UNIVERSITI PUTRA MALAYSIA

EFFECTS OF HYDROGEN FLOWRATE ON CARBON NANOTUBE PRODUCTION AT LOW TEMPERATURE VIA FLOATING CATALYST CHEMICAL VAPOR DEPOSITION METHOD

ELMIRA HABASHI ZADEH

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By

ELMIRA HABASHI ZADEH

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Chairman: Hamdan b. Mohamed Yusoff, PhD
Faculty: Engineering

Well-aligned multiwall carbon nanotubes (CNTs) were successfully synthesized by floating catalyst chemical vapour deposition method at 600 ˚C. In this research, benzene and ferrocene were used as carbon source and catalyst respectively. Hydrogen flow rate and preheating temperature were considered as variables in this research in order to study the impacts of these parameters on the quantity, structure and morphology of produced CNTs. The effect of hydrogen flow rate on the temperature variation inside the reactor was studied as well. The parameters such as reaction time (45 min), synthesis temperature (600 ˚C) and amount of ferrocene (200ml) were kept constant during the experiments. Argon was used at flow rate of 350 ml/min before and after the reaction for creating an inert atmosphere and preventing oxidation accordingly. The hydrogen flow rate was varied from 150 ml/min to 450 ml/min with increment of 50 ml/min. The preheating temperature was set at 200 and 300 ˚C. The morphology and structure of the produced CNTs were analysed by Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM), X ray Diffraction analysis (XRD). The purity of the product were determined by using Thermal Gravimetric Analyser (TGA).

The results show that the quantity of CNTs has been increased with the increase of hydrogen flow rate and preheating temperature. The quality of CNTs has been improved by increasing the hydrogen flow rate from 150 ml/min to 350 ml/min. The study reveals that lower pre-heating temperature (200 ˚C) has led to better quality. It was found that well-aligned CNTs were produced with different purity, depending on the hydrogen flow rates at 200 ˚C preheating temperature. The results indicate that the quality and quantity of CNTs are affected by both hydrogen flow rate and preheating temperature. The temperature variation inside the reactor in different hydrogen flow rate is negligible.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

KESAN KADARALIR HIDROGEN TERHADAP PENGELUARAN TIUB NANO KARBON PADA SUHU YANG RENDAH MELALUI KAEDAH DEPOSIT WAP KIMIA PEMANGKIN TERAPUNG

Oleh

ELMIRA HABASHI ZADEH

September 2014

Pengerusi: Hamdan b. Mohamed Yusoff, PhD
Fakulti: Kejuruteraan

Nanotiub Karbon (CNTs) yang mempunyai beberapa lapisan yang selari telah berjaya dihasilkan dengan mengunakan kaedah 'Floating Catalyst Chemical Vapour Deposition'(CVD) pada suhu yang rendah. Didalam kajian ini, benzena dan ferosena telah digunakan sebagai sumber karbon dan pemangkin. Kajian melibatkan kesan parameter (kuantiti, struktur dan morfologi) terhadap CNTs yang dihasilkan dengan menggunakan kadar aliran hidrogen dan suhu pra-pemanasan sebagai faktor pembolehubah. Parameter yang digunakan secara konsisten sepanjang kajian ini adalah masa tindak balas (45 min), suhu sintesis (600 °C) dan kuantiti ferosena (0.2 g). Argon juga telah digunakan (kadar aliran adalah 350 ml/min sebelum dan selepas tindakbalas) untuk mewujudkan tekanan atmosfera yang seimbang dan mencegah berlakunya pengoksidaan. Kadar aliran hidrogen telah di ubah dari kadar 150 ml/min kepada 450 ml/min dengan kenaikan sebanyak 50 ml/min. Suhu pra-pemanasan telah ditetapkan pada 200 °C dan 300 °C. Morfologi dan struktur CNTs yang dihasilkan dianalisa dengan menggunakan Mikroskop Pengesan Elektron (SEM), Mikroskop Transmisi Elektron (TEM) dan X ray Pengalihan (XRD). Ketulenan produk telah ditentukan dengan kaedah Analisis Gravimetrik Haba (TGA).

Keputusan dari hasil kajian telah menunjukkan peningkatan kuantiti CNTs dengan penambahan kadar aliran hidrogen. Kualiti CNTs pula telah berjaya ditingkatkan dengan penambahan kadar aliran hidrogen daripada 150 ml/min kepada 350 ml/min. Kajian ini juga menunjukkan bahawa suhu pra-pemanasan yang lebih rendah (200 °C) berjaya menghasilkan CNTs yang lebih berkualiti dan dalam kuantiti yang tinggi. CNTs yang tersusun cantik juga berjaya dihasilkan dengan ketulenan yang berbeza bergantung kepada kadar aliran hidrogen pada suhu pra-pemanasan 200 °C. Keputusan menunjukkan bahawa kualiti dan kuantiti CNTs sangat dipengaruhi oleh kadar aliran hidrogen dan suhu pra-pemanasan.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

Hamdan b. Mohamed Yusoff, PhD
Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Fakhrul-Razi b. Ahmadun, PhD
Professor
Faculty of Engineering
Universiti Putra Malaysia
(Member)

BUJANG BIN KIM HUAT, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

Signature:	______________________	Signature:	______________________
Name of Chaiman of Supervisory Committee:	**Hamdan b. Mohamed Yusoff**	Name of Member of Supervisory Committee:	**Fakhrul-Razi b. Ahmadun**

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LIST OF ABBREVIATIONS

Ar	Argon
Co	Cobalt
CO_2	Carbon Dioxide
CNT	Carbon Nanotube
CNTs	Carbon Nanotubes
CVD	Chemical Vapor Deposition
EDX	Energy-Dispersive X-ray spectroscopy
Fe	Ferum
FEDs	Field Emission Display
HRTEM	High Resolution Electron Microscopy
MgO	Magnesium Oxide
MWNTs	Multi Walled carbon Nanotubes
ND:YAG	Neodymium-doped Yttrium Aluminum Garnet
Ni	Nickle
PLD	Pulsed Laser Deposition
PSNT	Polystyrene Targets
SWNTs	Single Walled carbon Nanotubes
TEM	Transmission Electron Microscopy
TGA	Thermal Gravimetric Analysis
UV	Ultra Violet
XRD	X-Ray Diffraction analysis

CHAPTER 1

INTRODUCTION

1.1 Introduction

In recent years, carbon nanotubes (CNTs) have attracted the attention of scientists all over the world due to the extraordinary and unique mechanical, physical and electronic properties as well as nano scale dimension.

These special properties make carbon nanotubes an excellent candidate for flat panel field emission displays, nano electronic devices, chemical sensors, hydrogen storage and scanning probe tips (Dong et al., 2002).

The first carbon nanotube (CNT) which was a multi walled carbon nanotube discovered by Iijima in 1991 when he was studying the surface of a carbon electrode in the arc discharge apparatus. After the studies via high resolution transmission electron microscopy (HRTEM), the new carbon filament was named as multi walled carbon nanotube. Two years later, the single walled carbon nanotube was produced by Iijima and Ichihashi (Iijima and Ichihashi, 1993).

Since the discovery of carbon nanotubes by Iijima, (1991) several techniques have been used for carbon nanotube synthesis such as arc discharge, laser ablation and chemical vapor deposition methods. Although indicated methods can be applied to produce carbon nanotubes, vast researches are under development in order to find out an optimized process considering production cost, quantity, and quality factors.

Chemical Vapor Deposition method is considered as the best method because of its low system cost, easy control of experimental conditions and lowest synthesis temperature (Zhao et al., 2011). Generally in this method, an electrical furnace covers a reactor to provide the required heat to decompose carbon source and catalyst. Carbon source will be transferred into the reactor by a gas. The CNTs will be grown inside the reactor and will be collected after the reactor is cooled to room temperature.

The future use of carbon nanotubes depends on an easy and economical technique for synthesis. For obtaining the most proper method for carbon nanotubes production, it is necessary to study different parameters which are present in the process. Many researchers have worked on process parameters to achieve the best and economical method to produce carbon nanotubes. Most of these researches have been conducted in high temperature (Rao et al., 1998). Some investigations have also been performed in UPM about carbon nanotubes synthesis (Atieh, 2005; Othman, 2007; Atiyah, 2009).

Atieh, (2005) investigated the effect of hydrogen flow rate in the range between 50-500 ml/min on the synthesis of multiwall carbon nanotubes. He found that the maximum yield and purity of carbon nanotubes is obtained when the hydrogen flow rate was around 300ml/min. On the other hand, Othman, (2007) claimed that the quality of produced carbon nanotubes would be maximized when the hydrogen flow rate was set at 350 ml/min. These two researches both were done at 850 ˚C.

Moreover, Atiyah, (2009) successfully synthesized carbon nanotube between 500 ºC to 600 ºC by using chemical vapor deposition method. He employed ferrocene as catalyst, benzene as carbon precursors and hydrogen as carrier gas. Golshadi et al., (2014) studied the effect of three key process parameters (deposition time, temperature, and gas flow rate) on overall carbon mass deposition rate and CNTs wall thickness and morphology by chemical vapour deposition method.

1.2 Research Problem

Different parameters affect the synthesis process such as temperature, carbon source, and hydrogen flow rate. Hydrogen is frequently present in the hydrocarbon processing system so the study of hydrogen flow rate effects would be significantly important in this method (Lobo and Trimma, 1973).

Wasel et al., (2007) mentioned that chemical vapor deposition (CVD) is considered as one of the techniques that enables control of structure of grown CNTs. In particular, experimental data suggest that the amount of hydrogen in the gas flow affects the morphology of the resulting carbon products.Numerous attempts to control CNT growth have been performed but the effect of hydrogen on the structure and growth rate of CNTs is still not clear, and more investigations is needed (Lebedeva et al., 2011).

Hydrogen flow rate has an essential role in the production of carbon nanotubes. It is reported that the maximum yield and high purity of the CNTs will be achieved by optimization of hydrogen flow rate (Atieh, 2005). It is also believed that hydrogen prevents poisoning the catalytic surface with carbon deposition by providing the reducing environment for the catalytic metals (Danafar et al., 2009).

Most of the studies on hydrogen effects on CNTs production have been done in high temperature. Atieh, (2005) studied the effect of hydrogen flow rate at 800˚C reaction temperature. Samant et al., (2006) studied the effect of carrier gas (H_2, N_2 and Ar composition) flow rate on quality and yield of produced CNTs at 950˚C. Othman, (2007) investigated the effect of hydrogen flow rate at 850˚C reaction temperature. She also studied the effect of adding fresh hydrogen into the system.

However there in no evidence of conducting these investigations in the lower temperature which can lead to decrease in the cost and to ease the control of the process.

Atiyah, (2009) successfully synthesized CNTs in the range of temperatures from 530 ˚C to 600 ˚C. His results indicated that the highest value and highest quality of CNTs were obtained at 600 ˚C, so in this research the study has been performed at 600 ˚C to understand the limitations of CNTs production at low temperature by considering hydrogen flow rate and preheating temperature as variable parameters.

1.3 Significance of study

During the 21st century a lot of researches have been done on carbon nanotubes. Their applications to almost all the scientific areas, such as aerospace science, bioengineering, environmental energy, materials industry, medical and medicine science, electronic computer, security and safety, and science education encourage scientists to conduct more examinations in this field.

Synthesis of carbon nanotubes is one of the most significant topics in these years. The production of carbon nanotube for wide range of uses and obtaining desired quality and quantity play an important role in the application and commercialization of this kind of material. Tjong et al., (2008) said for successful commercialization of nano materials and application of them in different industries, precise understanding of fundamental aspects is required. For obtaining the most proper method for carbon nanotubes production, it is necessary to study different parameters which are present in the process. In this research, the effect of career gas flow rate is studied because the carrier gas affects structural characteristics and uniformity of grown CNTs (Danafar, 2009).

Many researches mentioned that the CNTs synthesis at low temperature is very critical for integrating nanotubes in electronics (Liao et al., 2006; Mora et al., 2007; Devaux et al., 2008). The CNTs which are produced in the synthesis temperature below 600 °C can be easily used in semiconductors (Maruyama et al., 2002). Moreover Liao et al., (2006) reported the CNTs, intended to be used in electronics, should be produced with reaction temperature lower than 660 °C. Current work presents the effect of hydrogen flow rate on carbon nanotube production by chemical vapor deposition method at 600 °C.

1.4 Objectives and Scope of Research

The objectives of this research are:

1) To investigate the effect of hydrogen flow rate and on carbon nanotube production via chemical vapor deposition method in low temperature.
2) To analyse the effect of preheating temperature on carbon nanotube production via chemical vapor deposition method in low temperature
3) To study the effect of hydrogen flow rate on reactor temperature during the process

The scope of this study is directed towards investigating the effect of hydrogen flow rate on the quantity and quality of CNTs produced in low temperature and identifying the optimum flow rate of hydrogen in which the synthesized carbon nanotubes quality and quantity is maximized . The effect of hydrogen flow rate on the reactor temperature which has a significant effect in CNTs production is studied furthermore the effect of preheating temperature on the quality and quantity of the CNTs is considered.

The hydrogen flow rate varied from 150-450 ml/min with 50 ml/min interval. The preheating temperature was considered 200 ˚C and 300 ˚C. The reaction involved catalytic decomposition of benzene in temperature 600 ˚C in CVD reactor. Ferrocene was chosen because no preparation is needed and it can be used directly. The reaction was done under atmospheric pressure to eliminate its dependency on pressure factor. The characterization of produced carbon nanotubes was performed by scanning electron microscopy (SEM), transmission electron microscopy (TEM), thermal gravimetric analysis (TGA), X-ray diffraction analysis (XRD) and energy-dispersive X-ray spectroscopy (EDX).

1.5 Outline of Thesis

The thesis consists of five chapters. Chapter 1 describes the introduction, research problem, objectives and scope of the research briefly.

Chapter 2 covers the literature reviews on the structure, properties, applications and synthesis methods of carbon nanotubes. Growth mechanism and purification of CNTs are described in this chapter too.

Chapter 3 covers the methodology which has been used in this study. In this chapter, the experimental work and characterization methods such as SEM, TEM, TGA and XRD will be described in details.

Chapter 4 presents the data that was collected during the experiments. In this part, the results are shown as figures, graphs and table. This chapter explains the results analysis and discussions.

Chapter 5 describes the conclusion of the work and recommends some improvement suggestion for future work.

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