanomaterials, CNTs have emerged as new alternative and efficient tools for transporting and translocating

therapeutic molecules [10, 11]. CNT can be functionalized with bioactive peptides, proteins, nucleic acids and drugs, and used to deliver their cargos to cells and organs. Because functionalized CNTs display low toxicity and are not immunogenic [12, 13], such systems hold great potential in the field of bio-nanotechnology and nanomedicine. The ultimate goal of this research work is to develop the preparation technique to synthesize the quality product of CNTs (MWCNTs).

2. Materials and Methods

The chemicals employed in this research work were of analytical graded and were obtained from Sigma

Aldrich, England with minimum purity of 99.5%. Water used was doubly distilled throughout the experiment. The aliphatic-aromatic compound mixture was prepared by using an analytical balance with a precision of $\pm 0.1\mu\text{g}$. Special care was taken to prevent evaporation and the introduction of moisture into the experimental samples. First, a solution of 40ml of equal aliphatic compound mixture (hexanol + octanol) and 60ml of equal aromatic compound, mixture (benzene + m-xylene) was prepared. 7g (about 15% by weight of carbon sources) of benzalkonium chloride (BZK) and 1g of FeCl_3 was then added and the mixture was stirred for 12 hours. BZK is a cationic surfactant which played a role in stabilizing nanoparticles to be formed. 0.5g of hydrazene hydrate was then added as reducing agent and the mixture was stirred again for 24 hours to get densed. In the mean time iron nanoparticles were formed by reduction of the FeCl_3 present in this combined densed solution. Then forced pyrolysis was carried out by introducing the obtained solution into a tube furnace with an inert atmosphere by argon gas at 600°C for 20 minutes. Synthesized CNTs thus obtained were then purified and characterized by Energy Dispersive X-Ray spectroscopy (EDX), Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), Fourier Transform Infra-red (FTIR) spectroscopic analysis. The tube diameter and morphology of the synthesized CNTs were analyzed by SEM characterization (Figure 2, 3). EDX spectroscopy provided the information about the chemical composition (Figure 4) of synthesized CNTs. XRD (Figure 5) and FTIR (Figure 6) spectra confirms CNT's growth identity.

3. Results and Discussion

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions

reveal information about the sample including external morphology (texture) to confirm the size and shape of nanoparticles. The SEM images were taken with an average magnification up to $\times 100000$ at room temperature. The study of the SEM images shows the clear size & shape of CNTs. The SEM images for CNTs are shown in Figure (2, 3) which show dense and clear nanotubes. Distinguishable CNTs are visible at high resolution and the cross section confirms CNTs specific diameter. Obviously, the quality is much better [14, 15]. The diameter of the nanotubes are about 70-95nm on average (Figure 3). The elemental analysis was successfully confirmed by EDX measurement. It's characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing a unique set of peaks in it's X-ray emission spectrum. From Figure 4 it can be observed that there is a clear abundance of carbon element which sharply supports that CNTs contain only carbons. The abundance was simply detected by the k (alpha) shell electrons at 0.277keV. The carbon percentage by mass and percentage by atomic abundance is 99.34%. Very little amount (0.66%) of unwanted oxygen may come from alcohols. X-ray crystallographer is a tool used for identifying the atomic and molecular structure of a crystal, in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific directions. In Figure 5 X-ray diffraction spectral images and corresponding data were provided for the synthesized CNTs. The height, FWHM (full width at half maxima), d-spacing value and relative intensity at $2\theta^0$ of CNTs give result of high accuracy. The X-ray diffraction image displays the clear sharp peak at 25.72 position which clearly confirms that respective compound is CNT. This higher density of electrons at this position is almost identical for every CNTs (MWCNTs).[16] Fourier Transform Infrared Spectroscopy, also known as FTIR analysis or FTIR Spectroscopy, is an analytical technique used to identify

organic, polymeric, and in some cases, inorganic materials. The resulting spectra produce a unique molecular “fingerprint” which can be used to easily screen and scan samples for many different components. FTIR spectra (Figure 6) of synthesized CNTs shows a lot of significant peaks in between 1400- 1650 cm^{-1} which indicates the C-C stretching present in CNTs in ring form (in CNT six carbons ring). Peak in 2368.4 cm^{-1}

which might be O=C=O stretching for linear molecules CO_2 due to unwanted oxidation of CNTs. Small peaks in 690-760 cm^{-1} are for C-Br band stretching. Unwanted element Br came from KBr used for the FTIR experiments. Peaks present in 2850- 2921 cm^{-1} for O-H bending may come from unreacted alcohols used as carbon source. The above information confirms that growth percentage of CNTs was very good.

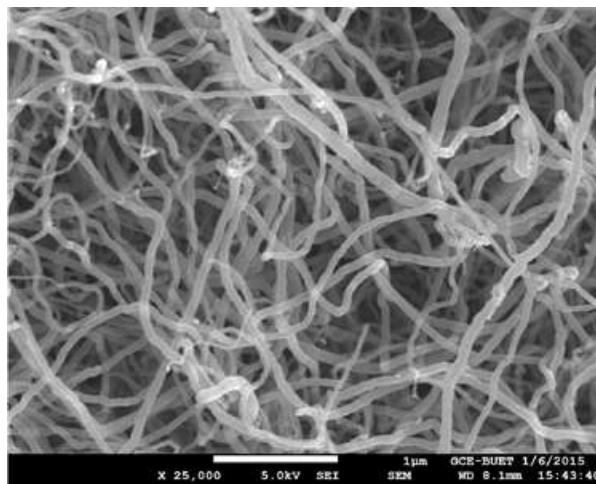


Figure 2: SEM image to observe the uniform tube shape of synthesized CNTs.

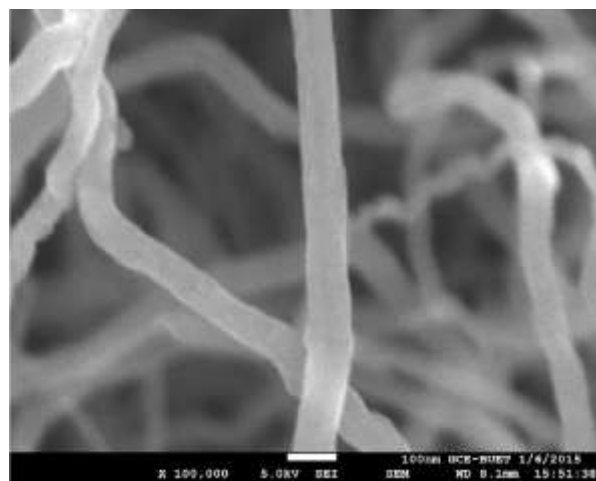
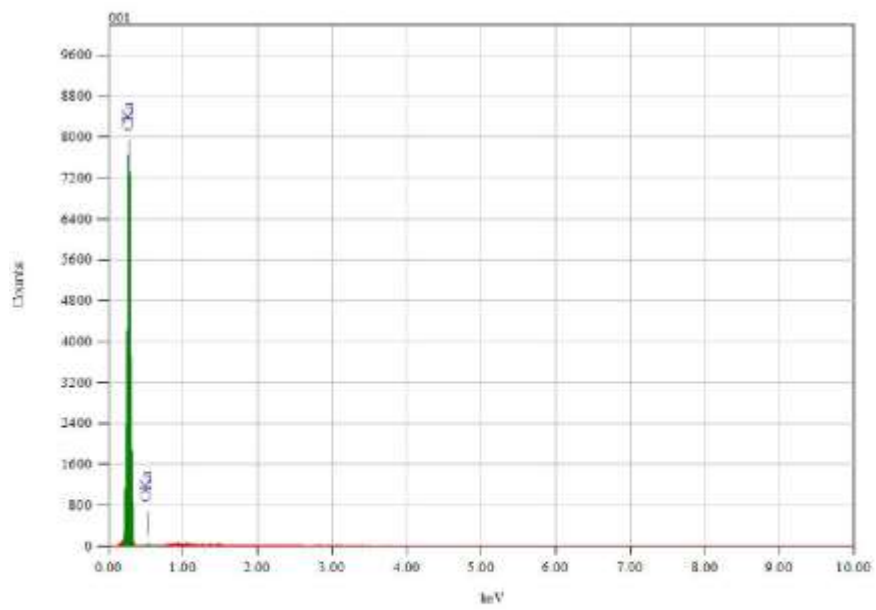
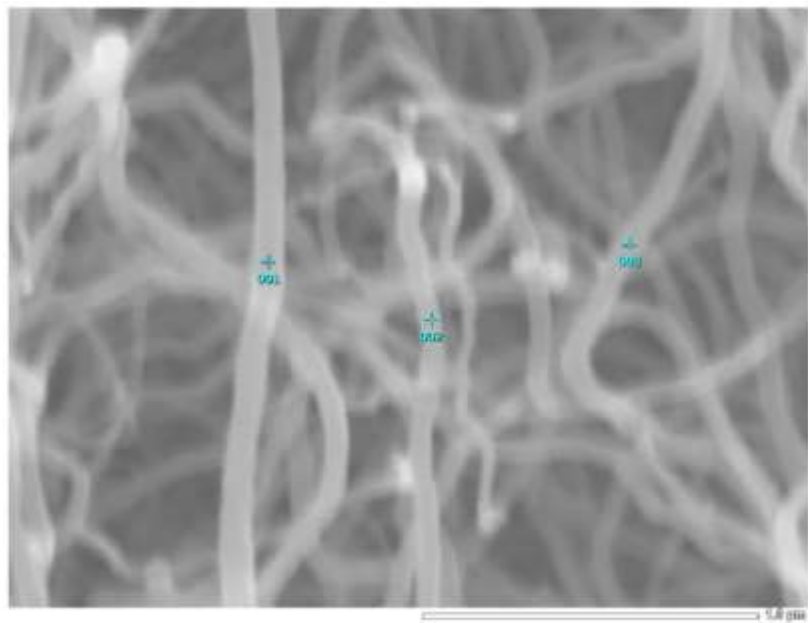


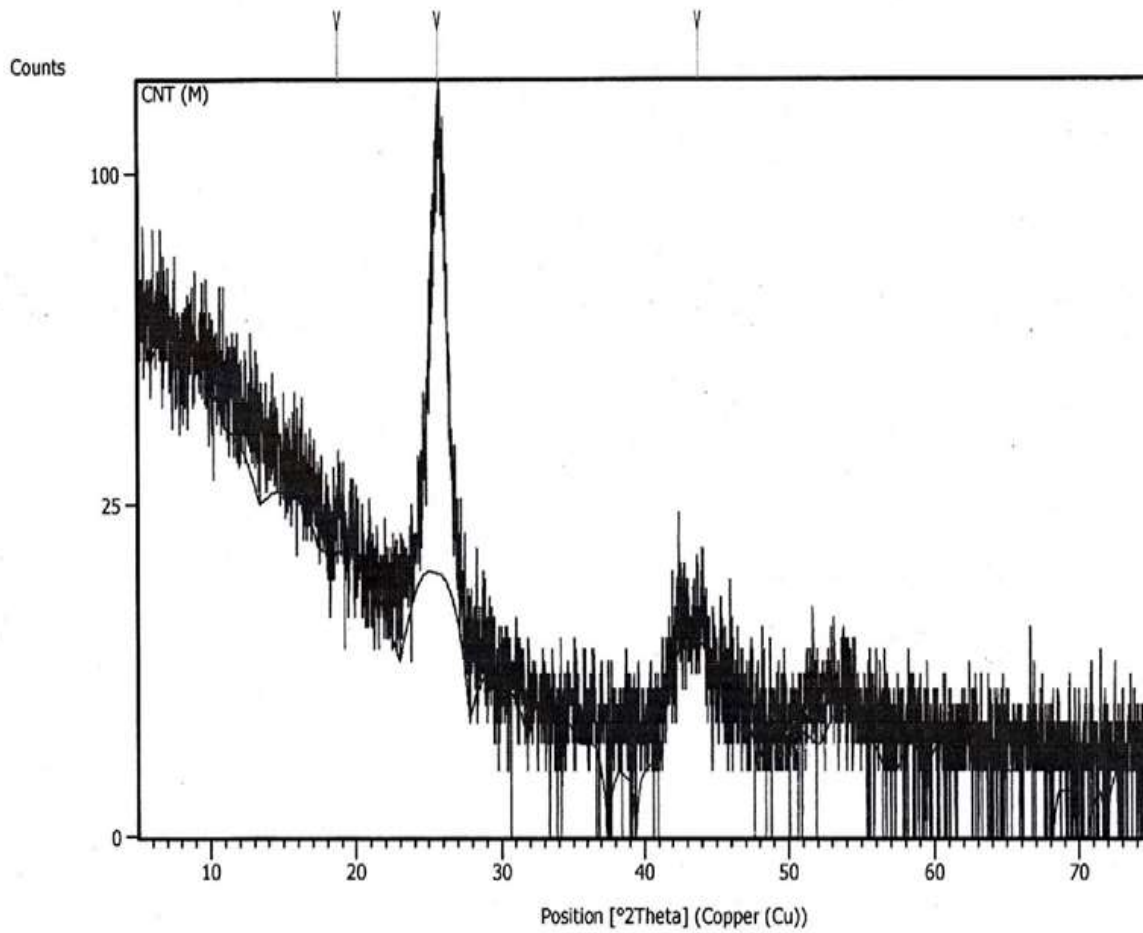
Figure 3: SEM image also confirms tube diameter as 70-95nm range.



Element	(keV)	Mass%	Sigma	Atom%	Cc
C K	0.277	99.12	0.24	99.34	
O K	0.525	0.88	0.07	0.66	
Total		100.00		100.00	

Figure 4: EDX data to observe the elemental confirmation of synthesized CNTs.

Main Graphics, Analyze View:



Peak List: (Bookmark 3)

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
18.8652	7.93	0.4723	4.70406	7.78
25.7250	101.91	0.4723	3.46315	100.00
43.8934	2.94	0.4723	2.06274	2.88

Figure 5: X-ray diffraction spectra and resultant data to confirm growth identity of synthesized CNTs.

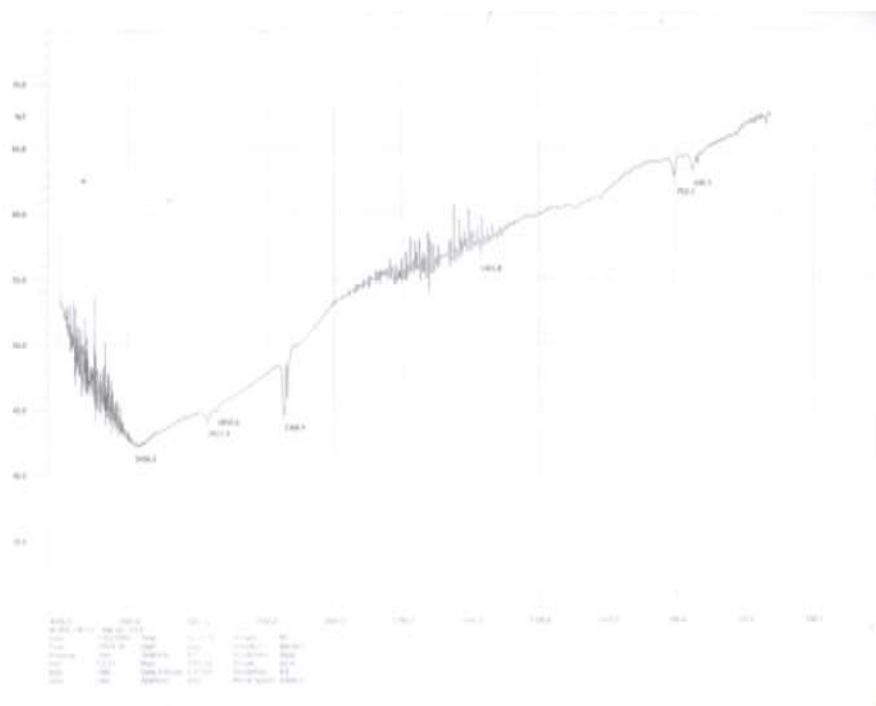


Figure 6: FTIR spectra of synthesized CNTs to observe significant bands.

The above characterizations confirm the significant growth of CNTs with highly uniform cylindrical structure. The method is capable in monitoring growth directions on the substrate mixtures and synthesizing a large quantity of CNTs. This advanced, integrated and authentic process can resolve the complexity of the uniformed growth of CNTs in large scale.

4. Conclusion

CNTs are low-dimensional Sp^2 carbon nanomaterials, exhibit many unique physical and chemical properties that are interesting in a wide range of areas including nanomedicine. Large scale production processes of CNTs depend on the understanding of several fundamental issues, which are yet to be addressed. The carbon arc discharge method is a technique that produces a complex mixture of components and requires further purification to separate the CNTs from the soot and the residual catalytic metals present in the

crude product which is very much complicated.[17, 18] Producing CNTs in high yield depends on the uniformity of the plasma arc and the temperature of the deposit forming on the carbon electrode that is difficult to maintain. Another formulation technique of CNTs is laser vaporization method.[19, 20] Arc discharge and laser vaporization methods involve evaporating the carbon source, so it has been unclear how to scale up production to the industrial level using these approaches. The role played in the chemical vapor deposition (CVD) of CNTs is not yet fully understood.[21] The simplistic view that only plays a catalytically passive role in the formation of CNTs requires examination. In this research work, selective growth of CNTs (MWCNTs) by organic bulk method using aliphatic- aromatic compound mixture has been studied. Evidences of self-assembled carbon nanostructures are presented, In addition, CNTs growth was assumed to be possible from other carbon sources like free long chain hydrocarbons. The integration of

CNTs in this manufacturing may solve the growth uniformities too.

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