

**Activated Carbon from Empty Fruit Bunches (EFB)**

by

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the  
Chemical Engineering Programme  
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(CHEMICAL ENGINEERING)

Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2014

## CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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NORATIKAH BINTI SHAMSUDDIN

## ABSTRACT

EFB was proposed as raw materials for activated carbon production due to their large availability and has high amount of carbon content. Besides, EFB wastes can be recycled and turn into value added product. For this experiment, shaggy and core part of EFB were investigated separately as there were no investigation has been done for separated part of EFB (shaggy and core) before. This experiment aims to prepare activated carbon product from EFB and determining its characteristics with respect to different treatment process. Experiment was carried out by undergoes two main process; pyrolysis and activation. The temperature for pyrolysis varies from 400°C, 450°C and 500°C in nitrogen atmosphere. For the activation process, the temperature varies from 600°C, 700°C and 800°C with the present of 15%, 60% and 100% of CO<sub>2</sub>, holding time of 30 minutes, 60 minutes and 120 minutes. Experimental results show that activation temperature, CO<sub>2</sub> percentage and holding time have significant effects on yield and morphology of activated carbon produced. The optimum conditions for activation to produce highest yield of activated carbon is found to be at the lowest activation temperature (600°C), lowest CO<sub>2</sub> percentage (15%) and shortest holding time (30 minutes). While, the activated carbon produced from core part produce bigger cross section pore diameter (32.6µm) compared to shaggy part (30.2µm) after activated at the optimum condition of activation temperature 700°C, 60% of CO<sub>2</sub> and a holding time of 60 minutes.

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Hopefully this project will be a great contribution to the industry and the society.

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## **CHAPTER 1**

### **PROJECT BACKGROUND**

#### **1.1 Background of Study**

In the early, commercialized activated carbons were prepared from wood and peat. In fact, activated carbons were obtained from any carbonaceous materials. Basically, biomass is produced from organic materials. They were obtained either directly from plants or indirectly from domestic, industrial, commercial or agricultural product. There are two categories of biomass. Those are from woody biomass and non-woody biomass (Ai We, 2008). For woody biomass it comes from forest products, untreated wood products, energy crops and short rotation coppice. However, animal waste, industrial and biodegradable municipal product from food processing and high energy crops is categorized as non-woody biomass. Biomass is very vital feedstock as they are renewable sources and has low cost materials. Moreover, biomass has low ash content which is very suitable for creating high porous structures within the activated carbon matrix as well as having high concentration of volatiles.

In Malaysia, palm oil sector contributing about 80 million dry tonnes in 2010 of biomass resources. This amount is expected to increase about 100 million dry tonnes by 2020, primarily driven by increases in yield. As a result, the National Biomass Strategy 2020 is focusing on oil palm biomass as a starting point, which may later be extended to include biomass from other sources. This amount make biomass is abundant in our country due to high sunlight intensity/time and high rainfall (Singh, 2010).

In 2010, the oil palm planted area in Malaysia is 4.8 million hectares. In 2011, Malaysian oil palm accounted for just 1.97% which is about five million hectares of the total 253.9 million hectares. It makes up to 71% of agriculture land or 14.3% of total land area. The overall average of 18.03 tones Fresh Fruit Bunches (FFB) per hectare of palm oil plantation has been produced from the oil palm industry (Choo, 2011). Based on this figure, palm oil plantation areas has produced more than 66.63 million tonnes of biomass residues such as Empty Fruit Bunches (EFB), mesocarp fiber, shell, palm kernel cakes, trunks and Palm Oil Mill Effluent (POME) in 2010 (Goh *et al.*, 2009). The EFB represent about 9% of this total. In a country that has significant amount of agricultural activities, biomass can be a very promising alternative source of renewable energy.

In this study, empty fruit bunches (EFB) of oil palm has been chosen as the feedstock due to its abundance and low cost of processing. Besides, it also cleans, non-toxic and renewable.

## **1.2 Problem Statement**

Nowadays, activated carbon has a large potential to be commercialized. However, the depleting and price increasing of fossil fuel (coal) make EFB as the good source of raw materials to replace former source of activated carbon production due to its low cost. Large amount of EFB can be recycled and turn into value added products. Besides, the usage of fossil fuel can give impact to human health.

### **1.3 Objective of Study**

There are three objectives need to be achieved in this project. Those are:

1. To prepare activated carbon product from EFB.
2. To determine the effect of different carbonization temperature, carbon dioxide percentage, and activation time on the characteristics of activated carbon produced.
3. To study morphology and structure of the prepared activated carbon produced.

### **1.4 Scope of Study**

The scopes of this study for this project are identification of activated carbon by shaggy EFB and core EFB feedstock and characterization of product after undergo two stages of pre-treatment those are pyrolysis and activation.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Overview

##### 2.1.1 Biomass

Worldwide, biomass is the fourth largest energy resource after coal, petroleum and natural gas. Biomass is biological material from living organism, most often referring to plants or plant-derived materials. Plants use light energy from the sun to convert carbon dioxide and water to sugar through a photosynthesis process. It remains the largest biomass energy source today for example dead trees, branches, tree stumps, wood chips and even municipal solid waste.<sup>1</sup>

Another type of plant matter, called cellulosic biomass, is made up of very complex sugar, and it is not generally used for food. Cellulosic biomass consists of three main components which are lignin, hemicellulose and cellulose. Among these components, the largest portion is cellulose which covers from 38% to 50% followed by hemicellulose (23%-32%) and lignin (15%-25%).<sup>2</sup>

Another type of plant matter is called as lignocellulosic biomass. It derived from agricultural by-products and has a large potential to be a promising type of raw material for producing activated carbon, especially due to its availability at a low price.

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<sup>1</sup> Biomass Energy Centre. <http://www.biomassenergycentre.org.uk>. Retrieved on December 6, 2012

<sup>2</sup> Valentas.K.,(2009). Biofuel from Cellulosic Biomass: An Overview of Current Technologies & Economic Feasibility. *Biotechnology Institute, University of Minnesota*. pp 1-5.

Lignocellulosic biomass consists of three different components, which are cellulose, hemicellulose, and lignin. Among these components, the largest portion is cellulose which covers from 38% to 50% followed by hemicellulose (23%-32%) and lignin (15%-25%).<sup>3</sup> Lignin is the main component in lignocellulosic biomass responsible for the adsorption process. It is the most abundant renewable carbon resource on earth compared to cellulose, with a worldwide production of 40–50 million tons per year. Lignocellulosic biomass is a good option to be used as precursor for producing activated carbon due to the rich carbon content of lignin. There are numerous sources of activated carbon with high adsorption capacity such as durian shell, coconut shell, rubber-seed shell, hazelnut shell, palm kernel shell and wood.<sup>4</sup>

Utilization of lignocellulosic biomass to produce activated carbon is one of the good approaches as it can be considered as abundant agricultural wastes. This waste can be converted into value added product such as activated carbon which could solve environmental problems such as accumulation of agricultural waste, air pollution and water pollution. Moreover, by utilizing activated carbon from lignocellulosic biomass instead of fossil sources such as coal can slightly reduce global warming's effects. Therefore, the circulation of carbon between atmosphere and pollutant removal process is merely a carbon-neutral cycle. Besides that, lignocellulosic activated carbon is also economical because it is come from agricultural sector wastes and is abundantly available.

In 2010, the oil palm planted area in the country is 4.8 million hectares. In 2011, this area has increase to 1.97% which is about five million hectares of the total 253.9 million hectares. This value makes up to 71% of agriculture land.<sup>5</sup> The overall average of 18.03 tones Fresh Fruit Bunches (FFB) per hectare of palm oil plantation has been produced from the oil palm industry (Choo, 2011). Based on this figure, palm oil plantation areas

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<sup>3</sup> Valentas.K.,(2009). Biofuel from Cellulosic Biomass: An Overview of Current Technologies & Economic Feasibility. *Biotechnology Institute, University of Minnesota*. pp 1-5.

<sup>4</sup> Nor, N. M., Chung, L. L., Teong, L. K., & Muhamed, A. R. (2013). Synthesis of activated carbon from lignocellulosic biomass and its applications in air pollution control. *Journal of Environmental Chemical Engineering 1* , 658–666.

<sup>5</sup> Palm Oil Facts and Figures 2011. <http://www.simedarbyplantation.com/Palm-Oil.pdf>

has produced more than 66.63 million tonnes of biomass residues such as Empty Fruit Bunches (EFB), mesocarp fiber, shell, palm kernel cakes, trunks and Palm Oil Mill Effluent (POME) in 2010 (Goh et al., 2009). The EFB represent about 9% of this total. They are the residue left after the fruit bunches has been processed to extract oil at oil mills.

In a country that has significant amount of agricultural activities, biomass can be a very promising alternative source of renewable energy. The awareness on reducing greenhouse gas emission has attracted global responsiveness to convert biomass residues into renewable such as activated carbon. The conversion of biomass to this functional compound involves two reaction processes which are pyrolysis and activation.

### 2.1.2 Empty fruit bunch (EFB)

Empty Fruit Bunch is composed of 45-50% cellulose and about equal amounts (25-35%) of hemicellulose and lignin (Deraman, 1993). Due to oil palm empty fruit bunch is available in large quantities and contain high amount of lignin, so empty fruit bunch fiber is appears to be a potential for activated carbon production. TABLE 1 below shows the composition of EFB under dry matter basis and fresh matter basis.

TABLE 1 Composition of EFB under dry matter basis and fresh matter basis

Parameter	Dry matter basis (mean)	Fresh wt. basis (mean)
Ash (%)	6.30	2.52
Oil (%)	8.90	3.56
Carbon (%)	42.80	17.12
Nitrogen (%)	0.80	0.32
Diphosphorous pentoxide (%)	0.22	0.09
Potassium oxide (%)	2.90	1.16
Magnesium oxide (%)	0.30	0.12
Calcium oxide (%)	0.25	0.10

Boron (mg/L)	10	4
Copper (mg/L)	23	9
Zinc (mg/L)	51	20
Ferum (mg/L)	473	189
Manganese (mg/L)	48	19
C/N ratio	54	54

Source: Cheng *et al.* (2007)

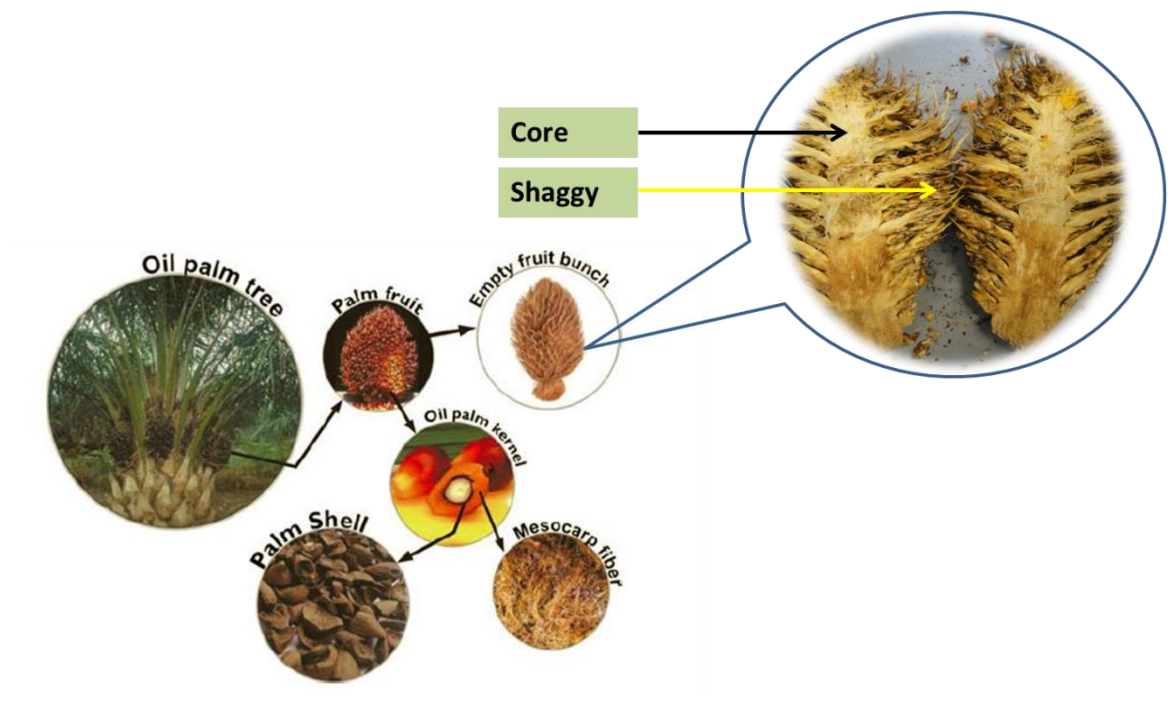


FIGURE 1 An empty fruit bunch (core and shaggy part)

In order to obtain the best advantages from the application of EFB in the field, inorganic supplements are also required. They are given for immature and mature plants (Gurmit *et al.* 1999). From the analysis done before, it is stated that one tonne of EFB (fresh weight) would have a fertilizer content equivalent of 3.8 kg urea, 3.9 kg rock phosphate,

18 kg muriate of potash and 9.2 kg kieserite. At current fertilizer prices, this would have a monetary value of RM12.00<sup>6</sup>

## 2.2 Pyrolysis

Pyrolysis is a thermochemical decomposition of biomass into a range of useful product, either in the total absence of oxidizing agents or with a limited supply. Pyrolysis of biomass is typically carried out in a relatively low temperature range of 300°C to 650°C. The nature of its product depends on several factors, including pyrolysis temperature and heating rate.

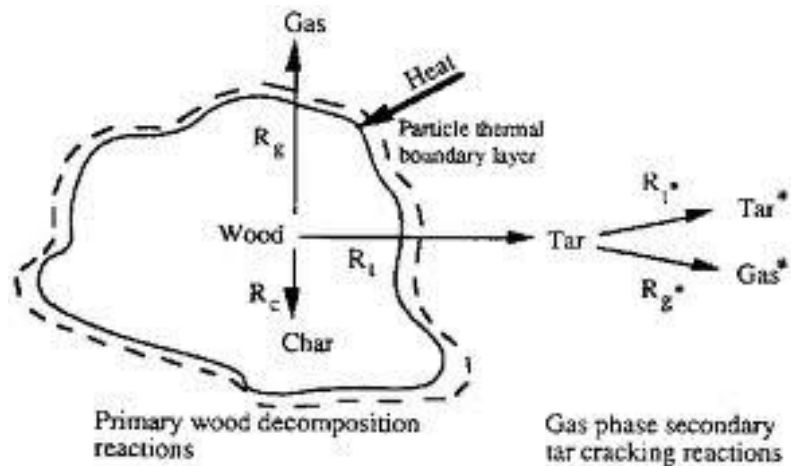


FIGURE 2 Pyrolysis in Biomass Particle

Figure 2 shows the initial product of pyrolysis is made of condensable gases and solid char. The condensable gas may break down further into non condensable gas ( $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{H}_2$  and  $\text{CH}_4$ ), liquid and char. (Basu, 2010)

<sup>6</sup> Chan, K.W., Chow, M.C., MA, A.N., and Yusof Basiron (2002). The global challenge of GHG emission on carbon reduction: palm oil industry. Paper presented at the 2002 National Seminar on Palm Oil Milling, Refining Technology, Quality & Environment. 19-20 August 2002. 12 pp.,



### 2.2.1 Pyrolysis Product

As mentioned before, pyrolysis process involves a breakdown of large complex molecules into several smaller molecules. The product from pyrolysis can be classified into three principal types:

1. Solid (most char and carbon)
2. Liquid (tars, heavier hydrocarbons, and water)
3. Gas (CO, CO<sub>2</sub>, H<sub>2</sub>, H<sub>2</sub>O, C<sub>2</sub>H<sub>2</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>6</sub>, C<sub>6</sub>H<sub>6</sub>, etc)

The relative amounts of these products depend on several factors including the heating rate and the final temperature reached by biomass. In proximate analysis, the liquid and gas yield are considered as 'volatile matter' while the char is called as 'fixed carbon'.<sup>7</sup>

#### *Solid*

Char is the solid product of pyrolysis. The primary contain is carbon. However it also contains some oxygen and hydrogen. If compared to fossil fuels, biomass contain small amount of inorganic ash.

#### *Liquid*

Tar, bio-oil or crude oil is the liquid product of pyrolysis. It consists mainly of homologous phenolic compounds. Bio-oil contains mixture of complex hydrocarbons with large amounts of oxygen and water. It was produced by rapidly and simultaneously depolymerizing and fragmenting the cellulose, hemicellulose and lignin component of biomass.

#### *Gas*

Condensable (vapor) and non-condensable gases (primary gas) are the products of primary decomposition of biomass. Vapors which are made from heavier molecules will condense during cooling, adding to the liquid yield of pyrolysis. For non-condensable

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<sup>7</sup> Basu, P. (2010). *Biomass Gasification and Pyrolysis*. Kidlington, Oxford: Elsevier Inc.

gas mixture it contains lower-molecular-weight gases like carbon dioxide, carbon monoxide and methane.

### **2.2.2 Types of Pyrolysis**

There are two types of pyrolysis based on heating rate which are slow pyrolysis and fast pyrolysis. Slow pyrolysis is also known as carbonization process. The primary product of carbonization is charcoal or char. The biomass will be heated slowly without oxygen to a relatively low temperature (~ 400°C) to the extended period of time. Fast pyrolysis will generate liquid and bio-oil as the main product. The biomass will be heated rapidly to reach the peak (pyrolysis) temperature before it decomposes. There are four factors that can increase the yield:

1. Very heating rate.
2. Reaction temperature within the range of 425 to 600°C.
3. Short residence time (< 3s) of vapor in the reactor.
4. Rapid quenching of the product.

### **2.3 Activation**

Preparation of activated carbon involves carbonization of the materials followed by activation. Basically, there are mainly two different methods for activation, namely physical and chemical. Physical activation process comprises treatment of the char obtained from carbonization with some oxidizing gases, generally steam or carbon dioxide at high temperature. The porous structure is created due to the elimination of volatile matter during pyrolysis and the carbon on the char is removed during activation. The main function of gasification is to widen the pores, creating large mesoporosity. In chemical activation, a chemical agent is impregnated to the precursors prior to heat treatment in an inert atmosphere. The pores are developed by dehydration and oxidation reactions of the chemicals.

## 2.4 Activated Carbon

In the early, commercialized activated carbons were prepared from wood and peat. In fact, activated carbons were obtained from any carbonaceous materials. Activated carbon has been employed in a wide number of applications on an industrial scale, including technologies for the purification of gases; the removal of organic pollutants from water and used as a catalyst/a catalyst support in the catalytic processes or electrode materials in electrochemical devices over the last few decades due to well-developed pore structures, high internal surface area and good mechanical characteristics. Besides, activated carbon also has played an important role in the chemical, pharmaceutical and food industries. Nowadays, the worldwide demand for activated carbon is kept increasing. The former raw materials used are non-renewable. Therefore, all possible sources should be investigated to replace former resources to produce activated carbon. Hence, the preparation of activated carbon from agricultural by-product is one of the potential investigations. (Guo, et al., 2009).

Activated carbon also known as activated charcoal, activated coal or carbon activates. It was a form of carbon with small and low-volume pores that increase the surface area available for adsorption or chemical reaction. Sometime activated can be replace with active. Activated carbon is usually derived from charcoal and increasingly, high-porosity biochar. It can be produced by physical activation or chemical activation process.

Some broad classification of activated carbon is made for general purpose based on their physical characteristics due to their complex products which are difficult to classify;

1. Powered activated carbon (R1) (PAC)
2. Granular activated carbon (GAC)
3. Extruded activated carbon (EAC)
4. Bead activated carbon (BAC)
5. Impregnated carbon
6. Polymer coated carbon

## CHAPTER 3

### METHODOLOGY

#### 3.1 Experimental

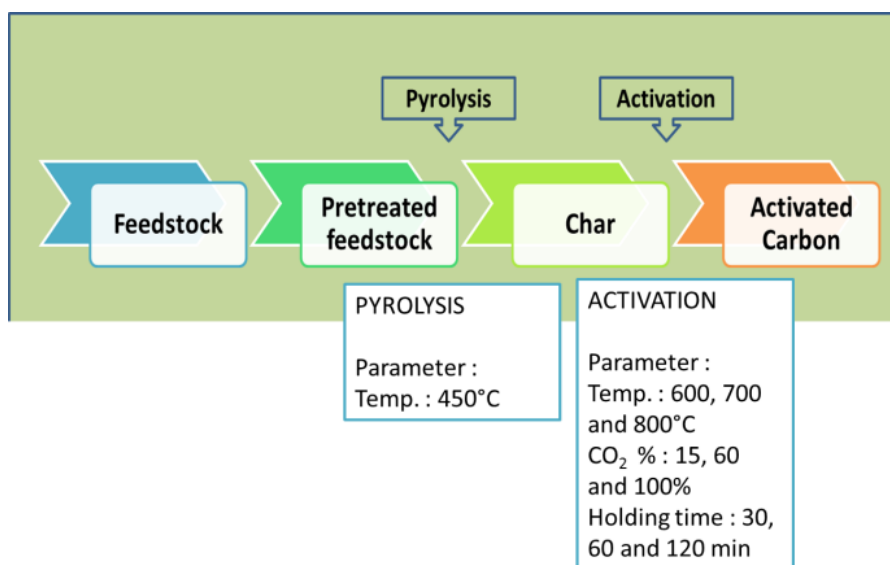


FIGURE 3 Scheme of activated carbon production

Figure 3 shows the overall process to produce activated carbon. The feedstock will undergoes pyrolysis to produce char. Char will be activated to produce activated carbon.

TABLE 2 Experimental strategies

		PYROLYSIS	ACTIVATION		
		Temp (°C)	Temp (°C)	CO2 percentage (%)	Activation time (min)
Shaggy 1	450	600	60	60	
		700	60	60	
		800	60	60	
		700	15	60	
		700	60	60	
		700	100	60	
		700	60	30	
		700	60	60	
		700	60	120	
Core 1	450	600	60	60	
		700	60	60	
		800	60	60	
		700	15	60	
		700	60	60	
		700	100	60	
		700	60	30	
		700	60	60	
		700	60	120	

Table 2 show different parameters are used to produce activated carbon from two different parts of EFB (shaggy and core).

### 3.2 Raw materials

#### 3.2.1 EFB preparation

EFB as the feedstock will be separated into two parts which are shaggy and core. The feedstock (shaggy and core) are oven dried at 105°C for 24 h. Dried feedstock will be stored in air-tight container. Then, the raw materials are grinded and sieved to the particle size in the range of 0.5 – 1.0 µm and stored in a desiccators at room temperature. The moisture content will be determined to achieve less than 10%.

### **3.2.2 Approximate analysis**

#### ***Moisture Content***

The moisture content is obtained by modified ASTM E1755 method, which is to determine water content in biomass. Separated shaggy and core were dried in the oven at 105°C. After 24 h of drying, the sample was weighed every one hour till the decrease in weight became negligibly small.

#### ***Ash Content***

A prescribed amount of sample (1 g) was weighed in a crucible, and was placed in an electric furnace and the temperature was raised to 700 °C. After 3 h, the furnace was turned off and was allowed to cool down; the crucible containing the ash was weighed.

### **3.2.3 Textural characterization by SEM**

The SEM image of dried raw FEB was carried by using scanning electron microscope Model TM3030 Tabletop Microscope HITACHI.

## **3.3 Pyrolysis experiments**

### **3.3.1 Pyrolysis of EFB**

Fast pyrolysis process (carbonization) will be run in a Drop Type Pyrolyzer. A scheme of the reactor is shown in Figure 4.

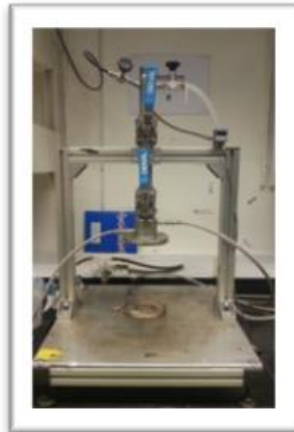


FIGURE 4 Drop Type Pyrolyzer

30g of biomass will be filled inside the pyrolyzer. Nitrogen gas ( $N_2$ ) was used as an inert gas to purge air from inside the reactor. Moreover,  $N_2$  was also employed to sweep the vapor products from the reactor into the condensation traps. The pyrolysis temperature was varied for 400°C, 450°C and 500°C but it had only a marginal influence on the quality of the activated carbon because the biomass was not completely devolatilized after pyrolysis. The reason is that activation took place at higher temperature than pyrolysis therefore the entire devolatilization had been realized in the activation step. The bio-char was collected from the inside of the reactor and weighed.

### **3.3.2 Ultimate Analysis**

CHNS equipment was used to determine the total carbon (C), nitrogen (N) and hydrogen (H). The metal contents in the bio-char and activated carbon were determined using Series 11 CHNS/O Analyzer 42400 Oarkim Elmer Precisely.

### **3.3.3 Textural characterization by SEM**

The SEM image of char from EFB was carried by using scanning electron microscope Model TM3030 Tabletop Microscope HITACHI.

## 3.4 Activation

### 3.4.1 Activation of the Pyrolised EFB Char

Activation will be run in a tube furnace as shown in Figure 5 below.

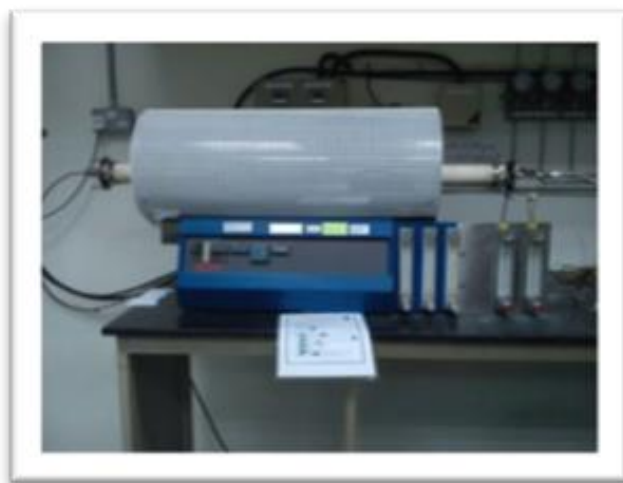


FIGURE 5 Tube Furnace

1.5 g carbonized chars were loaded inside the reactor situated in the tube furnace, and then heated up at a heating rate of 20 °C/min to the desired temperatures (600,700 and 800) under the N<sub>2</sub> gas flow and then the CO<sub>2</sub> gas (15%, 60% and 100%) was introduced to activate the samples to the desired activation time (30, 60 and 120 min). After activation, solid samples were cooled to room temperature under the N<sub>2</sub> gas flow. The products were kept for further characterization experiments.

### 3.4.2 Physical characterization

#### *Activated carbon yield*

The char characteristics and morphology are investigated. The final yield of bio-char is calculated using the following equation:



$$Y_p = \frac{X_1}{X_2} \times 100\%$$

where  $Y_p$  is the product yield,  $X_1$  is the mass of the desired product, and  $X_2$  is the initial mass of the raw material.

### **3.4.3 Textural characterization by SEM**

The SEM image of AC from EFB was carried by using scanning electron microscope Model TM3030 Tabletop Microscope HITACHI.

## **CHAPTER 4**

### **RESULT AND DISCUSSION**

#### **4.1 EFB Characterization**

##### **4.1.1 Proximate analysis**

Moisture content of shaggy and core were 3.8% whereas their ash content were 1.3% and 1.4% respectively.

#### **4.2 Bio-Char**

##### **4.2.1 Ultimate analysis**

Ultimate analysis was carried out to determine the amount of carbon content for two different char (shaggy and core) make them a good starting material for preparing activated carbon. (Law, Wan Daud, & Ghazali, 2007). The composition of char (shaggy and core) were shown in Table 3. The total carbon in C3 (char from shaggy) show the highest value which is 71.81% compare to other chars (shaggy and core) after undergo pyrolysis at 500°C.

TABLE 3 Char Composition

SAMPLE	PERCENTAGE (%)		
	C	H	N
<b>CHAR</b>			
<b>C1</b>	71.43	3.50	0.72
<b>C2</b>	69.47	3.21	0.61
<b>C3</b>	71.81	3.17	0.70
<b>C4</b>	62.13	4.49	0.90
<b>C5</b>	65.21	3.54	0.89
<b>C6</b>	62.71	3.96	0.80

### 4.3 Activated carbon

#### 4.3.1 Activated carbon yield

##### *Effect of Activation Temperature on the Yield of Activated Carbon*

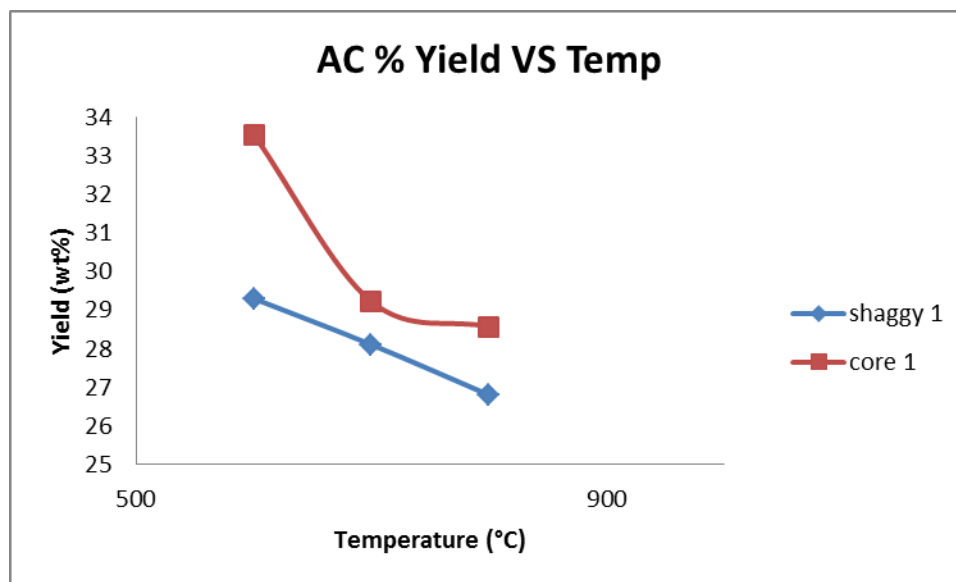


FIGURE 6 Graph of Activated Carbon Yield % VS Temperature

It is preferred to produce activated carbon that has high yield as it can improve process economy and market potential. The effect of activation temperature on the yield of activated carbon are covering an activation temperature from 600°C, 700°C and 800°C while the other parameters are maintained at carbon dioxide percentage 60% and a holding time of 60 minutes, respectively.

From Figure 6, it can be observed that the yield of activated carbon decrease progressively with increase in activated temperature from 600°C to 800°C.

The higher yield at low temperature could be due to the slower carbon and carbon dioxide reaction, with the weight loss commonly due to the release of volatile matters. The lower yield produced with the increase in activation temperature is due to the higher rate of the carbon and carbon dioxide reaction. (Yang, Peng, Xia, Zhang, Srinivasakannan, & Guo, 2010)

#### *Effect of CO<sub>2</sub> Percentage on the Yield of Activated Carbon*

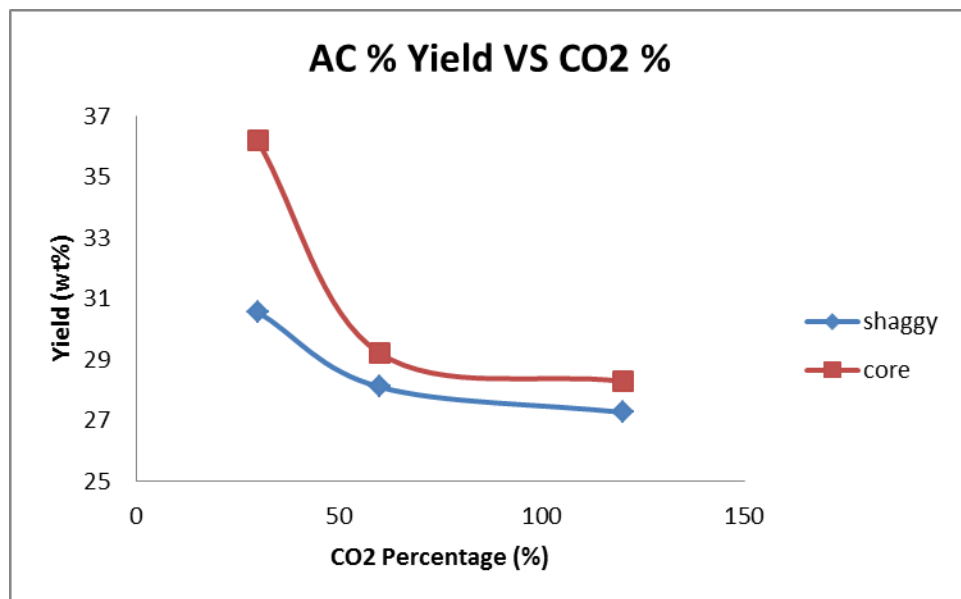


FIGURE 7 Graph of Activated Carbon Yield % VS CO<sub>2</sub> %

The effect of CO<sub>2</sub> percentage on yield of activated carbon is shown in Figure 7 (an activation temperature 700°C and holding time 60 min). It could be seen that CO<sub>2</sub> percentage has significant effect on the yield of the activated carbon, as it controls the rate of reaction of the carbon and carbon dioxide. At high percentage of CO<sub>2</sub> the rates of reactions is significantly higher to burn of the carbon evidenced by lower yield of activated carbon. (Yang, Peng, Xia, Zhang, Srinivasakannan, & Guo, 2010)

***Effect of Holding Time on the Yield of Activated Carbon***

TABLE 4 Effect of Holding Time on the yield of Activated Carbon

BIO-CHAR TYPE	ACTIVATION			Yield ( wt % )
	Activation time (min)	CO2 percentage (%)	Temp (°C)	
Shaggy 1 (450°C)	30	60	700	30.57
	60	60	700	28.10
	120	60	700	27.27
Core 1 (450°C)	30	60	700	36.18
	60	60	700	29.22
	120	60	700	28.29

The effect of holding time on yield of the activated carbon is studied in the ranges of 30–60 min at 700°C, CO<sub>2</sub> percentage of 60%. The effect of holding time on the yield of activated carbon is shown in Table 4. It could be observed that the yield of activated carbon decreases with the increase in holding time. At the time of 60 minutes and 120 minutes, there is no significant different of the yield of activated carbon. This could be expected as the longer holding time will give higher release of volatiles matter. Besides, the extent of carbon–carbon dioxide reaction will increase leading to higher carbon burn-off.

**4.3.2 Textural Characterization of Dried Raw EFB, Pyrolysis EFB Char and Activated Carbon by SEM**

Figure 8 shows the SEM image of outer surface and cross section for dried raw EFB (core), char from EFB (Core), and AC from EFB (Core). In the dried raw EFB (core), there were white circular craters are suspected to be silica-bodies which are one of the problematic constituent of EFB. The presence of silica could complicate pulping and bleaching of this material. The silica-bodies are hard. However, it can be dislodged mechanically. The EFB (core) average pore diameter of cross section is about 6-7 $\mu\text{m}$ . (Law, Wan Daud, & Ghazali, 2007)

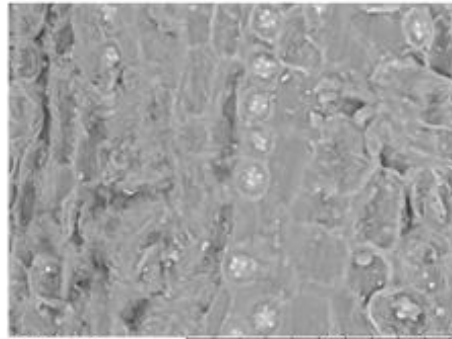
The image of the char from core seems to be disintegrated from the original structure of the EFB (core). Small cell cavities were shown from the image. Besides, non-developed porosity with some occasional cracks can be seen from the image too. The char average pore diameter is about 10-11 $\mu\text{m}$  which are wider compared to raw EFB (core) cross section pore diameter. (Ahmad, Wan Daud, Ahmad, Radzi, & Azmi, 2013)

The image shows the surface of the activated carbon (core) was full of irregular cavities. This indicates that the porosity was developed by activation agent. Different types of pores (small, transitional pores and large) with different shapes were also clearly visible under SEM image, which responsible for increased in pore cross section diameter. The average pore cross section diameter is about 30.2 $\mu\text{m}$ . It can be concluded that uniform pore of activated carbon can be developed using CO<sub>2</sub> activation.

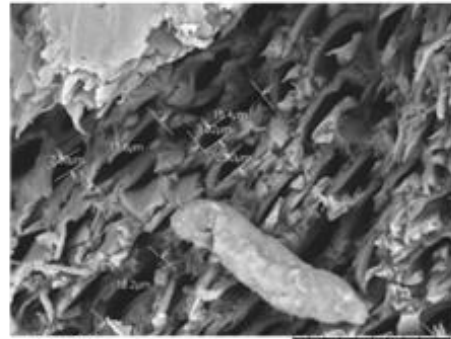
Figure 9 shows the image of dried raw EFB (shaggy) with white circular crater similar as spotted on raw EFB from core. The EFB (shaggy) cross section pore diameter is about 10-14 $\mu\text{m}$  which are wider than pore diameter of EFB from core.

Irregular cavities were spot on the outer surface of char indicated the effect of pyrolysis. Different size of pore can be spotted on the outer surface of activated carbon from shaggy under SEM image. Porosity was developed by CO<sub>2</sub> activation. The average cross section pore diameter is about 32.6 $\mu\text{m}$ . (Ahmad, Wan Daud, Ahmad, Radzi, & Azmi, 2013).

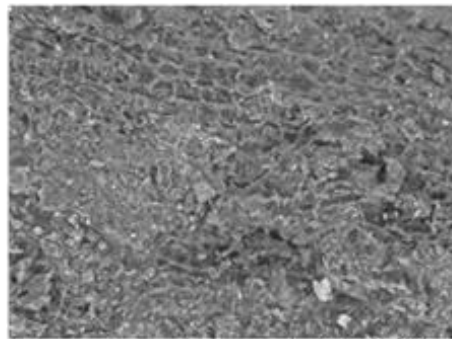
## EMPTY FRUIT BUNCHES (CORE)



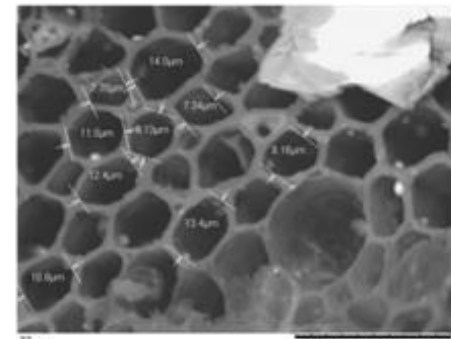
Raw EFB (Outer Surface)



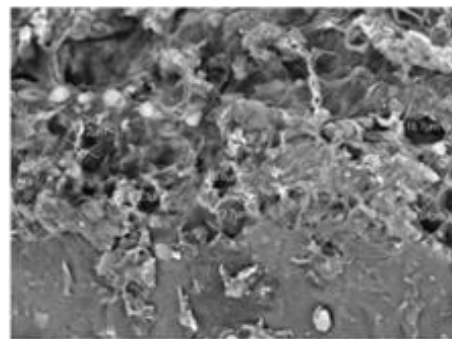
Raw EFB (Cross Section)



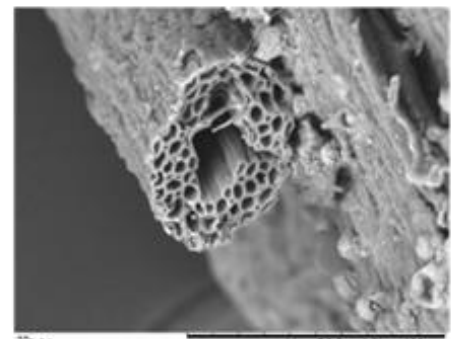
Char from EFB (Outer Surface)



Char from EFB (Cross Section)



AC from EFB (Outer Surface)



AC from EFB (Cross Section)

FIGURE 8 Scanning electron micrographs of raw EFB (core), char from EFB (core); activated carbon from EFB (core).

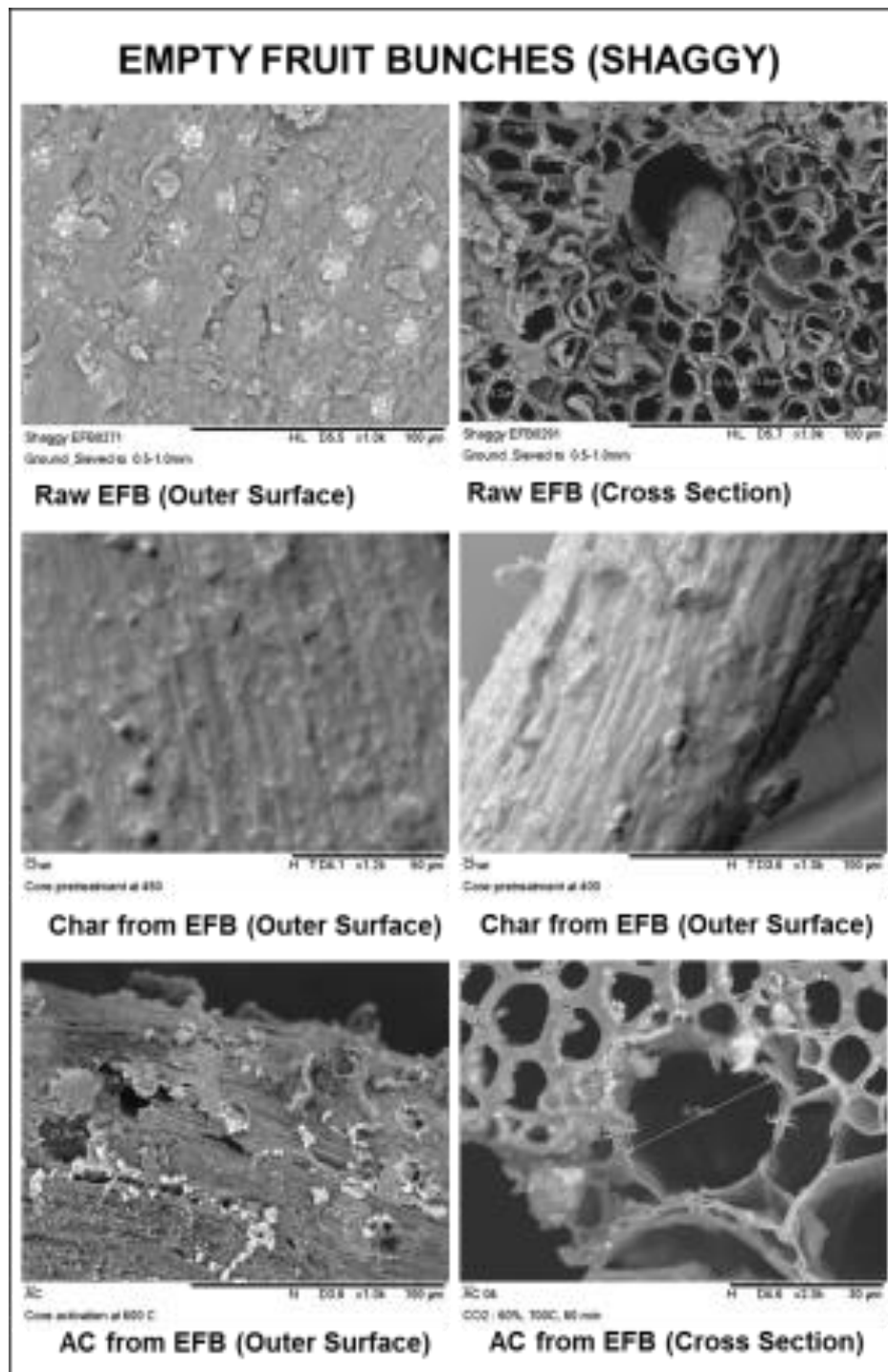


FIGURE 9 Scanning electron micrographs of raw EFB (shaggy), char from EFB (shaggy); activated carbon from EFB (shaggy).



## **CHAPTER 5**

### **CONCLUSION AND RECOMMENDATION**

Both part of EFB (shaggy and core) have high amount of carbon content that make them a good starting materials to produce activated carbon. The char yield, which was in the range of 29 – 33% at various carbonization temperatures. Carbonization at 450°C produced highest amount of yield. The optimum yield of activated carbon produced at the lowest activation temperature (600°C), lowest CO<sub>2</sub> percentage (15%) and shortest holding time (30 minutes). Activated carbon produced from shaggy EFB has bigger pore diameter (32.6µm) compared to core EFB (30.2µm) after activated at 700°C, 60 percent of carbon dioxide for 60 minutes of holding time. From the ultimate analysis, activated carbon produced still has high amount of carbon content. The highest is about 70.38 percent. For further study, the activation temperature needs to increase to burn off the entire carbons element. Hence, the pore diameter might increase and give a result of better activated carbon.

## REFERENCES

- [1] Ai We, I. T. (2008). *Preparation, Characterization And Evaluation Of Mesoporous Activated Carbons Derived From Agricultural By-Products For Adsorption Of Methylene Blue And 2,4,6-Trichlorophenol*. USM.
- [2] Alam, M. Z., S. A., & Kamaldin, N. (2008). *Production of Activated Carbon from Oil Palm Empty Fruit Bunches for Removal of Zinc*. Alexandria: Twelfth International Water Technology Conference, IWTC12.
- [3] Basu, P. (2010). *Biomass Gasification and Pyrolysis*. Kidlington, Oxford: Elsevier Inc.
- [4] Corporation, 2. J. (2007). *Reserach and Development*. Retrieved February 2014, from JFE Steel Corporation: <http://www.jfe-steel.co.jp/en/research/syozai.html>
- [5] Elisabeth Schröder<sup>1</sup>, K. T. (2011). *Activated Carbon from Waste Biomass*. InTech.
- [6] Environmental & Recycling Plant, Construction Dept.. JFE Engineering. (2004). *An Up-to-date Minicipal Waste Treatment Facility, "Tokorozawa City Tobu Clean Center"*. JFE GIHO.
- [7] F. A., A. A.-N., W. D., & J. S. (2013). *Characterization of Bio-oil and Bio-char from Pyrolysis of Palm Oil Waste*. Kuala Lumpur: Springer.
- [8] Faisal, Arami-Niya, A., Wan Daud, W., & Sahu, J. (2013). *Characterization of Bio-oil and Bio-char from Pyrolysis of Palm Oil Wastes*. Kuala Lumpur: Springer.
- [9] FLOW. (2014). *Palm Empty Fruit Bunch (EFB)*. Retrieved from Dp Clean Tech Global: <http://www.dpcleantech.com/biomasslab/example-analysis/palm-empty-fruit-bunch-efb-june>

- [10] Lehmann, J., & Joseph, S. (2010). *Biochar for Environmental Management Science and Technology*. UK: Earthscan.
- [11] Li, W., Yang, K., Peng, J., Zhang, L., Guo, S., & Xia, H. (2008). *Effects of carbonization temperatures on characteristics of porosity in coconut shell chars and activated carbons derived from carbonized coconut shell chars*. China: Elsevier B.V.
- [12] MANOCHA, S. M. (2003). *Porous carbons*. India: S<sup>ˆ</sup>adhan<sup>ˆ</sup>a.
- [13] Metcalf, & Eddy. (2004). *Wastewater Engineering Treatment and Reuse*. New York: McGraw-Hill.
- [14] NG, W.J., A.C.C. Goh and J.H. Tay, 1987. *Palm oil mill effluent (POME) treatment- an assessment of coagulants used to aid liquid-solid separation*. *Biological Wastes*, 21: 237-248.
- [15] Nor, N. M., Chung, L. L., Teong, L. K., & Muhamed, A. R. (2013). Synthesis of activated carbon from lignocellulosic biomass and its applications in air pollution control. *Journal of Environmental Chemical Engineering 1* , 658–666.
- [16] Singh, D. D. (2010). *National Biomass Strategy 2020: New wealth creation for Malaysia's palm oil industry* . Agensi Inovasi Malaysia.
- [17] Sukiran, M. A. (2008). *Pyrolysis Of Empty Oil Palm Fruit Bunches Using The Quartz Fluidised- Fixed Bed Reactor*. Kuala Lumpur: Universiti Malaya.
- [18] Yang, K., Peng, J., Xia, H., Zhang, L., Srinivasakannan, C., & Guo, S. (2010). Textural characteristics of activated carbon by single step CO<sub>2</sub> activation from coconut shells. *Journal of the Taiwan Institute of Chemical Engineers*, 367-372.
- [19] Faisal, Arami-Niya, A., Wan Daud, W., & Sahu, J. (2013). *Characterization of Bio-oil and Bio-char from Pyrolysis of Palm Oil Wastes*. Kuala Lumpur: Springer.

- [20] Law, K. N., Wan Daud, W. R., & Ghazali, A. (2007). Morphological and Chemical Nature of Fibers Strands of Oil Palm Empty-Fruit-Bunches (OPEFB). *Bioresources*, 351-362.
- [21] BIBLIOGRAPHY Guo, S., Peng, J., Li, W., Yang, K., Zhang, L., Zhang, S., et al. (2009). Effects of CO<sub>2</sub> activation on porous structures of coconut shell-based activated carbons. *Applied Surface Science*, 8443–8449.
- [22] Wu, M., Guo, Q., & Fu, G. (2013). Preparation and characteristics of medicinal activated carbon powders by CO<sub>2</sub> activation of peanut shells. *Powder Technology*, 188–196.
- [23] Jung, S.-H., & Kim, J.-S. (2014). Production of biochars by intermediate pyrolysis and activated carbons from oak by three activation methods using CO<sub>2</sub>. *Journal of Analytical and Applied Pyrolysis*, 116–122.
- [24] Alam, M. Z., Muyibi, S. A., Mansor, M. F., & Wahid, R. (2007). Activated carbons derived from oil palm empty-fruit bunches: Application to environmental problems. *ScienceDirect*, 103–108.
- [25] Molina-Sabio, M., Gonzalez, M. T., Rodriguez-Reinoso, F., & Sepulveda-Escribano, A. (1996). Effect of Steam and Carbon Dioxide Activation in the Micropore Size Distribution on Activated Carbon. *Carbon*, 505-509.
- [26] BIBLIOGRAPHY Islam, M. S., & Rouf, M. A. (2012). Waste biomass as sources for activated carbon production-A review. *Bangladesh Journal of Scientific and Industrial Research*, 347-364.
- [27] Zanzi, R., Bai, X., Capdevila, P., & Björnbo, E. (2011). Pyrolysis of Biomass in Presence of Steam for Preparation of Activated Carbon, Liquid and Gaseous Products. *6th World Congress of Chemical Engineering*, 23-27.
- [28] A., W. A., & N, G. V. (2013). Preparation of Low Cost Activated Carbon from Tea Waste using Sulphuric Acid as Activating Agent. *International Research Journal of Environment Sciences*, 53-55

## APPENDICES



Felcra Berhad Nasaruddin Oil Palm Mill



Palm oil processing area



Large amount of Empty Fruit Bunches (EFB) waste



Two parts of EFB (shaggy and core)



Author was having plant visit with one of the staff



Author with CBBR members after plant visit