# Removal of Oil and Grease from Refinery Desalter Effluent Using Carbon Derived from Agricultural Wastes

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Dissertation submitted in partial fulfillment of the requirements for the Bachelor of Engineering (HONS) (Chemical Engineering)

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## **CERTIFICATION OF APPROVAL**

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A project dissertation submitted to the Chemical Engineering Programme Universiti Teknologi PETRONAS in partial fulfillment of the requirement for the Bachelor of Engineering (Hons) (Chemical Engineering)

Approved:

Prof. Thanabalan Murugesan (Chemical Engineering Department) Project Supervisor

> UNIVERSITI TEKNOLOGI PETRONAS TRONOH, PERAK JUNE 2010

## **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.

AHMAD FIKREE BIN KAMIL

#### ABSTRACT

This report basically discusses the research done and basic understanding of the chosen topic, which is **Removal of oil and grease from refinery desalter effluent using carbon derived from agricultural wastes**. The study takes Petronas Penapisan (Melaka) Sdn. Bhd. as a model work where the sample of effluent water from refinery desalter is taken. Currently in PP(M)SB, the desalter effluent is being sent to the effluent treatment system and discharged to the environment. The main objective of the project is to reduce the cost and consumption of process water by recycling back the desalter effluent water by removing the oil and grease from the waste water. It was reported that the amount of waste water produced is about 2000 m3/day on average [21]. The proposed raw materials to be used as an adsorbent in this project are the rubber seed.

Rubber seed are found to be a good raw material for production of activated carbon [17, 18, 19]. It is revealed that particle size of raw material does not really affect the properties of the activated carbon. While the activation time and the use of chemical agent gives a significant effect on the produced activated carbon in terms of surface area, total pore volume and as well as pore structures.

It was found that 46 % of oil and grease removal was reported in the experiment. It is proved that activated carbon prepared from rubber seed, an agricultural solid waste, can be effectively used as adsorbent for the removal of oil and grease from refinery desalter effluent.

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# **CHAPTER 1**

## **INTRODUCTION**

#### 1.1 Background of study

Crude oil in refinery always containing water, salts, metal as well as suspended solid. All of the contaminants especially salts will lead to the corrosion, plugging and fouling of equipments. Besides, it is also can poison the catalyst in the processing unit. In order to remove all the contaminants, desalting process is needed.



Figure 1: Desalter system process flow

Water wash is used and injected into the crude oil stream in order to dilute salts in the crude oil. In the desalter drum, there is an electrical field used to ease the separation of diluted salts in water and the crude oil. The process will produced waste water that contain oil, grease, salts, mud and other impurities to be sent to the effluent treatment system (ETS). It is then being discharged to the aquatic environment since there is no further treatment to recycle the water back into the process.

#### **1.2 Problem statement**

In this project, the study takes Petronas Penapisan (Melaka) Sdn. Bhd. as a model work. In PP(M)SB, the produced effluent water from desalter that being send to the ETS will contribute to the company loss in term of processed water wasted to the environment. The amount of produced water effluent is about 2000  $m^3$ /day on average that will cost the company about RM 1,000,000/year [21].

Since there is no further treatment of the produced water effluent, it is possible to have one water treatment system that can treat the effluent water so that, the water can be recycle back into the desalter system which is the best way in solving the problem. The proposed system for the problem can be divided into two sections which are the adsorption section and the reverse osmosis section by membrane technology. The adsorption section is where the removal of oil & grease occur while for the RO section is where the removal of salts occurs.



Figure 2: Proposed solution for the desalter effluent system

As for this project, the author will cover only the first section which is the adsorption section that will removes the oil & grease from the effluent. Second section will be

study on a separate research where the focus is on the salt removal. Nowadays, there is a lot of technology or method used for treatment of waste water. One of the popular methods used is the separation by adsorption using an activated carbon derived from the agricultural waste.

Rubber seed is well known activated carbon derived from agricultural waste that can be used as an adsorbent. Hameed and Daud (2008) had done a research regarding the removal of basic dye by using the rubber seed coat. Besides, there is also a research about the potential of rubber seed coat in removing phenol and methylene blue [18,19]. Below is the summary of earlier work on activated carbon using rubber seed coat:

AUTHOR	YEAR	METHOD	APPLICATION
B.H.Hameed and	2007	KOH activation and CO <sub>2</sub>	Removal of basic dye
F.B.M. Daud		activation	
S. Rengaraj et al.	2001	Physical activation with N <sub>2</sub> gas	Removal of phenol
N. A. Oladoja et al.	2008	Physical activation with N <sub>2</sub> gas	Removal of
			methylene blue

Table 1: Summarize of earlier work on activated carbon using rubber seed coat

This type of activated carbon are proved to be one of the good activated carbon adsorbent in removing phenol, basic dye and methylene blue based on the available research. However, no investigation had been done to study the potential of the particular activated carbon on adsorbing oil and grease in waste water. Therefore, further study is needed to identify the potential of rubber seed in removing oil and grease that contain in the produced effluent water of refinery desalter system.

## **1.3 Objective**

The main objectives of this research are:

- To study the effect of activation parameters on the structure of carbon produced from rubber seed.
- To study the effect of activating agent (chemical) on the development of pore structure on activated carbon produced
- To evaluate the potential of activated carbon produced on removing oil and grease from the refinery desalter effluent water.

### 1.4 Scope of study

The study is divided into 4 major parts as follows:

1. Literature Review

In the literature review stage, the existing research that using activated carbon from agricultural waste as adsorbent is referred and reviewed. The activation method, characterization of activated carbon and experimental method on the activated carbon by other researchers are the important highlights to be studied during this stage.

2. Laboratory Set Up

Tools and equipment to be used will be identified and familiarized prior to the laboratory tests to avoid malfunctioning of the equipments. Accuracy of equipments used in the tests also will be checked in order to get accurate results.

3. Laboratory Tests

A series of laboratory tests on the chosen agricultural waste which is rubber seed will be performed starting from activation of the carbon, characterization of the activated carbon produced and as well as the experimental to see the effectiveness of the activated carbon on the sample.

# 4. Analysis of Results

Results obtained from the laboratory tests will be analyzed and interpreted. The result is important in order to achieve all the objective of the project.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Activated Carbon

Carbonaceous materials have long been known to provide adsorptive properties. The earliest applications may date back centuries with the discovery that charred materials could be used to remove tastes, colours and odours from water [1]. Activated carbons are widely used as adsorbents. They represent extremely versatile adsorbents of industrial significance and are widely used in many applications which concern principally with the removal of undesirable species from liquids or gases. They are also used as catalysts or catalyst supports or gas storages [2].

Activated carbons comprise elementary micro crystallites stacked in random orientation and are made by the thermal decomposition of various carbonaceous materials followed by an activation process. Raw materials include hard and soft woods, rice hulls, refinery residuals, peat, lignin, coals, coal tars, pitches, carbon black and agricultural waste such as coconut shell, palm oil shell and rubber seed [1].

#### 2.2 Formation and Manufacturing of Activated Carbon

The Manufacture of activated carbon involves two main steps which are the carbonization of the carbonaceous raw material at temperatures below 800°C in the absence of oxygen and activation of the carbonized product [3]. The final result is depending on the raw material used, activating agent and the condition or parameters used in the activation process [3]. The study is needed in order to identify what is the

best raw material and activating agent as well as the optimum parameters of the activation process in order to produce a good adsorbent.

The objective of carbonization and activation process is to increase the volume and diameter of the pores and also to create some new porosity, created during the process [3, 4]. The structure of the pores and their pore size distribution are largely predetermined by the nature of the raw material and the history of its carbonization [3]. In this research, the proposed activation method is the chemical activation method. In chemical activation both processes of carbonization and activation take place simultaneously. In this process, the raw material which in most of the cases is of cellulosic nature, is impregnated or kneaded with certain inorganic salts and this impregnated mass is then carbonized at low temperatures to ultimately yield an activated char, after washing with acid and water. The products obtained by the process of chemical activation are mostly used in liquid phase purification system [4].

There are several types of activating agent that can be used but the most widely used in industry nowadays is the Phosphoric Acid,  $H_3PO_4$ , Potassium Hydroxide, KOH and also Zinc Chloride, ZnCl<sub>2</sub>. The common feature of these activating agents is that they are dehydrating agents which influence the pyrolytic decomposition and inhibit the formation of tar. They also decrease the formation of acetic acid, methanol and enhance the yield of carbon [3].

Chemical activation is usually carried out at temperature between 400 and 800 °C [3]. There is an optimum temperature which is different for different types of raw materials. Some studied is needed in order to find the optimum activation temperature for a particular raw material. Allwar et al [2] study on textural characteristic of activated carbons prepared from Oil Palm Shells shows that there is an optimum activation temperature in order to get the best pore size distribution and large surface area. The study was varying the activation temperature from 400 to 800 °C and the results shows that at 500 °C gives the optimum temperature. At the activation temperature of 400 °C, the developing rudimentary pores of activated carbon were formed by removing the

low-molecular-weight volatile compounds from the matrix structure. Increasing the activation temperature to 500 °C enhanced the removal of molecular weight volatile compounds and further created new pores, resulting in the acceleration of porosity development of the activated carbon. However, when the activation temperature was increased to 600 °C, excessive heat energy was given to the carbon resulting in the knocking and breaking of some of porous wall, thus blocking the porosity formation. Hence, the pyrolysis at this activation temperature would yield decreasing the surface area of the activated carbon.

#### 2.3 Characterization and Physical properties of activated carbon

The performance of the activated carbon as the adsorbent relates in large measure to their intraparticle properties. Surface area and the distribution of area with respect to the pore size generally are the primary determinants of the adsorption capacity. The nature of the intraparticle surface area markedly affects the types of adsorption interactions that will be operative for an adsorbent, and it is a major distinguishing factor between the activated carbon and other synthesis adsorbent [5].

The most widely used commercial active carbons have a specific surface area of the order of 600- 1200 m<sup>2</sup>/g. The pore volume limits the size of the molecules that can be adsorbed whilst the surface area limits the amount of material which can be adsorbed, assuming a suitable molecular size. The adsorptive capacity of adsorbent is related to its internal surface area and pore volume.

The specific surface area  $(m^2/g)$  of porous carbon is most usually determined from gas adsorption measurement using the Brunauer-Emmett-Teller BET theory. [6]

The structure of an activated carbon is composed of pores classified into three groups, namely micropores, mesopores and macropores. Micropores usually account for over 95% of the total surface area of activated carbons. The volumes of the micropores range

from 0.15 up to 0.6  $\text{cm}^3/\text{g}$ . Conventional activated carbons are tridisperse, having all three types of pores present within their structure [7].

According to IUPAC:

- Micropores : below 1 nm radius
- Mesopores : 1-25 nm radius
- Macropores : radius > 25 nm

The actual adsorption occurs almost only in the micropores. The macropores will determine the accessibility of the adsorbent, while the mesopores influence the transport of the adsorbate from the gas phase to the micropores. An adsorbent with a high activation degree, and therefore a high total pore volume, will possess a high maximum adsorption capacity [8].

#### 2.4 Adsorption

Adsorption is the process by which Activated Carbon removes substances from water. Defined, adsorption is "the collection of a substance onto the surface of adsorbent solids." It is a removal process where certain particles are bound to an adsorbent particle surface by either chemical or physical attraction. Adsorption is often confused with Absorption, where the substance being collected or removed actually penetrates into the other solid. The reason that activated carbon is such an effective adsorbent material is due to its large number of cavernous pores. These provide a large surface area relative to the size of the actual carbon particle and its visible exterior surface. An approximate ratio is 1 gram = 1000 m<sup>2</sup> of surface area [22].

Activated carbon adsorption proceeds through 3 basic steps:

- 1. Substances adsorb to the exterior of the carbon granules
- 2. Substances move into the carbon pores
- 3. Substances adsorb to the interior walls of the carbon



Figure 3: Activated carbon

### 2.5 Adsorption isotherm

Adsorption isotherms should conventionally be plotted on the basis of relative pressure, p/po (x-axis) versus amount adsorbed expressed as a molar quantity (y-axis) in mmol/g, to allow comparisons to be made. The experimental procedure involves the use of partial pressure, where the actual pressure is expressed with respect to the saturation vapour pressure at a constant temperature of adsorption, hence the process is isothermal. Isotherms provide a significant amount of information about the adsorbent used and the interaction with the adsorbate in the system, including: [15]

- 1. Assessment of the surface chemistry and fundamentals involved in the adsorption process;
- 2. Estimates of the surface area, pore volume and pore size distribution;
- 3. Efficiency profiles for carbons used in industrial processes.

Basically there are six types of adsorption isotherm based on the IUPAC classification (Figure 2) shows the all six shapes of adsorption isotherm:

Type I isotherm indicates that the pores are microporous and that the exposed surface resides almost exclusively within the micropores, which once filled with adsorbate; leave little or no external surface for additional adsorption [10].

Type II isotherm mostly happened when adsorption occurs on the adsorbent with pore diameters larger than micropores [10]. Besides, physical adsorption of gases by non-porous solids is typified by this class of isotherm. Monolayer coverage is followed by multi-layering at high relative pressure [15]

Type III isotherm is convex, looking upwards and are characteristic of adsorption at sites of low adsorption potential and weak adorbate-adsorbent interactions [11, 15]

Type IV isotherm resemble Type II isotherm but additionally, instead of adsorption on open surfaces at high relative pressures, adsorption takes place in mesoporosity [11]. Its feature a hysteresis loop generated by the capillary condensation of the adsorbate in the mesopores of the solid [16]

Type V isotherms are those of a low energy, homogenous solid surface possessing mesoporosity. Type VI isotherm are of surfaces with an extremely homogenous structure (e.g. pyrolytic graphite) using for example, argon and methane ad adsorbate [11].



Relative pressure of adsorbate (P/Po)

Figure 4: The IUPAC classification of adsorption isotherm shapes

## 2.6 Liquid phase adsorption isotherm

When a quantity of adsorbent is contacted with a liquid that contain the absorbable solute, adsorption process will occur until the process reach equilibrium where there is no further adsorption occur. The common manner to represent this process is by expressing the amount of substance adsorbed per unit weight of adsorbent, q, as a function of the residual equilibrium concentration, c, of substance remaining in the

liquid phase [12]. This relationship is called adsorption isotherm model, defined the functional equilibrium distribution of adsorption with concentration of adsorbate at constant temperature. [13] The equation for the liquid phase adsorption isotherm is as below:

$$q = V(Co-C)/W$$
(1)

Where q is the adsorbate adsorbed per unit weight of adsorbent (mg/g), V is the volume of solution (L), Co is the initial concentration of solution (mg/L), C is the final concentration of solution (mg/L) and W is the weight of adsorbent (g).

The value q and c can be fitted into one or more standard isotherm equations, so that the q and c can be express in mathematical form to ease the analysis [14]. Isotherms are empirical relations, which are used to predict how much solute can be adsorbed by activated carbon. Besides, the isotherm can be use for theoretical evaluation and interpretation of thermodynamics parameters. The three most well known isotherms are the Freundlich, Langmuir and Linear isotherms. In environmental engineering and specifically water treatment application, the most commonly used isotherm is the Langmuir and Freundlich. Shown below is the Langmuir and Freundlich isotherm

Langmuir Isotherm 
$$q = q_o c / (K + c)$$
 (2)

Where qe is the amount adsorbed per unit mass of adsorbent (wt/wt),  $q_0$  and K are empirical constants, and Ce is the equilibrium concentration of adsorbate in solution after adsorption. The constants  $q_0$  and K can be determined by plotting Ce/qe vs. Ce and rewriting equation (2) as:

$$1/q = (K/q_o)(1/c) + 1/q_o$$
(3)

However, an empirical equation describes the Freundlich isotherm and typically proves to be the better relation. For the Freundlich isotherm, adsorption is described by:

Freundlich Isotherm 
$$q = Kc^n$$
 (4)

Where K and n are constants. Rewriting the equation as:

$$\log q = \log K + n \log C \tag{5}$$

And plotting log  $q_e$  vs. log  $C_e$  allows for the determination of K and n.

# CHAPTER 3

# METHODOLOGY

### **3.1 Introduction**

This chapter will explain about the equipments, apparatus and raw materials required as well as the procedures in order to conduct the experimental laboratory work. The laboratory work can be divided into 4 sections which are preparation of raw materials, chemical activation of raw materials, characterization of the activated carbon produced and adsorption experimental. For this project, the proposed raw material to be used is the rubber seed.

#### 3.2 Equipments, Apparatus, Raw Materials and Chemical Required

For different sections of the laboratory works need different equipments and apparatus as well as the chemical used.

NO	TITLE	(MAIN)
		EQUIPMENT/APPARATUS
1	Preparation of raw	1. Oven
	material	2. Dryer
		3. Grinder
		4. Siever
2	Chemical Activation	1. Fixed Bed Activation Unit

3	Characterization	1. Micromeritics ASAP 2020
		(surface area and porosity
		analyzer)
		2. Scanning Electron
		Microscopy (SEM)
4	Experimental	1. Oil & grease analyzer
		2. Floc tester
		3. Separatory funnel
1	1	

**Table 2**: List of equipments

## 3.2.1 Scanning electron microscope, SEM

- Type of microscope that images the sample surface by scanning it with a highenergy beam of electrons in a raster scan pattern.
- Operates on the same basic principles as the light microscope but uses electrons instead of light
- Light microscope is limited by the wavelength of light
- It use electrons as "light source" and their much lower wavelength makes it possible to get a resolution a thousand times better than with a light microscope.
- Provides topographical and elemental information at magnifications of 10 times to 100,000 times with virtually unlimited depth of field.

#### **Applications**

Use for research purpose in materials evaluation like:

- Grain size
- Surface roughness
- Porosity
- Particle size distributions
- Material homogeneity
- Intermetallic distribution and diffusion

## 3.2.2 Micromeritics ASAP 2020

- Type of surface area and porosity analyzer.
- It is a revolutionized measurement techniques and designed instrumentation to enable the accurate, precise, and reliable characterization of powdered and porous materials.
- uses the gas sorption technique to generate high-quality data

## **Applications**

Materials evaluation:

- Particle size and porosity
- Active surface area, size of active particles, total pore volume and average pore size diameter

## **3.3 Experimental Procedures**

## **3.3.1 Preparation of Raw Materials**

The main raw material to be used is the rubber seed. The rubber seed can be collected from the local rubber industry at the nearest location.



Figure 5: Rubber Seed

First, the rubber seed is washed with distilled water to remove any unwanted material attached on the rubber seed. It is then being dried in the oven for 24 hours at a temperature of 100°C in order to remove the moisture content inside the rubber seed as much as possible. Later then, the dried rubber seed is grinded and sieved into several sizes by using lab grinder and siever.



Figure 6: Grinder

Figure 7: Siever

The rubber seed is then being classified based on the size of the material. Further experiment will be conducted to see the effect of particle size of raw material in order to produce the better activated carbon.



Figure 8: 501 µm - 1 mm

**Figure 9:** 251 - 500 µm

## **3.3.2** Chemical Activation

For the activation section, the process was done at different parameters and conditions where sample A is the main sample, sample B with a different particle size, sample C at 30 minutes of activation time and sample D without a chemical agent. The parameters are based on the table below:

SAMPLE	SIZE	TIME	CHEM.	TEMP.
А	1 mm	3 hr	КОН	500 °C
В	500 µm	3 hr	КОН	500 °C
С	1 mm	30 min	КОН	500 °C
D	1 mm	3 hr	-	500 °C

Table 3: Parameters of activation process

Firstly, the grinded and sieved rubber seed is dried in an oven about 80 °C for a week. About 10g of the rubber seed is impregnated with 100 ml of freshly prepared concentrated solution of KOH with the impregnated ratio of 1:1.



Figure 10: Mixture of rubber seed and KOH solution



Figure 11: After being heated in water bath

The sample is then being heated in the water bath at 80°C with the shaker speed of 150 rpm. Later it was dried in an oven at 120°C overnight.





Figure 12: Water bath

Figure 13: Oven



Figure 14: Fixed Bed Activation Unit

The impregnated rubber seed is then being carbonized in the Fixed Bed Activation Unit. The furnace temperature was set at  $500^{\circ}$ C under nitrogen gas flow for 30 min and 3 hours. The resulting activated carbon is then cooled to a room temperature before being washed with hot distilled water for several times until pH 6–7 to remove any remaining KOH and then dried in the oven at  $110^{\circ}$ C.



Figure 15: Rubber Seed Activated Carbon

## **3.3.3** Characterization of the activated carbon

Characterization of the produced activated carbon is carried out using the Scanning electron microscopic and as well as the Micromeritics ASAP 2020 surface area and porosity analyzer. The results from these equipments will justified which samples had the higher adsorption capacities based on the surface roughness, porosity and also active surface area.

#### **3.3.4 Adsorption Experimental**

Adsorption of oil & grease on activated carbon is carried out using a beaker and floc tester. 10 g of prepared activated carbon was added into a beaker containing 300 ml of waste water from refinery desalter effluent. The sample was then stirred under the floc tester for 3 hours with 150 rpm of spinning blade. After 3 hours, the sample is then filtered by using a vacuum filter in order to separate between treated water and activated carbon.



Figure 16: Floc tester



Figure 17: Vacuum filter

The sample is then being analyzed using the oil and grease analyzer to observe the final concentration of oil and grease in the waste water. Tetrachloroethylene is used as a solvent to extract the oil from the sample water. 50 ml of the sample water was transfer into the separatory funnel and then added with 10 ml of tetrachoroethylene. The separatory funnel is then shakes for 1 minute and then waits 15 minutes so that the extraction will be completed. Later than, 10 ml of the bottom layer (mixture of tetrachloroethylene + extracted oil) from the funnel is taken to proceed with the analysis using the analyzer.



Figure 18: Separatory funnel



Figure 19: Oil and grease analyzer

# **CHAPTER 4**

# **RESULTS AND DISCUSSION**

## 4.1 Characterization

Some studies concerning the preparation of activated carbon from rubber seed with different parameters for activation under nitrogen had been reported based on Table 3. The SEM images of the prepared activated carbon are shown in Figure 20. The images can give clear view of the surface roughness on the prepared activated carbon and as well as the pores structure. The well developed pores had lead to the large surface area that means higher adsorptive capacities. The textural or physical characteristics of the prepared activated carbon are reported in Table 4. The textural characteristic of activated carbon involving BET specific surface area, total pore volume and average pore size diameter are very important because it can indicate the adsorptive capacities for the particular activated carbon.

#### 4.1.1 Scanning Electron Microscopy





**Figure 20**: SEM photograph of Rubber Seed after activation at 500°C for samples (i) Sample A (ii) Sample B (iii) Sample C (iv) Sample D

Based on the image above, by varying the size of the sample (sample A and sample B), there is not much different between the two of them in terms of surface roughness. Both surfaces show a lot of new porosity developed after the activation process. In addition, it is recommended to use the Transmission Electron Microscope in order to see the different in terms of porosity distribution since TEM can shows higher magnification up to 500 000 times.

By comparing between sample A and sample C, sample A at 3 hr of activation gives a better surface and porosity development. While at 30 minutes of activation time, sample C shows that the sample is in the initial process of developing porosity and not much of new porosity developed.

Besides, the study also included the comparison to see the effects of chemical on the produced activated carbon. Sample D is being activated without impregnated with chemical compare to the sample A where potassium hydroxide (KOH) is used as chemical agent. Based on the image above, it shows that chemical agent does give effect on the development of new porosity for the activated carbon.

#### **4.1.2 Physical Properties**

Sample	BET Specific surface area (m <sup>2</sup> /g)	Total pore volume (cm <sup>3</sup> /g)	Average pore diameter (nm)
А	1214.52	0.78	2.38
В	1157.69	0.73	2.24
С	1001.66	0.61	2.46
D	0.1816	-	-

Table 4: Physical properties of the prepared activated carbon

Table 3 shows the results of BET specific surface area, total pore volume and average pore diameter for all 4 samples of the prepared activated carbon. The results shows that sample A had the highest BET surface area and total pore volume which are 1214.52  $m^2/g$  and 0.78 cm<sup>3</sup>/g respectively. All samples shows the similar average pore diameter in the range of 2.24 – 2.46 nm.

By comparing between sample A and sample B where the particle size of the raw materials are in the range of 501  $\mu$ m - 1 mm and 251 - 500  $\mu$ m respectively, the effect on the characteristic of the produced activated carbon is not significant. These results show that the adsorption capacities does not determine by the initial particles size of the raw material. Instead, it is affected by the activation process parameters such as activation time and temperature.

In order to study the effect of activation time on the produced activated carbon, the activation process was run under two different activation times. Sample A was run at 30 minutes while sample C at 3 hours. The results show significant effects in terms of specific surface area and total pore volume. This indicates that further study is needed in order to determine the optimum activation time in producing the activated carbon.

Sample D was activated without using any chemical agents for the activation process. Based on the result, there are errors in determining the characteristic of the particular activated carbon. Based on the expected result, activation with chemical agent will gives a better surface area and porosity development. The presence of KOH as an impregnation agent would increase the heat energy on the activation process, and thus initiate to develop the porosity of activated carbon.

#### 4.2 Adsorption experimental

In order to observe the potential of prepared activated carbon in removing oil and grease from refinery desalter effluent, the adsorption experiment have been done. Due to the limited amount of Tetrachloroethylene, the experimental was conducted only to observe the concentration of oil and grease before and after the adsorption process. The adsorption experiment was done by using 30 grams of rubber seed activated carbon at 27°C (room temperature) with a mixing time of 3 hours and pH7. Below is the result of the experiment:

**Table 5:** Oil and grease concentration

Initial concentration, Ci	Final concentration, Cf
(ppm)	(ppm)
25.1	13.5

Based on the result, it was found that about a 46% of reduction in oil and grease was obtained throughout the experiment. It indicates that the activated carbon prepared from rubber seed, an agricultural solid waste, can be effectively used as adsorbent for the removal of oil and grease from refinery desalter effluent. However, further study is needed in order to determine the optimum condition of the adsorption process. By using the data obtained from the study, the adsorption equilibrium studies can be conducted. The isotherm of the oil adsorption can be represented by applying the Langmuir and Freundlich adsorption models.

## **CHAPTER 5**

# **CONCLUSION AND RECOMMENDATION**

The main objective of this project is to study the removal of oil & grease from the desalter's effluent water by using the activated carbon derived from agricultural waste. By taking the PP(M)SB as a model work, there is a possibility to treat the effluent water in order to reduce the process water consumption by recycle it back into the system. Based on the data from PP(M)SB, the recycle water can save the company up to RM 1,000,000/year in general excluding the capital and operating cost of the recycle system unit.

Rubber seed are found to be a good raw material for production of activated carbon. Studies are done on various parameters like the particle size of raw material, the activation time and as well as the use of chemical agent in activation process. It is revealed that particle size of raw material does not really affect the properties of the activated carbon. While the activation time and the use of chemical agent gives a significant effect on the produced activated carbon in terms of surface area, total pore volume and as well as pore structures.

Besides, it was found that 46 % of oil and grease removal was reported in the experiment. It is proved that activated carbon prepared from rubber seed, an agricultural solid waste, can be effectively used as adsorbent for the removal of oil and grease from refinery desalter effluent.

The outcome of this project is not only useful for preliminary studies of adsorption phenomenon, but it is also provide the reliable methodology, steps and procedure which can be followed by future students that studies a similar application.

For further studies, it is recommended to study the adsorption process by varying parameters affecting the process. The parameters were adsorbent dosage, mixing speed, mixing time and pH. This study will determine the optimum values of the parameters. As for the activation process, the optimum parameters and conditions should be determined by varying the parameters including the activation temperature.

In terms of commercializing the rubber seed activated carbon, a comparison with the other activated carbon used in industry should be done on similar experimental scale, based on the adsorption capacity of both activated carbons. From the data gained, basic economic justification can be done to support the fact.

### Recommendation

- The author had a problem in using several equipments due to broken equipments and long queue for some equipment. The author recommends that several important equipments such as fixed bed activation unit, SEM and as well as Micromeritics ASAP 2020 should be spare for final year project students rather than giving priorities to PhD or post graduate students. This is because duration for FYP is 1 year which is very critical and urgent.
- Besides, a list of equipments with the location of the equipments should be provided to students since the author didn't know where to find some equipment. This action will save some times so that students can book the equipments to avoid any delay in their works.

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