



**PRODUCING LOW TEMPERATURE GRAPHITIC  
COMPOUND FROM OIL PALM TRUNK WASTE**

by

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## TABLE OF CONTENTS

	<b>PAGE</b>
<b>DECLARATION OF THESIS</b>	<b>i</b>
<b>ACKNOWLEDGMENT</b>	<b>ii</b>
<b>TABLE OF CONTENTS</b>	<b>iii</b>
<b>LIST OF TABLES</b>	<b>vii</b>
<b>LIST OF FIGURES</b>	<b>viii</b>
<b>LIST OF ABBREVIATIONS</b>	<b>xiii</b>
<b>LIST OF SYMBOLS</b>	<b>xiv</b>
<b>ABSTRAK</b>	<b>xv</b>
<b>ABSTRACT</b>	<b>xvi</b>
<b>CHAPTER 1 : INTRODUCTION</b>	<b>1</b>
1.1 Research Overview	1
1.2 Problem Statement	3
1.3 Objectives	5
1.4 Scope of Research	6
1.5 Thesis Outline	7
1.6 Significance of Study	8
<b>CHAPTER 2 : LITERATURE REVIEW</b>	<b>9</b>
2.1 Carbon Derivatives	9
2.2 Graphite	10
2.2.1 Synthetic Graphite	11
2.2.2 Synthesizing synthetic graphite	12

2.2.3	Heat Treatment	15
2.3	Pyrolysis Process	16
2.3.1	Types of Pyrolysis Process	17
2.3.1.1	Slow Pyrolysis	18
2.3.1.2	Flash Pyrolysis	18
2.3.1.3	Fast Pyrolysis	18
2.3.2	Advantages of Pyrolysis Process	19
2.4	Manufacturing of carbon based product from waste	19
2.5	Characterization of Synthetic Graphite and Graphene Oxide	20
2.5.1	X-Ray Diffraction (XRD) Analysis for Graphitic Matter	20
2.5.2	RAMAN Analysis for Graphitic Nature	22
2.5.3	Scanning Electron Microscope (SEM) Analysis for Graphitic Structure	26
2.5.4	Thermogravimetry (TGA) Analysis for Graphitic Phase	28
2.5.5	Fourier Transform Infra-Red (FTIR) Analysis of Graphitic Nature	28
2.6	Oil Palm History and Industry	32
2.7	Oil Palm Biomass Waste	36
2.8	Chemical Composition of Oil Palm Biomass Waste	39
2.9	Oil palm trunk biomass waste	41
2.10	Emerging industries on oil palm trunk biomass	44
2.11	Anatomy of Oil Palm Trunk	45
2.11.1	Vascular Bundles	46
2.11.2	Parenchyma Tissues	49
2.12	Summary	51
	<b>CHAPTER 3 : MATERIALS &amp; METHODOLOGY</b>	<b>52</b>
3.1	Introduction	52
3.2	Experimental Design	52

3.3	Raw Material, Chemicals and Equipment	54
3.3.1	Raw Material	54
3.3.1.1	Raw Material Selection	54
3.3.2	Chemicals	56
3.3.3	Equipment	56
3.4	Synthesis and Characterization	57
3.4.1	Synthesis of synthetic graphite from Oil Palm Trunk Waste	57
3.4.2	Characterization of Synthetic Graphite Produced	60
3.4.3	Exfoliation of Synthetic Graphite via Modified Hummers Method	61
3.4.3.1	Pre-Oxidized Graphite	61
3.4.3.2	Modified Hummers Method	64
3.4.3.3	Characterization of Reduced Graphene Oxide Produced	66
3.5	Synthetic Graphite and Reduced Graphene Oxide Analysis	66
3.5.1	X-Ray Diffraction (XRD) Machine	66
3.5.2	RAMAN Spectroscopy Basics	68
3.5.3	Scanning Electron Microscope (SEM)	70
3.5.4	Thermogravimetry (TGA) Analysis	71
3.5.5	Transmission Electron Microscopy (TEM)	72
3.5.6	Fourier Transform Infra- Red (FTIR)	73
3.6	Summary	74
<b>CHAPTER 4 : RESULTS AND DISCUSSIONS</b>		<b>75</b>
4.1	Introduction	75
4.2	Raw Materials Selection	76
4.3	Characterization of synthetic graphite produced from oil palm trunk waste	78
4.3.1	X-Ray Diffraction (XRD) Analysis	79
4.3.2	RAMAN Analysis	87

4.3.3	Morphological analysis by Scanning Electron Microscopy (SEM)	94
4.3.4	Thermogravimetry (TGA) Analysis	103
4.4	Synthetic Graphite from Oil Palm Trunk Waste	112
4.5	Density Measurement of Synthetic Graphite	113
4.6	Further Exfoliation of Synthetic Graphite for Graphene Oxide Production via Modified Hummers Method	114
4.6.1	X-Ray Diffraction (XRD) Analysis of Graphene Oxide	114
4.6.2	RAMAN Analysis	115
4.6.3	Transmission Electron Microscope (TEM) Analysis	117
4.6.4	Fourier Transform Infra- Red (FTIR) Analysis	120
4.7	Summary	125
<b>CHAPTER 5 : CONCLUSION AND RECOMMENDATIONS</b>		<b>126</b>
5.1	Conclusion	126
5.2	Recommendations	127
<b>REFERENCES</b>		<b>129</b>
<b>APPENDICES</b>		<b>140</b>

## LIST OF TABLES

NO.		PAGE
Table 2.1	A short comparison of graphite production by heat treatment	15
Table 2.2	A short comparison of graphite production by heat treatment (continued)	16
Table 2.3	Carbon based product manufacture from waste	20
Table 2.4	Regions of the Infra Red Spectrum (Pfaffeneder-kmen et al., 2017)	31
Table 2.5	Typical vibrational frequency of functional groups (Petit et al., 2018)	32
Table 2.6	Manufactured products from oil palm trunk waste	45
Table 4.1	Three different yield after heat treatment from outer, middle and inner part of oil palm trunk waste	77
Table 4.2	Density of commercial graphite and synthetic graphite produced oil palm trunk waste	113
Table 4.3	Typical vibrational frequency of functional groups in commercial graphite	121
Table 4.4	Typical vibrational frequency of functional groups presence in the synthetic graphite produced from oil palm trunk waste	122
Table 4.5	Typical vibrational frequency of functional groups presence in the graphene oxide produced from synthetic graphite synthesized from oil palm trunk waste	123

## LIST OF FIGURES

NO.		PAGE
Figure 1.1	Graphene (top left) shown as a honeycomb lattice of carbon atoms. While, graphite (top right) can be viewed as a stack of graphene layers. Carbon nanotubes can be viewed as rolled-up cylinders of graphene (bottom left). Fullerenes in the other hand (C60) are molecules consisting of wrapped graphene through the introduction of pentagons on the hexagonal lattice (Kulla et al., 2012)	1
Figure 2.1	Structure of graphite (Soldano, Mahmood, & Dujardin, 2010)	10
Figure 2.2	Typical X-ray diffraction pattern for graphitic material (Bannov et al., 2018)	22
Figure 2.3	X-ray diffraction pattern for graphite (Li et al., 2007)	22
Figure 2.4	RAMAN Spectroscopy of graphitic nature (Merlen et al., 2017)	26
Figure 2.5	Waste from oil palm plantation. (A) Oil palm trunks (OPT) and (B) Oil palm fronds (OPF) (Awalludin et al., 2015)	39
Figure 2.6	Waste from palm oil mills. (A) Empty fruit bunches (EFB), (B) Mesocarp fiber and (C) Palm kernel shells (Awalludin et al., 2015)	39
Figure 2.7	Chemical composition of oil palm biomass (Gogoi et al., 2017)	41
Figure 2.8	Availability of dry weight oil palm biomass in Malaysia in 2009 (Mn/ T <sup>1</sup> / <sub>4</sub> Million Tonnes) (Abdullah & Sulaiman, 2013)	43
Figure 2.9	Chemical composition in oil palm trunk biomass (Gogoi et al., 2017)	44
Figure 2.10	Different region in the oil palm trunk with different density (Bakar et al., 2017)	48

Figure 2.11	Figure showing the ground parenchyma and vascular bundle (Bakar et al., 2017)	48
Figure 2.12	Vascular bundles and Parenchyma Tissue (Hanafi et al., 2014)	49
Figure 3.1	Flowchart of the experimental design	53
Figure 3.2	Chopped oil palm trunk waste	55
Figure 3.3	Outer area, middle area and inner area	55
Figure 3.4	Oil palm trunk chip	57
Figure 3.5	Tube furnace	58
Figure 3.6	Oil palm trunk charcoal	59
Figure 3.7	Grinded synthetic graphite	59
Figure 3.8	Densimeter	60
Figure 3.9	Chemicals used to pre oxidized graphite	62
Figure 3.10	Production of pre-oxidized graphite	63
Figure 3.11	Oxidation processes of graphite via Modified Hummers method	65
Figure 3.12	BRUKER D2 Phaser XRD machine	67
Figure 3.13	XRD powder specimen holder	68
Figure 3.14	RAMAN Spectroscopy machine	69
Figure 3.15	JEOL JSM-6460LA scanning electron microscope (SEM)	70
Figure 3.16	Themogravimetry (TGA) machine	71
Figure 3.17	Transmission Electron Microscope (TEM) machine	72
Figure 3.18	Fourier Transform Infra Red Spectrum 65 (Perkin Elmer)	74
Figure 4.1	XRD pattern of commercial graphite	80

Figure 4.2	XRD pattern of Raw OPT	80
Figure 4.3	Comparison of diffraction pattern at 300°C heating temperature at various heating rate	81
Figure 4.4	Comparison of diffraction pattern sample heated at 500°C at various heating rate	83
Figure 4.5	Comparison of diffraction pattern for various heating rate at 800°C heating temperature.	84
Figure 4.6	Comparison of diffraction pattern for various heating rate at 1000°C heating temperature	86
Figure 4.7	RAMAN spectroscopy of commercial graphite (Merlen et al., 2017)	88
Figure 4.8	RAMAN spectroscopy for sample heated at heating temperature of 300°C at various heating rate.	89
Figure 4.9	RAMAN spectroscopy for sample heated at heating temperature of 500°C at various heating rate.	91
Figure 4.10	RAMAN spectroscopy for sample heated at heating temperature of 800°C various heating rate.	92
Figure 4.11	RAMAN spectroscopy for sample heated at heating temperature of 1000°C at various heating rate	94
Figure 4.12	Commercial graphite with different magnification	95
Figure 4.13	Sample heated at 300°C at various heating rate which is 5°/min, 10°/min and 20°/min	97
Figure 4.14	Sample heated at 500°C with different heating rate which is 5°/min, 10°/min and 20°/min	99
Figure 4.15	Sample heated at 800°C at different heating rate which is 5°/min, 10°/min and 20°/min	101

Figure 4.16	Sample heated at 1000°C at different heating rate which is 5°/min, 10°/min and 20°/min	102
Figure 4.17	TGA of commercial graphite	103
Figure 4.18	TGA Analysis of Raw Oil Palm Trunk	104
Figure 4.19	TGA Analysis of sample heated at 300 at different heating rate	106
Figure 4.20	TGA analysis sample heated at the heating temperature of 500°C at different heating rate	108
Figure 4.21	TGA analysis sample heated at the heating temperature of 800°C at different heating rate.	110
Figure 4.22	TGA analysis sample heated at the heating temperature of 1000°C at different heating rate	112
Figure 4.23	XRD analysis of reduced graphene oxide produce by Modified Hummers Method	115
Figure 4.24	RAMAN analysis of graphene oxide produce from synthetic graphite synthesized from oil palm trunk waste	116
Figure 4.25	RAMAN Spectroscopy of Graphene Oxide Produced From Commercial Graphite	117
Figure 4.26	TEM Images at 1µm	118
Figure 4.27	TEM images at 200 nm	119
Figure 4.28	TEM Images of GO at 100nm	119
Figure 4.29	FTIR Spectrum of commercial graphite	120
Figure 4.30	FTIR Spectrum of Synthetic Graphite produced from Oil Palm Trunk Waste	121
Figure 4.31	Comparison of FTIR Spectrum of Synthetic graphite and Commercial Graphite	122

Figure 4.32	FTIR spectrum of reduced graphene oxide	123
Figure 4.33	Comparison of FTIR Spectrum between synthetic graphite, commercial graphite and graphene oxide	124

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## LIST OF ABBREVIATIONS

XRD	X-Ray Diffraction
FTIR	Fourier Transform Infra -Red
TGA	Thermogravimetry Analysis
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscope
OPT	Oil Palm Trunk
OPF	Oil Palm Frond
EFB	Empty Fruit Bunch
PKS	Palm Kernel Shell
POME	Palm Oil Mill Effluent
MF	Mesocarp Fiber
OPL	Oil Palm Leaves

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## LIST OF SYMBOLS

$\Theta$	Theta ( diffraction angle)
$\tau$	Scherrer equation
$\lambda$	wavelength
$\beta\tau$	Broadening of the reflection due to small crystallite sizes
$\Gamma$	$\Gamma$ point of the Brillouin zone
$\Delta E$	Vibrational energies

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## Penghasilan Sebatian Grafit Pada Suhu Rendah Dari Sisa Batang Kelapa Sawit

### ABSTRAK

Karbon mempunyai pelbagai *allotropes* atau polimof yang berbeza dari segi susunan atom. Grafit, grafin, fulleren dan berlian adalah contoh *allotropes* atau polimof karbon yang berbeza dari segi susunan atom. Grafin sangat terkenal dan mendapat perhatian yang hangat dalam bidang elektronik, opto-elektronik, kimia sensor, nano komposit dan penyimpanan tenaga. Walaubagaimanapun, grafin ini terhasil dari proses kimia grafit. Terdapat dua jenis grafit iaitu grafit semulajadi dan grafit buatan atau sintetik. Kajian terdahulu menunjukkan grafit sintetik dihasilkan pada suhu pemanasan yang sangat tinggi iaitu pada suhu melebihi 2500°C melalui proses yang sangat kompleks. Penemuan ini ditemukan oleh seorang saintis yang bernama *Acheson*. Sehingga kini, proses untuk menghasilkan grafit sintetik masih mengekalkan nama proses *Acheson*. Kajian terdahulu menunjukkan grafit sintetik diperbuat dari bahan seperti *petroleum coke*, tar, dan juga antrakit pada suhu pemanasan yang sangat tinggi. Walaubagaimanapun, pada kajian ini, melalui proses yang diberi nama pirolisis, dalam keadaan pemanasan yang terkawal, dan kadar pemanasan yang spesifik, grafit sintetik berjaya dihasilkan pada suhu pemanasan yang jauh lebih rendah dengan menggunakan sisa batang kelapa sawit sebagai bahan primer. Pada kajian ini, suhu pemanasan di variasikan kepada 300°C, 500°C, 800°C, 1000°C dan 1200°C. Manakala, kadar pemanasan pula di variasikan kepada 5°/min, 10°/min and 20°/min. Selepas pemanasan, sampel telah di analisis kepada beberapa analisis melalui *X-Ray Diffraction (XRD) analysis*, *RAMAN analysis*, *Scanning Electron Microscope (SEM) analysis*, *Thermogravimetry (TGA) Analysis*, and *Fourier Transform Infra-Red (FTIR) Analysis*. Keputusan yang diperolehi daripada analisa tersebut, dibandingkan dengan komersil sintetik grafit. Daripada perbandingan tersebut, parameter yang paling terbaik untuk menghasilkan grafit sintetik dari sisa batang kelapa sawit adalah pada suhu pemanasan 800°C dan kadar pemanasan 20°/min. Hal ini adalah kerana melalui analisa *XRD* yang menunjukkan darjah 26° pada 2 $\theta$ . Keputusan ini di sokong oleh analisa *RAMAN* yang menunjukkan kehadiran puncak D, G dan 2D pada 1250 cm<sup>-1</sup>, 1625cm<sup>-1</sup> dan 2700 cm<sup>-1</sup> masing-masing. Analisa morfologi oleh *SEM* juga menunjukkan pembentukan kepingan grafit sintetik. Analisa oleh *TGA* juga menunjukkan pengurangan berat ketika pertambahan suhu pemanasan sebanyak hanya 10%. Analisa oleh *FTIR* juga menunjukkan kehadiran kumpulan berfungsi yang sama di bandingkan dengan grafit komersil. Grafit sintetik yang berjaya di hasilkan pada suhu 800°C pada kadar pemanasan 20°/min ini seterusnya menjalani proses pengupasan melalui kaedah ubah suai *Hummers* untuk menghasilkan grafin oksida. Grafin oksida ini seterusnya di analisa dengan menggunakan *XRD*, *RAMAN*, *FTIR*, dan *TEM* untuk di bandingkan dengan grafin oksida yang terhasil dari grafit komersil.

## Producing Low Temperature Graphitic Compound from Oil Palm Trunk Waste

### ABSTRACT

Carbon has many allotropes or polymorphs which differ in the arrangement of atom. Graphite, graphene, fullerenes and diamond are common example of carbon allotrope. Graphene receive a great impact on electronic and optoelectronic devices, chemical sensors, nano-composites and energy storage. However, the graphene is derived from the chemical processes and treatment of graphite. There are two types of graphite, which are, natural graphite and synthetic graphite. Previously, synthetic graphite was produced under higher heating temperature, which is above 2500°C in complex processing method, which bear the name of the scientist whose discover the synthetic graphite namely “Acheson Process”. Previously, synthetic graphite was produced under higher heating temperature and by using petroleum coke, anthracite, and coal tar pitch as the pre cursor raw materials. However, in this study, via pyrolysis process, in controlled heating condition, and specific heating rate by utilizing oil palm trunk waste, synthetic graphite was manage to produce at much lower heating temperature. In this study, the heating temperature was varied in five heating temperature series, which are, 300°C, 500°C, 800°C, 1000°C and 1200°C. The heating rate applied was also varied in 3 different series, which are, 5°/min, 10°/min and 20°/min. After the heating treatment, the sample was characterized by different analysis tools, including, X-Ray Diffraction (XRD) analysis, RAMAN analysis, Scanning Electron Microscope (SEM) analysis, Thermogravimetry (TGA) Analysis, and Fourier Transform Infra-Red (FTIR) Analysis. The results obtained for produced synthetic graphite was compared with the commercial graphite. From the conducted characterization process, among all parameters varied, the best parameter to produce synthetic graphite from oil palm trunk waste was at the heating temperature of 800°C and with the heating rate of 20°/min. XRD analysis of the synthetic graphite produced at 800 °C, 20°/min, shows the significant graphite peak in XRD diffraction pattern at 26 ° in 2 $\theta$ , which is comparable with the commercial synthetic graphite. This was further supported with RAMAN analysis as to confirm the graphitic nature of the synthetic graphite produced. RAMAN spectroscopy shows the graphitic nature of the graphite obtained in the presence of D, G and 2D peaks at 1250 cm<sup>-1</sup>, 1625 cm<sup>-1</sup> and 2700 cm<sup>-1</sup> respectively. From the morphological characterization conducted by scanning electron microscope (SEM), the formation of graphite flakes also formed for the sample heated at 800 °C at 20°/min which comparable with the commercial synthetic graphite. Thermogravimetry (TGA) analysis also show the 10 % of percentage weight loss as a function of increasing temperature for sample heated at 800 °C, 20°/min. Fourier transform Infra-Red (FTIR) analysis also show existence of functional group of graphite. The synthetic graphite obtained from the best parameters was further exfoliated by using Modified Hummers Method to produce reduced graphene oxide. The reduced graphene oxide produced was further characterized by using XRD, RAMAN, FTIR and TEM.

## CHAPTER 1 : INTRODUCTION

### 1.1 Research Overview

Carbon is one of the most widely used groups of materials in nearly every field of science and engineering. It is one of the important elements that most widely studied and applied in various adsorbent materials, battery, fuel cell electrodes, and supercapacitors. Carbon has many allotropes or polymorphs which differ in the arrangement of atom. Graphite, graphene, fullerenes and diamond are common example of carbon allotrope. The differences in the arrangement of atom in allotropes of carbon can be shown in Figure 1.1

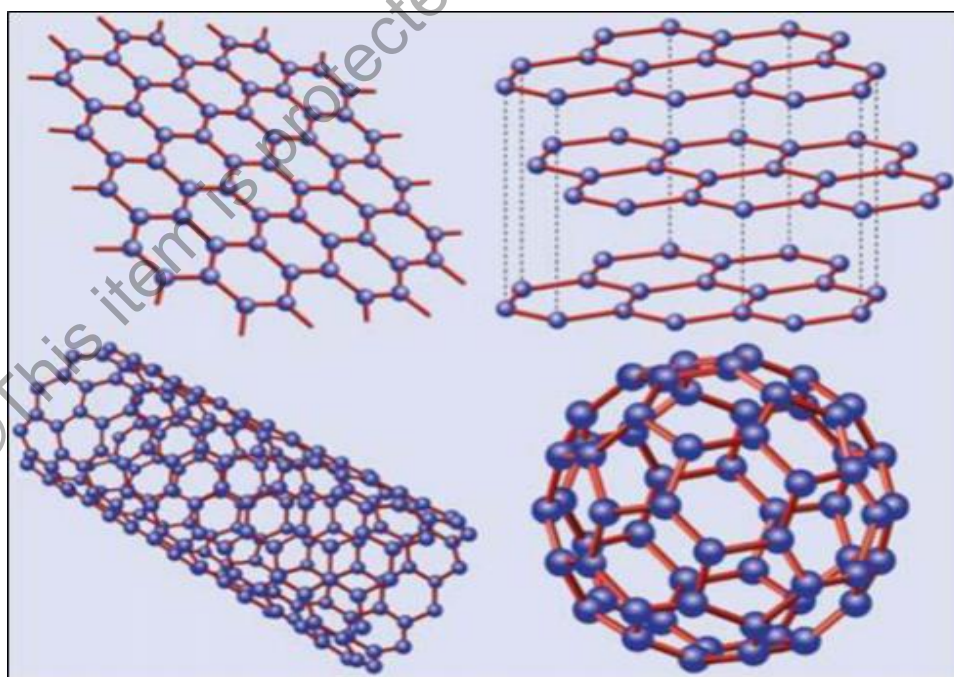


Figure 1.1 Graphene (top left) shown as a honeycomb lattice of carbon atoms. While, graphite (top right) can be viewed as a stack of graphene layers. Carbon nanotubes can be viewed as rolled-up cylinders of graphene (bottom left). Fullerenes in the other hand (C<sub>60</sub>) are molecules consisting of wrapped graphene through the introduction of pentagons on the hexagonal lattice (Kuila et al., 2012)

More recently graphene receive a great impact on energy storage, chemical sensors, nano-composites, and optoelectronic and electronic devices. However, these super great graphene derive from the chemical processes and treatment of graphite. There are two types of graphite which are natural graphite and synthetic graphite. History of natural graphite begin in metamorphic rocks as a result of the reduction of sedimentary carbon compounds during metamorphism. Furthermore, natural graphite also occurs in the igneous rocks and in meteorites. There are some minerals associated with graphite, which include quartz, micas and tourmaline. In meteorites it occurs with troilite and silicate minerals. Small graphitic crystals in meteoritic iron are called cliftonite (Kottegoda et al., 2015). Over the past few years, synthetic graphite have attracted increasing attention and have been considered in many graphene production field. Literally, synthetic graphite is a substance synthesized from the high temperature processing of amorphous carbon materials. The types of amorphous carbon used as precursors to synthesize graphite are various, such as, it can be derived from petroleum, coal, or natural and synthetic organic materials. However, in some cases graphite can even be manufactured by the direct precipitation of graphitic carbon from pyrolysis of a carbonaceous gas such as acetylene (pyrolytic graphite). The key similarities between all graphite precursors is that they must contain carbon. Graphite is one type of carbon, a specific form of carbon, so it can only be derived from other carbon containing substances (Tamashausky, 2006).

Graphite either it is natural or synthetic, it is recognized as the primary source for graphene materials preparation by the top down technology. Previously, the preparation of synthetic graphite always requires temperatures above 2500°C which represents a high energy cost (Sierra et al., 2015). As reported by previous research, synthetic graphite was discovered by accident during the late 1800's by Edward Goodrich Acheson. While striving to manufacture silicon carbide (Carborundum) in an electric furnace from a combination of silica and amorphous carbon, Acheson discover unintentional reaction product, graphite crystals, was also formed. In fact, one of the furnace types still used to manufacture graphite, as well as the process method still bears Acheson's name: the Acheson furnace and the Acheson process (Tamashausky, 2006). Nevertheless, the graphitization of the synthetic graphite produced still occurs during heat treatment at temperatures often in excess of 2500°C. Thus, in this study, we attempted to use oil palm trunk waste as a carbon source for synthetic graphite production possibly to graphitize at the lower heating temperature below 1000°C.

## **1.2 Problem Statement**

In Malaysia, the oil palm cultivation has started almost a century ago. Oil Palm trees was introduced into Malaysia in 1870 through the Singapore Botanic Gardens. However, during that time, oil palm trees were planted as a decorative plant, before deliberately being used as national crop. (Othman Sulaiman et al., 2012). Nowadays Malaysia is facing a vigorous development of new oil palm plantations and palm oil mills. Significantly, oil palm has become the most important agricultural crop in Malaysia and has been the backbone to the national economic expansion (Wan Asma, Mahanim, Zulkafli, Othman, & Mori, 2010). Malaysia is one of the countries that

successfully retained the beneficiaries of the oil palm industry and being responsible for uprising this industry globally through significant contributions and continuous commitment. For decades, Malaysia has retained the title as one of the most productive palm oil producers in the world (Umar, Jennings, & Urme, 2013). This commodity plays a vital role in the Malaysia economic growth. Oil palm plantation in the country become numerous and can be found everywhere. In spite of that, as the industry become bigger and wider a huge amount of oil palm wastes is generated and lead to problem of biomass waste overload (Hosseini & Wahid, 2014). This problem finally will burden the operators with disposal difficulties and escalates the operating cost. The wastes such as oil palm trunks (OPT), palm kernel shell (PKS), empty fruit bunches (EFB), oil palm fronds (OPF) , mesocarp fiber (MF), palm oil mill effluent (POME), and oil palm leaves (OPL) are generated after the oil palm fruits harvesting, palm oil processing or during oil palm trees replantation (Hafi, Mat, & Johari, 2006). During the present time, due to the realisation of oil palm waste's potential for producing various useful resources, palm trunk waste is attempted to use as carbon source for synthetic graphite production.

Formerly, negative-cost (money losing) materials are now being processed into positive-earning (money earning) materials (Pogaku & Hj. Sarbatly, 2014). In other hands, waste has been converted into wealth. During earlier time, synthetic graphite was synthesized by using silica and other amorphous carbon at excess 2500°C graphitization or heating temperature. However, as far as research is concern, innovation towards methods and primary materials of the synthetic graphite obtained is emphasized. It is by utilizing the oil palm trunk waste as the primary materials in synthetic graphite production and lower down the graphitization or heating temperature

below 1000°C whereby the method implemented is pyrolysis or controlled heating condition.

### **1.3 Objectives**

The main focus of this research is to produce the synthetic graphite from oil palm trunk biomass waste via heat treatment method with lower graphitization temperature. Then, the synthetic graphite is exfoliated using chemical method to obtain reduced graphene oxide materials. The specific objectives of this research are summarized as follows:

1. To produce the synthetic graphite from oil palm trunk biomass waste with lower graphitization or heating temperature, which is, below 1000°C via pyrolysis process.
2. To characterize and study the properties of obtained synthetic graphite by using X-Ray Diffraction (XRD) Analysis, RAMAN Spectroscopy Analysis, Scanning Electron Microscope (SEM), and Thermogravimetric (TGA) Analysis
3. To further exfoliate the synthetic graphite for synthesizing reduced graphene oxide and characterize the reduced graphene oxide produced via Modified Hummers Method; by using X-Ray Diffraction (XRD) Analysis, RAMAN spectroscopy analysis and Transmission Electron Microscopy (TEM) Analysis and Fourier Transform Infra-Red (FTIR) Analysis.

## 1.4 Scope of Research

The scope of research basically cover the following areas:

1. Synthesize the graphitic compound from oil palm trunk waste at various heating temperature and heating rate. The heating temperature will be in the range of 300°C, 500°C, 800°C and 1000°C. Meanwhile the heating rate also varies at 5°/min, 10°/min and 20°/min.
2. Graphitic phase obtained from XRD is analysed using Xpert Highscore Plus Software.
3. SEM is used to analyse the morphological studies of the graphitic compound obtained.
4. Study the amount of weight change of a material, either as a function of increasing temperature, or isothermally as a function of time, in an atmosphere of nitrogen by using TGA analysis.
5. Graphitic nature of the synthetic graphite obtained as to support the XRD analysis is determined by RAMAN spectroscopy.
6. Reduced graphene oxide is produced from the graphitic compound obtained via Modified Hummers Method and further characterized the graphene oxide by using XRD, RAMAN, FTIR, and TEM.

## 1.5 Thesis Outline

The thesis is separated into five chapters. Chapter 1 introduces the research background, problem statement, research objective, scopes of study, and the outline of the thesis and significance of the study.

Chapter 2 discusses about the general information of carbon derivatives, graphite includes natural and synthetic graphite and also graphene. Literature review on pyrolysis processes also discussed. Summary on manufacturing carbon based product waste also being highlighted. In addition, history of oil palm industry and oil palm biomass waste specifically oil palm trunk biomass waste also reviewed. Emerging industries of oil palm trunk biomass waste also summarized in this section. Finally the study on anatomy of oil palm trunk also discussed in this chapter.

Chapter 3 explains the details of the materials used and the methodology applied in the research producing low temperature synthetic graphite from oil palm trunk waste for reduced graphene oxide exfoliation. This chapter also describes the characterization conducted to the produced synthetic graphite and reduced graphene oxide.

Chapter 4 presents the results and discuss on the findings from the characterization being conducted. The results obtained from each of the parameters applied will be compared to each other and to the commercially available synthetic graphite and graphene oxide.

Chapter 5 states the conclusions of this study and some recommendations for the future work. The thesis ends with a Reference List and several Appendices.

## **1.6 Significance of Study**

This research will be very imperative since it is emphasizing on utilization of oil palm trunk biomass waste to be transformed into high value product which is graphitic compound. Synthesizing the graphitic compound from oil palm trunk biomass waste will solve the problem on the tonnes of biomass waste left rotten and open combusted in the oil palm plantation. Avoiding open combustion of the oil palm trunk biomass waste will keep the environment clean without the air pollution. Apart from keeping the environment safe, this research is becoming more interesting since it is implementing the waste to wealth concept. The negative-cost (money losing) materials are now being processed into positive-earning (money earning) materials. Furthermore, the production of graphitic compound from oil palm trunk biomass waste in controlled heating condition also improved the existing method. The heating temperature applied in this research is very much lower which is below 1000°C and in a very simple heating technique which is pyrolysis process.