UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERIZATION OF CARBON NANOTUBE FROM WASTE COOKING OIL USING FLOATING CATALYST CHEMICAL VAPOUR DEPOSITION METHOD

NOOR LYANA BINTI ADNAN

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By

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DEDICATIONS

Alhamdulillah.

Every challenge need self-efforts as well as guidance of elders especially those who very close to our heart. My humble effort, I dedicate to my sweet and loving

Ibu, Ayah, Fahir and Shafiyyah

DON'T STOP UNTILL YOU PROUD OF YOURSELF

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

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December 2017

Chairman: Ismayadi Ismail, PhD
Institute: Institute of Advanced Technology

Research in nanotechnology is gaining interest due to its unpredictable nature and unique properties, making it one of the most research topic in the century. Due to the extraordinary properties of carbon nanotubes (CNTs), a lot of scientific research on the synthesize of CNTs structures have been studied around the world. The floating catalyst chemical vapor deposition (FCCVD) technique is a very promising and desirable technique for bulk CNTs cotton synthesis due to its simplicity, low cost and yield and does not require chemical processes to produce the final product. However, conventional CVD methods typically produce CNTs from carbon source which is available commercially such as ethanol.

Disposing of used cooking oil becoming a problem because the solid waste regulations restrict the disposal of liquids in landfills. Subsequently unlawful disposal arises which includes open burning that causes black smokes, pouring down to drains that can clog the sewer system and eventually lead to unsanitary conditions. All this has disrupted the ecological environment, marine life and leading to global warming. Therefore, we have discovered a new method to produce bulk CNTs cotton using waste cooking oil as carbon source. The objective of this thesis is to synthesize CNTs using waste cooking oil as carbon source via FCCVD method.

In this thesis single stage floating catalyst chemical vapor deposition (FCCVD) were used, the liquid hydrocarbon solution were waste cooking oil and ethanol (CH_3CH_2OH) as carbon source, ferrocene ($C_{10}H_{10}Fe$) as a catalyst and thiophene (C_4H_4S) as promoter, argon (Ar) and hydrogen (H_2) as gas carrier. The liquid hydrocarbon solution is injected into a hot furnace along with hydrogen as carrier gas. Upon entering the furnace, these compound break down and react rapidly to form carbon nanotubes,

which then interact to form a continuous cylindrical-shaped aerogel that is collected at the end of the tube reaction. In this thesis, production of CNTs cotton depends on various process parameters such as thiophene concentration, gas ratio of $Ar:H_2$, ferrocene concentration, and liquid hydrocarbon solution flow rate were investigated. The morphology and structures of multiwall carbon nanotubes (MWCNTs) produced were characterized using Field Emission Scanning Electron Microscope (FESEM), High Resolution Transmission Electron Microscope (TEM), Thermo Gravimetric Analysis (TGA), X-ray Photoelectron Spectroscopy (XPS) and electrical properties were studied.

Result reveal that the addition of 1.0 wt% thiophene to waste cooking oil causes a great increase on the amount of CNTs cotton obtained compared to that other concentration at 1150 °C. The ratio of $Ar:H_2$ which was 400:300 sccm when using waste cooking oil as carbon source could cause more energy consumption, while it helps to achieve a high growth rate and aligned CNTs, due to the more presence of direct carbon precursor. As the concentration of ferrocene was increased by using ethanol as carbon source, causing the iron cluster to become bigger for the nucleation of CNTs. Concentration of 1.0 wt% and 1.5 wt% of ferrocene gave good morphological in structure and better properties. Lastly, high residence time, which is 5 ml/h liquid hydrocarbon flow rate by using ethanol as carbon source, may cause the excessive of carbon source supplement and accumulation of byproducts that lead to hybrid structure called graphenated carbon nanotubes. In conclusion, synthesize of CNTs using waste cooking oil were successfully carried out. Therefore, we provided an alternative idea for utilization of waste cooking oil to usable product for various applications in the future.

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

SINTESIS DAN PENCIRIAN KARBON NANOTIUB MENGGUNAKAN MINYAK MASAK TERPAKAI MELALUI TEKNIK KATALIS APUNGAN PEMENDAPAN WAP KIMIA

Oleh

NOOR LYANA BINTI ADNAN

Disember 2017

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Penyelidikan dalam nanoteknologi semakin menarik kerana sifatnya yang luar biasa dan unik, menjadikannya salah satu topik penyelidikan yang paling dibincangkan pada abad ini. Oleh kerana sifat karbon nanotiub (CNTs) yang luar biasa, banyak penyelidikan saintifik mengenai sintesis struktur CNTs sedang berjalan di seluruh dunia. Teknik katalis apungan pemendapan wap kimia (FCCVD) adalah teknik yang sangat sesuai untuk sintesis CNTs kapas pukal kerana mudah dikendali, kos rendah dan hasilnya tidak memerlukan proses / rawatan kimia untuk menghasilkan produk akhir. Walau bagaimanapun, kaedah CVD konvensional biasanya menghasilkan CNTs dari sumber karbon yang boleh didapati secara komersil seperti etanol.

Pembuangan minyak masak yang telah digunakan menjadi masalah apabila peraturan sisa pepejal membatasi pelupusan cecair di tempat pembuangan sampah. Oleh itu pembuangan haram timbul termasuk pembakaran terbuka yang menyebabkan asap hitam, menuangkan ke parit yang menyebabkan sistem pembetung tersumbat dan akhirnya membawa kepada keadaan yang tidak bersih. Semua ini sangat mengganggu ekologi alam sekitar, kehidupan marin dan membawa kepada pemanasan global. Oleh itu, kami telah menemui kaedah baru untuk menghasilkan CNTs kapas secara pukal menggunakan minyak masak terpakai sebagai sumber karbon. Objektif tesis ini adalah untuk mensintesis CNTs menggunakan minyak masak terpakai sebagai sumber karbon melalui kaedah FCCVD.

Tesis ini menggunakan kaedah tunggal katalis apungan pemendapan wap kimia (FCCVD), larutan hidrokarbon cecair terdiri daripada minyak masak terpakai dan etanol (CH_3CH_2OH) sebagai sumber karbon, ferrocene ($C_{10}H_{10}Fe$) sebagai pemangkin dan thiophene (C_4H_4S) sebagai promoter, argon (Ar) hidrogen (H_2) sebagai pembawa gas. Larutan hidrokarbon cecair disuntikkan ke dalam relau panas bersama dengan

hidrogen/argon sebagai gas pembawa. Apabila memasuki relau, sebatian ini pecah dan bertindak balas dengan cepat untuk membentuk karbon nanotiub, yang kemudiannya berinteraksi untuk membentuk udaragel berbentuk silinder yang berterusan yang dikumpulkan pada akhir tindak balas tiub. Dalam tesis ini, pengeluaran CNTs bergantung kepada pelbagai parameter proses seperti kepekatan thiophene, kadar alir larutan hidrokarbon cecair, kepekatan ferrocene, dan nisbah gas Ar:H_2 telah diselidiki. Morfologi dan struktur karbon nanotiub multi dinding (MWCNTs) yang dihasilkan dicirikan menggunakan Mikroskop elektron imbasan medan pancaran (FESEM), Mikroskop transmisi elektron resolusi tinggi (HRTEM), Analisis Thermogravimetri (TGA), Spektroskopi fotoelektron sinar-X (XPS) dipelajari.

Keputusan menunjukkan bahawa penambahan 1.0 wt% thiophene menggunakan minyak masak terpakai menyebabkan peningkatan besar pada jumlah kapas CNTs yang diperoleh berbanding dengan kepekatan lain pada 1150 ° C. Nisbah Ar:H_2 yang 400: 300 sccm, menggunakan minyak masak terpakai sebagai sumber karbon dapat menyebabkan lebih banyak penggunaan energi, sementara itu membantu mencapai tingkat pertumbuhan yang tinggi dan CNTs yang lurus, kerana semakin banyaknya pendahulunya karbon langsung. Oleh kerana kepekatan ferrocene meningkat dengan menggunakan etanol sebagai sumber karbon, menyebabkan kluster Fe menjadi lebih besar untuk pertumbuhan CNTs. Kepekatan 1.0 wt% dan 1.5 wt% ferrocene memberikan struktur morfologi yang baik dan sifat yang lebih baik. Akhir sekali, pada masa reaksi yang tinggi, iaitu kadar aliran hidrokarbon 5 ml / j cecair dengan menggunakan etanol sebagai sumber karbon, boleh menyebabkan sumber karbon yang berlebihan dan pengumpulan produk sampingan yang membawa kepada struktur hibrid dipanggil graphene nanotub karbon karbon. Kesimpulannya, hasil eksperimen menunjukkan bahawa CNTs telah berjaya disintesis. Oleh itu, kami memberikan idea alternatif untuk penggunaan minyak masak terpakai untuk produk yang boleh digunakan untuk pelbagai aplikasi pada masa akan datang.

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I certify that a Thesis Examination Committee has met on 27 December 2017 to conduct the final examination of Noor Lyana binti Adnan on her thesis entitled "Synthesis and Characterization of Carbon Nanotube from Waste Cooking Oil using Floating Catalyst Chemical Vapour Deposition Method" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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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:

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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
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LIST OF ABBREVIATIONS

CNTs	Carbon nanotubes
CNFs	Carbon nanofibers
FCCVD	Floating catalyst chemical vapour deposition
MWNTs	Multi wall nanotubes
SWNTs	Single wall nanotubes
DWNTs	Double wall nanotubes
G-CNTs	Graphenated carbon nanotubes
CVD	Chemical vapour deposition
SDS	Sodium dodecyl sulfate
PVA	Polyvinyl alcohol
FESEM	Field Emission Scanning Electron Microscope
HRTEM	High Resolution Transmission Electron Microscope
TGA	Thermogravimetric analysis
XPS	X-ray photoelectron spectroscopy
nm	Nanometer
sccm	Standard cubic centimeters per minutes
eV	Electron volt
HiPco	High pressure carbon monoxide
Fe	Iron
VLS	Vapour-solid-liquid

CHAPTER 1

INTRODUCTION

1.1 Overview

Investigating on new innovation is drawing consideration of scientists around the world. Research are being done to enhance the properties of the materials and to find elective forerunners that can give productive properties of the materials. Nanotechnology, is one of the new innovations which allow to the improvement of structures, gadgets, and frameworks whose size shifts from 1 to 100 nanometers (nm) (Wu et al., 2014). Earlier decade has seen the improvement in each side of nanotechnology, for example, powders, nanoparticles, nanolayers and coats, electrical optic mechanical nanodevices and nanostructured organic materials. Nanotechnology is relied upon to be critical for years from now, in all fields of science and innovation.

Since the disclosure of buckminsterfullerene, carbon nanotubes (CNTs), and carbon nanofibers (CNFs), carbon nanostructure materials are getting to be broad business significance with enthusiasm developing quickly (Chen et al., 2000). The most understood materials in the primary rank of insurgency in nanotechnology were CNTs and CNFs. The staggering properties of these structures are their mechanical, electronic, synthetic and optical attributes, which open a route for up and coming applications. Numerous method to synthesize CNTs have been developed to produce bulk production such as laser (Melezhyk et al., 2013), Electric Arc Discharge (Feng et al., 2014), and Chemical Vapor Deposition (CVD) (Samant et al., 2007). CVD is more encouraging and cheaper method for synthesizing bulk CNTs, for large scale production.

Most of the CNTs has been synthesized from source that were based on fossil fuel such as methane, acetylene, benzene and xylene (Teo et al., 2003). These sources are lessening in several decades time. Furthermore, the cost of these raw materials is predictable to rise in the future. Therefore, it is necessary to look for different source. Recently, the use of bio-hydrocarbon source such as neem oil, camphor oil, turpentine oil, eucalyptus oil, castor oil, palm oil, waste cooking oil, and coconut oil have been reported (Kumar et al., 2011). The main aspect to utilizing plant based source as carbon source is its continuous feature which acts as renewable and cheap raw materials for bulk and extensive CNTs production.

1.2 Problem Statement

40,000 tonnes per year of waste cooking oil was estimated to produced in Asia countries such as Indonesia, Malaysia, Thailand, China, etc. (Hanisah et al., 2013). Inappropriate waste management of waste cooking oil prompts release to condition and

this will cause ecological contamination. Also, the blend of oil and water expands the chemical oxygen demand of water and makes it be dangerous as a result of the nearness of oil debasement side-effects. Carcinogenic compounds are absorbed by the sea creatures and will returned to human through food chain (Wu et al., 2014). Although waste cooking oil is known to be a carcinogenic element, it can be used as value-added products such as biodiesel, and as carbon source for carbon nanotubes. Recycling of waste cooking oil as carbon source for CNTs could provide a solution to solve this problem.

1.3 Objectives

The main objectives of this research is to synthesize carbon nanotubes from waste cooking oil as carbon source via floating catalyst chemical vapour deposition method. Also, as a preliminary study, the other objective of this research is to study the properties of carbon nanotubes from waste cooking oil. Below are the work phases to achieve the objective of this research:

1) To prepare waste cooking oil and ethanol as carbon source in liquid hydrocarbon solution for synthesis of CNTs cotton via floating catalyst chemical vapour deposition method.
2) To synthesize and study CNTs cotton from (1) above with variation of synthesis parameters.
3) To study the effect of parameters such as catalyst concentration, liquid hydrocarbon flow rate, promoter concentration and carrier gas ratio to the quality of carbon nanotubes from waste cooking oil and ethanol as carbon source.

Thus, according to main objectives, this study hypothesized, synthesized CNTs from waste cooking oil as carbon source by floating catalyst CVD method would be successful. The preparation of liquid hydrocarbon solution with right amount of carbon source (waste cooking oil and ethanol) will able to synthesize CNTs cotton by variation of parameters. By increasing catalyst concentration, liquid hydrocarbon flow rate, promoter concentration and carrier gas ratio from waste cooking oil and ethanol as carbon source would enhance the properties and structure of CNTs cotton.

1.4 Limitation Study

This research work will be carried out as a proof of concept recycling of waste cooking oil into nanotechnological materials which is carbon nanotubes (CNTs) in the bulk structure of cotton. Ethanol will also be used as comparison. Variation of synthesis parameters will be carried out and their effect on the quality of CNTs produced will be studied.

The quality and performance of CNTs material are highly dependent on the synthesis parameters. Moreover, when creating bulk macrostructures from these nanomaterials,

distribution, alignment and uniformity will drastically alter the mechanical and electrical properties of the material. This research will not deal with application of the synthesized product.

1.5 Thesis Outline

This thesis contained of five chapters, will discussed as follows: Chapter 1 includes background overview of CNTs synthesize using floating catalyst chemical vapor deposition method. The problem statement and objectives of the research work are mentioned, followed by the limitation study of the research.

Chapter 2 discusses the history of CNTs structure and properties, a summary of production of bulk CNTs using CVD and other method. It also explains numerous parameters that influence the production of CNTs and the thermophoretic phenomenon become the ultimate effect to synthesize bulk CNTs cotton were elaborated. In addition, this research also provides an initiative to utilize waste material into bulk CNTs cotton by using waste cooking oil and the detailed about waste cooking oil as carbon source were discussed. Finally, the potential applications of bulk CNTs were elaborated.

Chapters 3 discuss the detailed research methodology of CNTs production using floating catalyst chemical vapour deposition method. In addition, the method preparing liquid hydrocarbon solution consists of carbon source, catalyst and promoter for production CNTs are mentioned in this chapter follow up with the characterization of CNTs analysis.

Chapter 4 The results are mainly focused on the variation of process parameters on CNTs synthesis and detail characterization of CNTs. First discussion is the effect of thiophene ratio on the growth of CNTs from waste cooking oil, secondly the critical role of argon: hydrogen ($Ar:H_2$) gas ratio on CNTs from waste cooking oil was also discussed. Third, the effect of ferrocene ratio on the structure of CNTs cotton. Lastly, the effect liquid hydrocarbon flow rate on hybrid G-CNTs cotton formation and suggested mechanism will be discussed.

Chapter 5 In this chapter, production of CNTs by using floating catalyst chemical vapour deposition using waste cooking palm oil as carbon source and significant discussions of the process, structure and properties will be reported. A few suggestions were listed to provide a better research for future. The benefit of replacing ethanol to waste cooking oil as carbon source is that it can be more economical and environmental friendly thus, it can became wealth creation through industrial production of highly demanded technological devices, creation of new industries and life-quality enhancement via green technology adoptions.

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