Preparation and Characterization of Activated Carbon using Rubber-seed Shell via Chemical Activation using Phosphoric Acid

by

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Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

AUGUST 2012

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CERTIFICATION OF APPROVAL

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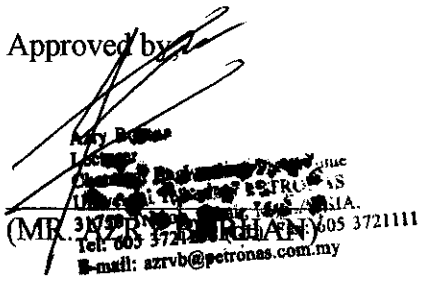
Approved by,



(MR. AZRI BIN RAHMAT)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

AUGUST 2012

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.

_____

NAIM AL-RASHID B. MOHAMAD ZAFARULLAH

Special dedication to my beloved ummi and abuya, for everything,
all my family members who always supported me.

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Finally, I would like to express deepest appreciation to those involved directly and indirectly in the completion of this research project.

ABSTRACT

Being known as an agricultural waste by-product, therefore the rubber-seed shell is being investigated to be used as alternative raw material to produce activated carbon via chemical activation using phosphoric acid H_3PO_4. The influence of carbonization temperatures (400-600°C), impregnation ratios (1:1 - 1:20) and sample size (45μm - 500μm) of the prepared activated carbon on the pore development and yield were investigated. The raw materials and produced activated carbons were characterized by Nitrogen adsorption isotherms and Variable Pressure Field Emission Scanning electron microscope. Oil and grease adsorption results show that rubber-seed shell is a good precursor for activated carbon. The optimal activation temperature is: temperature 500°C, impregnation ratio of 1:10 and activation time of 1.5 hour. Characteristics of activated carbon shows H_3PO_4 as a dehydrating agent that more to cleaning the surface of the activated carbon and promotes pore widening effect (1.09 nm – 6.53 nm) producing more shallow and wide mesoporous rich sites which makes a low specific surface area, thus low micropores sites. It is established that rubber-seed shell is an attractive source of raw material for producing high capacity of mesoporous and macroporous activated carbon by chemical activation with phosphoric acid H_3PO_4.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

Chapter 1

Introduction

1.1 Background of Study

The rubber tree or scientifically known as Hevea Brasiliensis is one of the major source of raw materials for the rubber making industries. The impact of the rubber tree towards the economy mainly because its latex which the primary source of natural rubber. It was initially grown only in the Amazon Rainforest, but the British had an idea to extensively propagate the rubber plantation in other British colonies during the days. The seed was smuggled out by Henry Wickham at the service of the British Empire (Dreifuss, 2002). The seeds were sent to several Botanical Gardens in Singapore, Java, Ceylon and up until 1898 only the first rubber plantation had been established in Malaya, and today majority of plantations are in South and Southeast Asia.

Since the early introduction of the plant, rubber industry in Malaysia had proven to be one of our country's major earnings; Malaysia also is the world's top three rubber-producing countries alongside Thailand and Indonesia. The Malaysia External Trade Development Corporation (MATRADE) also listed rubber products as 2.6% from a total of RM513.59 billion of export commodities. Latest statistics updates from October 2011 (DOS, 2011) show a natural rubber production of 85,800 tones of natural rubber which is an increase of 10, 4% compared to the previous month. This translates to a large area of rubber plantations here in Malaysia, because as of 2009 the total area rubber planted; smallholding and estate is a total of 1.02 million hectares. The government decided to give special focus under the Economic Transformation Plan (ETP) due to rubber's industries presence in downstream strengths and potential, they decided to expand rubber cultivation area to 1.2 million hectares by 2020 under the rubber industry development plan.

Therefore, expansion of rubber cultivation area will provide to develop other product aside from latex, such as production of biodiesel from rubber-seed oil which has now started in China (Sun, 2010), organic waste production from rubber-seed shell, rubber tree bark and others. These new findings can help to fully utilize the rubber tree which can add value to the rubber industry as a whole.

Activated Carbon is one of the alternatives, where rubber-seed shell can be a potential free raw material. Activated Carbon is a high surface area and porous carbon has been widely used as an adsorbent for separation, purification, decolourization etc. it can be produce by chemical and physical activation.

Figure 1: Dried rubber seed shell

1.2 Problem Statement

Oil Refinery is where crude oil is processed and refined into more useful petroleum products, such as naphtha, gasoline, diesel fuel, asphalt base, heating oil, kerosene, and liquefied petroleum gas (Gary, 1984). The contents of a crude oil usually contains water, salts, suspended solid, metals and etc. These contaminants especially salts can cause damage to equipment due to corrosion, plugging and fouling. It also can cause catalyst poisoning in the processing unit. Therefore desalting process is used to remove all the contaminants.

Therefore, the study project takes Petronas Penapisan (Melaka) Sdn. Bhd. as a model work. The produced effluent water from Refinery Desalting Unit (PSR-1) is being used for the case study. The effluent usually is being sent directly to Effluent Treatment System before being discharge to the sea. The study aims to find an alternative approached to treat the effluent with physical adsorption using Activated Carbon that will removes the oil and grease from the effluent. A possibility of the treated effluent to be recycled back to the fresh water stream can be a valuable outcome from the research as it will cut the cost for the fresh water for desalting process.

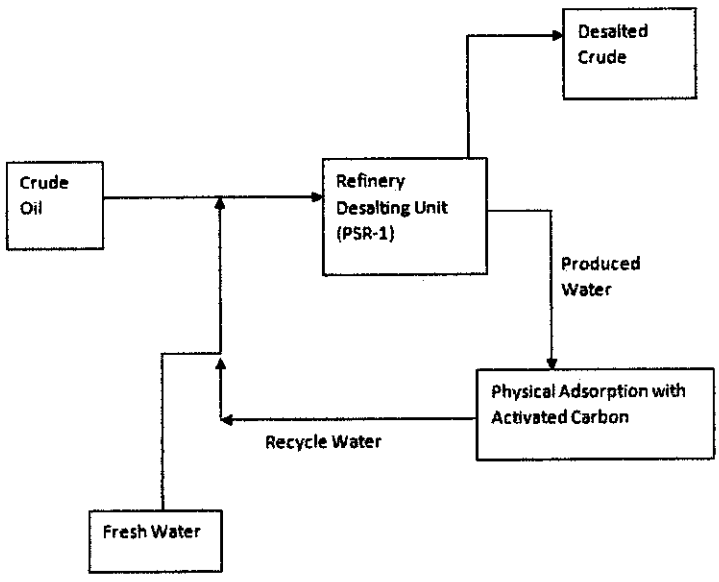

Figure 2: Alternative for effluent treatment from desalter

Activated carbon derived from the agricultural waste (rubber-seed shell) is used to separate by adsorption of oil and grease and it is one of the popular methods used.

Activated carbon has many of its usefulness, it can be applied in a variety of purification and separation, in the abatement of hazardous contaminants, municipal and industrial wastewater treatment, as catalyst or catalyst support in medicine, and the recovery of valuable metals etc (Sun, 2010). Many other options for raw materials are being used to produce activated carbon and some of the common agricultural wastes are tropical wood (Maniatis, 1992), saw dust (Xiongzun, 1986), pistachio shell (Abe, 1990), coconut shell (Kirubakaran, 1991) and almond shell (Hayashi J. K., 2000). The rubber-seed shell is an agricultural waste which has caused environment contamination problems in rubber tree plantations, and so far, there are few reports of the preparation of rubber-seed activated carbon (Sun, 2010).

In example (S. Rengaraj, 2002) use rubber-seed based activated carbon to study on removal of phenol from aqueous solution. Meanwhile, Hameed and Daud (B.H. Hameed, 2008) used it for basic dye adsorption studies. However no study has been made to study the effect of chemical activation using phosphoric acid on the rubber-seed shell activated carbon. Therefore, oil and grease adsorption studies on the particular activated carbon are needed to explore the effects of such chemical activation.

1.3 Objectives

The main objectives of this research are:

1. To study the effect of activation parameters on the structure of carbon produced from rubber seed shell

2. To study the effect of activating agent (phosphoric acid) on the development of pore structure on activated carbon produced

3. To analyse the absorption of potential activated carbon produced on removing oil and grease from the refinery desalter effluent water.

1.4 Scope of Study

The study is divided into four major parts as follows:

1. Literature Review

   In the literature review stage, the existing research of activated carbon from agricultural waste as adsorbent was referred and reviewed. The comparison on experimental methods, characterization of activated carbon, and results gained by other researchers were the important highlights to be studied during this stage.

2. Laboratory Set Up

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

3. Laboratory Tests

   A series of tests being done to the selected rubber-seed shell, starting from activation of the carbon, characterization of the produced activated carbon and adsorption experiment to see effectiveness of the activated carbon samples.

4. Analysis of Results

   Results obtained from laboratory tests will be analyzed and interpreted. The results are the important indicators to see the effectiveness of the system in order to achieve the project goals.

Chapter 2

Literature Review and Theory

2.1 Activated Carbon

Activated carbon which is a versatile adsorbent because of its good adsorption properties can be produced from variety of raw materials. Among them, coal is the most commonly used precursor due to its low cost and large supply, and also activated carbon prepared from coal is superior to those derived from lignocellulosic materials in terms of mechanical properties (Ahamadpour, 1996). Activated carbon is well known as a porous material and has a large specific surface area. Therefore, such material has desirable adsorption properties and has been used for purification and elimination of hazardous components in the gas and liquid phases. Due to current environmental pollution problems, activated carbon is expected to play an important role in pollution abatement (Hayashi J. H., 2002).

Many other options for raw materials are being used to produce activated carbon and some of the common agricultural wastes are tropical wood (Maniatis, 1992), saw dust (Xiongzun, 1986), pistachio shell (Abe, 1990), coconut shell (Kirubakaran, 1991) and almond shell (Hayashi J. K., 2000). The rubber-seed shell is an agricultural waste which has caused environment contamination problems in rubber tree plantations, and so far, there are few reports of the preparation of rubber-seed activated carbon (Sun, 2010).

2.2 Method of producing different activated carbon

Chemical activation: Prior to carbonization, the raw material is impregnated with certain chemical. The chemical is typically an acid, strong base, or a salt (phosphoric acid, potassium hydroxide, sodium hydroxide, zinc chloride, respectively). Then, the raw material is carbonized at lower temperatures (450-900°C). It is believed that the carbonization/ activation step proceeds simultaneously with the chemical activation.

Chemical activation is preferred over physical activation owing to the lower temperatures and shorter time needed for activating material.

In physical activation the precursor is developed into activated carbons using gases. This is generally done by using one or a combination of the following processes:

> Carbonization: Material with carbon content is pyrolyzed at temperatures in the range (600-900°C), in absence of oxygen (usually in inert atmosphere with gasses like argon or nitrogen)
>
> Activation/Oxydation: Raw material or carbonized material is exposed to oxydizing atmospheres (carbon dioxide, oxygen, or steam) at temperatures above 250°C, usually in the temperature range of 600-1200°C.

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 (Roop Chand Bansal, 1988), (Chemical activation). 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 (Roop Chand Bansal, 1988).

Several types of activating agent are recorded being used widely, the chemicals which are frequently used for activation like $ZnCl_2$, KOH, NaOH and K_2CO_3 (Guo J. L., 2003); (Lua, 2004); (Raymundo-Pinero, 2005); (Adinata, 2007). However, alkali hydroxides such as KOH and NaOH are hazardous, expensive and corrosive (Lillo-Rodenas, 2004) and $ZnCl_2$ is unfriendly towards the environment and creating problem on waste disposal (Guo J. L., 2003). Therefore, a more subtle chemical is desired; H_3PO_4 is a nontoxic when diluted as it is frequently used in many soft drinks ie Cola. Using H_3PO_4 for chemical activation, the activation temperature is relatively low (usually around 400oC), while the product yield is of higher grade. Another unique property of H_3PO_4 AC is their remarkable cation-exchange capability, chemical and thermal stability (Guo Y. R., 2007). It is also proposed that H3PO4 has two important functions: it promotes the pyrolytic decomposition of the initial material and the formation of the crosslinked structure (Jagtoyen, 1998).

2.3 Characterisation of activated carbon

Variable Pressure Field Emission Scanning Electron Microscope

In variable pressure or low vacuum SEM's, the sample is maintained at higher than normal pressures due to the introduction of a gas (usually water vapour or air) into the sample chamber. Unlike conventional high-vacuum SEM, this low vacuum, gaseous environment permits direct imaging of insulating and semiconductive materials without the need for a conductive surface coating. The theoretical principles of imaging non- and semiconductive materials in variable pressure environments has been documented in detail elsewhere (Toth, 2002) **and** (Stokes, 2003). However in brief, insulating materials can be imaged in a variable pressure SEM due to ionization of the chamber gas molecules by secondary electrons emitted from the sample during primary beam electron irradiation. Positive ions generated during these ionization events reduce negative charge build-up at the sample surface and largely eliminate the need for a conductive sample (Clode, 2006).

Field emission scanning electron microscopy (FE-SEM) provides a range of strategies for investigating the structural organization of biological systems, varying from isolated macromolecules to tissue organization and whole organisms. This review covers some of the results so far obtained using FE-SEM observation and various protocols of sample fixation to analyze the structural organization of parasitic protozoa and their interaction with host cells. The employment of FE-SEM can be broadened through the use of gold-labeled molecules or tracers, gradual extraction by detergents, and cleavage techniques. These analyses provide significant contributions to the characterization of these organisms concerning ultrastructure, cytoskeleton, motility and intracellular behaviour (De Souza, 2008).

Brunauer Emmet Teller (BET)

BET theory aims to explain the physical adsorption of gas molecules on a solid surface and serves as the basis for an important analysis technique for the measurement of the specific surface area of a material. BET is a standard (recommended by IUPAC (Sing, 1985) and (Rouquerol J. A., 1994) the most widely used procedure to characterize various adsorbents (R.Ch. Bansal, 2005); (T.H. Marsh, 2006); (Rudzin´ ski, 1992); (Rouquerol F. R., 1999) and (Lowell, 2004).

BET surface area is provided in almost each paper where activated carbons are applied, it is a standard parameter (Gauden, 2010).

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 cm3/g. Conventional activated carbons are tridisperse, having all three types of pores present within their structure (Nevin Yalc¸ın*, 2000).

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 (Saad, 2007).

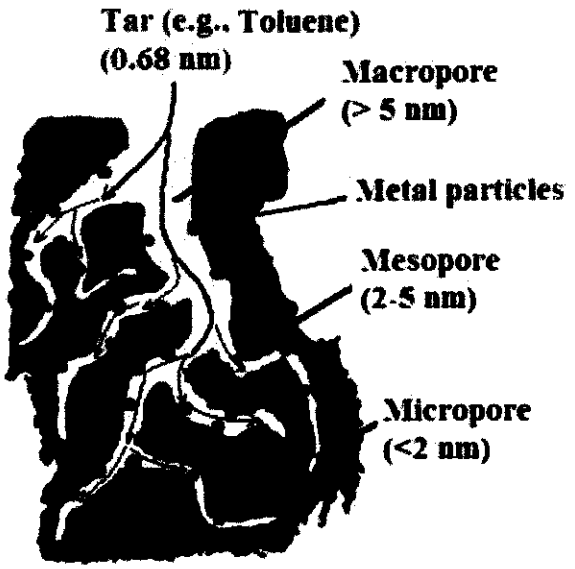

Figure 3: Different pore sizes (C. Xua, 2010)

2.4 Adsorption Isotherm

When a gas comes into contact with a solid surface, molecules of the gas will adsorb to the surface in quantities that are a function of their partial pressures at a single temperature results in a graph known as an adsorption isotherm. Many different types of isotherms have been observed in the literature (Findenegg, 1984) these isotherms can have very different shapes depending on the type of adsorbent, the type of adsorbate and intermolecular interactions between the gas and the surface.

Types of isotherms (SING, et al., 1985):

Type I isotherms are given by microporous solids having relatively small external surfaces (e.g. activated carbons, molecular sieve zeolites and certain porous oxides), the limiting uptake being governed by the accessible micropore volume rather than by the internal surface area

Type II isotherm is the normal form of isotherm obtained with a non-porous or macroporous adsorbent. The Type II isotherm represents unrestricted monolayer-multilayer adsorption. Point B , the beginning of the almost linear middle section of the isotherm, is often taken to indicate the stage at which monolayer coverage is complete and multilayer adsorption about to begin

The reversible Type III isotherm is convex to the pip° axis over its entire range and therefore does not exhibit a Point B . Isotherms of this type are not common, but there are a number of systems (e .g. nitrogen on polyethylene) which give isotherms with gradual curvature and an indistinct Point B .In such cases , the adsorbate-adsorbate interactions play an important role.

Type IV isotherm are its hysteresis loop, which is associated with capillary condensation taking place in mesopores, and the limiting uptake over a range of high p/p°. The initial part of the Type IV isotherm is attributed to monolayer-multilayer adsorption since it follows the same path as the corresponding part of a Type II isotherm obtained with the given adsorptive on the same surface area of the adsorbent in a non-porous form. Type IV isotherms are given by many mesoporous industrial adsorbents.

The Type V isotherm is uncommon; it is related to the Type III isotherm in that the adsorbent-adsorbate interaction is weak, but is obtained with certain porous adsorbents.

The Type VI isotherm, in which the sharpness of the steps depends on the system and the temperature, represents stepwise multilayer adsorption on a uniform non-porous surface. The step-height now represents the monolayer capacity for each adsorbed layer and, in the simplest case, remains nearly constant for two or three adsorbed layers. Amongst the best examples of Type VI isotherms are those obtained with argon or krypton on graphitised carbon blacks at liquid nitrogen temperature

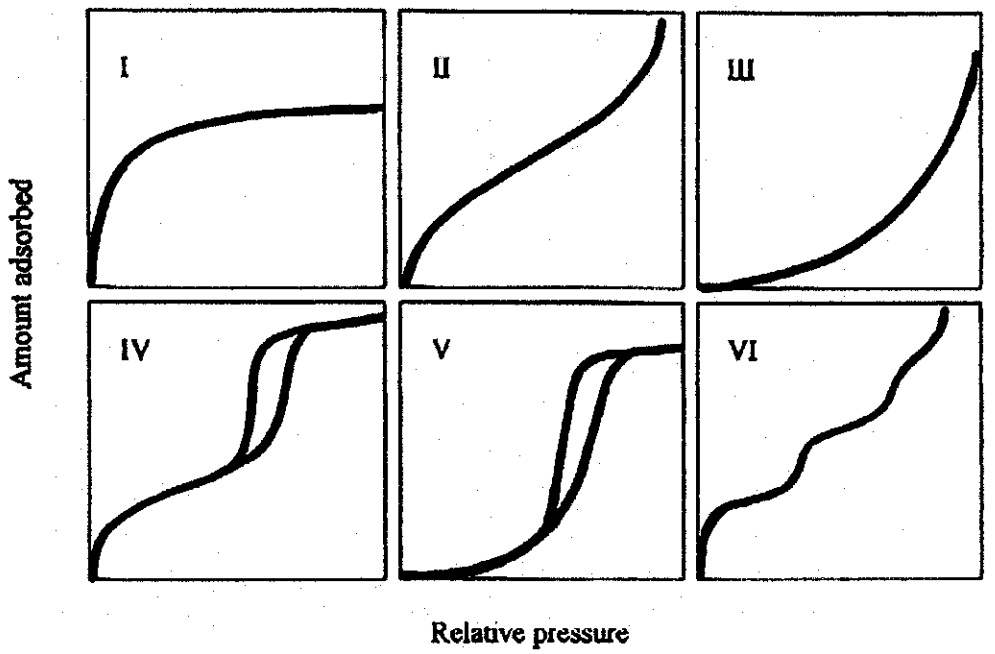

Figure 4: Types of isotherm (M.D Donohue, 1998)

2.5 Liquid Phase Adsorption Theorem

Adsorption is process when a quantity of adsorbent is contacted with a liquid that contain the absorbable solute until the process reach equilibrium where there is no further adsorption to occur. Adsorption or adsorption isotherm model can be represented by calculating the amount of substance adsorbed per unit weight of adsorbent, q, as a function of the residual concentration, c, of substance remaining in

the liquid phase (Seader, 1998) . The equation for a liquid phase adsorption isotherm is as below:

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

q = adsorbate adsorbed per unit weight of adsorbent (mg/g)
v = volume of solution (L)
Co = initial concentration of solution (mg/L)
C = final concentration of solution (mg/L)
W = weight of adsorbent (g)

Value for q and C is applicable to one or more standard isotherm equations, in order to ease calculation and analysis q and C can be express mathematically (Din, 2004). As empirical relations, isotherms are used to project how much amount of solute could be adsorbed by activated carbon. Isotherms like the Freundlich, Langmuir and Linear are being used to interpret thermodynamics parameters and theoretical evaluation. In our case of adsorption from a wastewater source effluent, usually Freundlich and Langmuir isotherm are being used as shown below in their general form (Seader, 1998)

$$\text{Freundlich Isotherm } q = Kc^{n} \quad (2)$$

K and n = consonants. We can rewrite the equation as:

$$\log q = \log K + n \log C \quad (3)$$

By plotting log qe vs. log Ce we can determine the value of K and n.

$$\text{Langmuir Isotherm } q = q_o c / (K + c) \quad (4)$$

qe = amount adsorbed per unit mass of adsorbent (wt/wt)
qo = empirical constant
K = empirical constant
Ce = equilibrium concentration of adsorbate in solution after adsorption

We can determine both qo and K constant by plotting Ce/qe vs. Ce and reqriting equation (4) as:

$$1/q = (K/qo)\,(1/c) + 1/qo \qquad (5)$$

However, the Freundlich isotherm typically proves to be a better relation to be described as an empirical equation.

2.6 Determination of Oil and Grease

In order to control oil and grease, one must have a basic understanding of its characteristics. Oil and grease are found in waste water either as an emulsion or as free-floating agglomerates. (J.P. Dodd, 2002)

The oil and grease contents of domestic and certain industrial wastes and the sludge, is of an important consideration in the handling and treatment of these material for ultimate disposal. Knowledge of the quality of the oil and grease present is helpful in proper design and operation of wastewater treatment system. The term grease applies to wide variety of organic substance that is extracted from aqueous solution or suspension by hexane. Hydrocarbons, esters, oils, fats, waxes and high molecular weight fatty acids are the major materials dissolved by hexane. All these material have a greasy feel and are associated with the problems in wastewater treatment related to grease. (Encyclopedia, 2009)

The Department of Environment (DOE) has been conducting monitoring of river since 1978, primarily to establish baselines and to detect water quality changes in river water quality and has since been extended to identifying of pollution sources as well. A total of 1,064 manual stations located within 143 river basins throughout Malaysia.

Water quality data were used to determine the water quality status weather in clean, slightly polluted or polluted category and to classify the rivers in Class I, IIA, IIB, III or IV based on Water Quality Index (WQI) and Interim National Water Quality

Standards for Malaysia (INWQS) every year. Water Quality Index (WQI) is computed based on 6 main parameters:-

- Biochemical Oxygen Demand (BOD)
- Chemical Oxygen Demand (COD)
- Ammoniacal Nitrogen (NH_3N)
- pH
- Dissolved Oxygen (DO)
- Suspended Solids (SS)

Other parameters such as heavy metals and bacteria would be measured according to site requirement. (DOE, 2010)

Interim National Water Quality Standard (INWQS) is used to determine the water quality in this country. The Department of Environment of Malaysia used this as a classification standard for river effluent content standards. The water quality parameter can be classified in a predetermined class base on the reading acquired from the source. There are a total of 6 main class in INQWS as listed below.

Class 1 = Conservation of natural environment water supply
= Practically no treatment
= Fishery 1, very sensitive aquatic species

Class IIA = Water Supply II, conventional treatment required
=Fishery II, sensitive aquatic species

Class IIB =Recreational use with body contact

Class III =Water Supply III. Extensive treatment required
= Fishery III, common of economic value and tolerant species livestock drinking

Class IV = Irrigation

Under the Environmental Quality (Sewage and Industrial Effluents) Regulations 1979 P.U.(A) 12-79 they have listed a parameter limits of effluent of standards A and B. Each of the standards listed catchment areas where each of the standards applies and also the maximum concentration of oil and grease in particular of this study that can be release.

Standard A	= not detectable
Standard B	= 10 mg/l
Other than Standard A & B	= 100 mg/l

Chapter 3

METHODOLOGY

3.1 Introduction

This chapter will explain further on the equipments, apparatus and raw materials used, as well as the procedures on conducting experimental laboratory work. The laboratory works are divided in 4 sections which are preparation of raw materials, chemical activation of raw materials, characterization of the activated carbon produced and adsorption experimental works. In this particular project, the targeted raw material to be tested is the rubber seed shell with chemical activation using phosphoric acid.

3.2 Equipments, Raw Materials, and Chemical Required.

Each of the equipments or apparatus, raw materials and chemical required to run the laboratory works are summarized in the table below:

No	Step	Main Equipment
1.	Preparation of raw material	1. Oven 2. Dryer 3. Grinder 4. Siever
2.	Chemical Activation	1. Fix Bed Activation Unit 2. Container for impregnation 3. Water Bath
3.	Characterization	1. Micromeritics ASAP 2020 (surface area and porosity analyser) 2. Field Emission Scanning Electron Microscopy (FE SEM)
4.	Adsorption Experiment	1. Infracal TOG/TPH Analyzer (total oil and grease/ total petroleum hydrocarbon

		analyser) 2. Sample Bottle 3. Measuring Flask 4. Micropipette

Table 1: List of Equipment

3.2.1 Field Emission Scanning Electron Microscope

Principle of Operations, a field –emmision cathode in the electro gun of a scanning electromicroscope provides narrower probing beams at low as well as high electronenergy, resulting in both improved spatial resolution and minimized sample charging and damage.

- Produces clearer, less electrostatically distorted images withspatial resolution down to 1 ½ nm. 3 to 6 times better than conventional SEM. (SEM provides topographical and elemental information at magnifications of 10 times to 100,000 times with virtually unlimited depth of field.
- Smaller-area contamination spots can be examined at electron accelerating voltages compatible with Energy Dispersive X-ray Spectroscopy.
- High quality, low voltage images are obtained with neglible electrical charging of samples. (Accelerating voltages ranges from 0.5 to 30 kV)

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

Micromeritics' ASAP 2020 Accelerated Surface Area and Porosimetry analyzer uses the gas sorption technique to generate high-quality data for research and quality control applications. Available options include the micropore option, the high-vac option, and the chemisorption option, which uses the static volumetric technique to determine:

- percent metal dispersion
- active metal surface area
- size of active particles
- surface acidity of catalyst materials
- characterize the active and support surfaces of catalysts
- determine the high surface areas of adsorbents
- determine the microporosity and hydrogen storage capacity of various nano materials

3.2.3 Micropipette

Made to measure and release small amounts liquid. Micro pipettes released between the 1 and 1,000 microliters. They are frequently used in injection experiments and measurements, because they are very precise.

3.2.4 InfraCal IR TOG/TPH Platform Analyzer, Model HATR-T2

Applications (Geneq):

- Measuring total oil and grease (TOG), total petroleum hydrocarbon (TPH), or fat oil greas level (FOG) in water and soil samples.
- Using hexane,pentane or Vertrel MCA as the extracting solvent.
- Measurement procedures are based on evaporation techniques and measuring the residual oil and grease.

- Light-end volatile components will be lost in the evaporation process.
- Detect levels as low as 2 ppm or as high as 999 ppm

3.3 Procedures

3.3.1 Preparation of Raw Materials

The rubber (H. basiliensis) seed shell was collected from a rubber plantation estate in Gelung Pepuyu, Perak, Malaysia and was used as the precursor for the preparation of activated carbon in this work. The precursor was first washed with water to remove dirt. It was the dried in an oven overnight at 100°C overnight to remove the moisture inside the rubber seed shell as much as possible. Crushed with stone mortars and ground to particle size of 500µm – 45µm mesh and stored in plastic containers and kept in desiccators for further use.

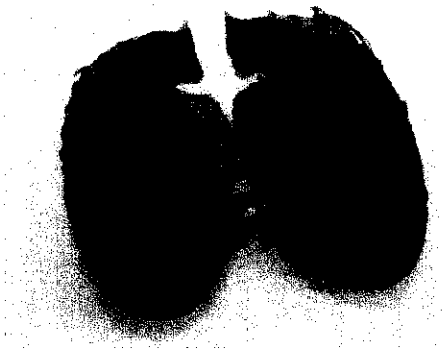

Figure 5: Raw Rubber Seed Shell

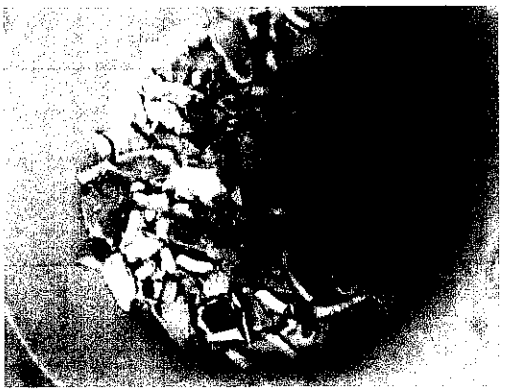

Figure 6: Crushed with stone mortar

Figure 7: Grinder IKA MF-10

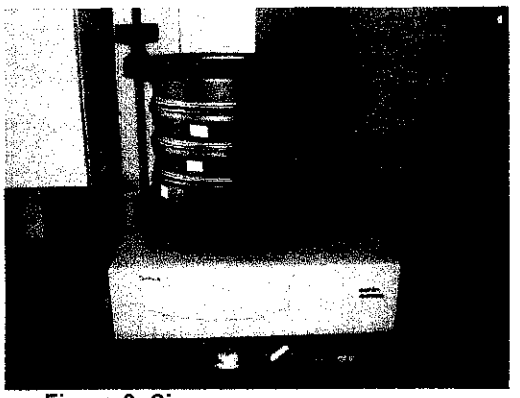

Figure 8: Sieve

After grinding process the rubber seed shell are sorted based on their material size. Further experiment procedures will be conducted to see the effect of particle size of raw material in order to produce the better activated carbon.

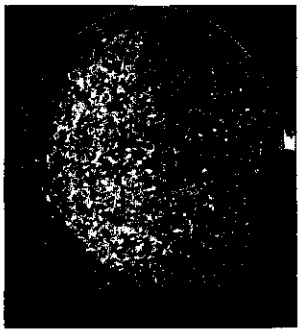

Figure 9: 500 µm

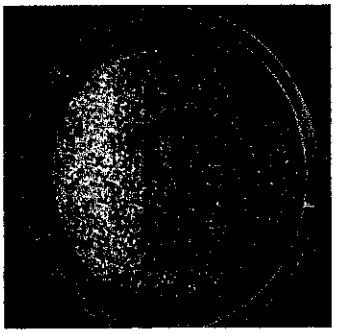

Figure 10: 125 µm

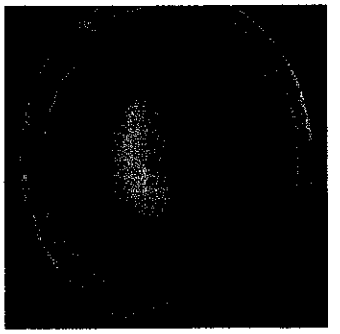

Figure 11: 45 µm

3.3.2 Chemical Activation

For the activation part, the process was done at different parameters and conditions, where we are looking at several variables to be tested; different particle size, different activation temperature, different impregnation ratio and different activation time.

Table 2: Parameters of activation process

SAMPLE	SIZE	TIME	RATIO	TEMPERATURE
A1	500 µm	2 hr	1 : 1	500 °C
A4	125 µm	2 hr	1 : 1	500 °C
A7	45 µm	2 hr	1 : 1	500 °C
A2	500 µm	2 hr	1 : 20	500 °C
A3	500 µm	2 hr	1 : 10	500 °C
B1	500 µm	2 hr	1 : 1	600 °C
C1	500 µm	2 hr	1 : 1	400 °C
E1	500 µm	1 hr	1 : 1	500 °C
D1	500 µm	1.5 hr	1 : 1	500 °C
A9	45 µm	1.5 hr	1 : 10	500 °C

After grinding and sieving the raw materials to desired sizes, the samples are dried in an oven about 100 °C for a couple of days to remove any trapped moisture. About 10g of the rubber seed is impregnated with different 10ml, 100ml, and 200ml volume of concentrated solution of H3PO4 respectively.

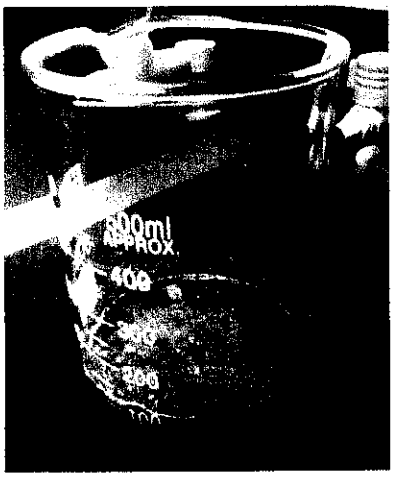

Figure 12: Rubber seed shell impregnated with H3PO4 solution

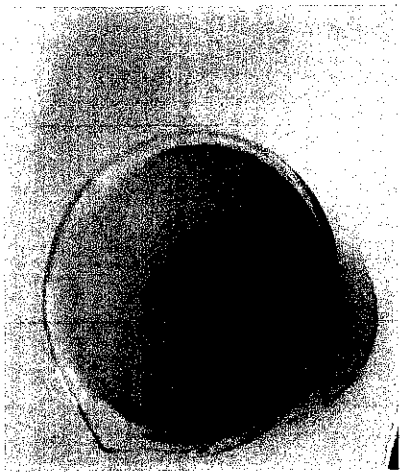

Figure 13: After heating in water bath

After adding the concentrated solution of H3PO4, the mixture is then being heated in the water bath at 80 °C with the shaker speed of 150 rpm. Then it was dried in an oven at 100 °C overnight.

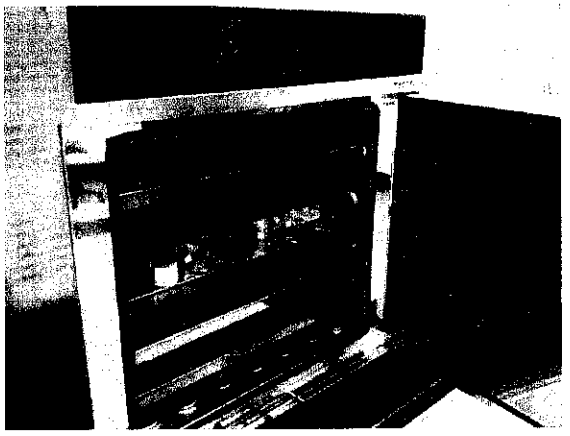

Figure 14: Oven

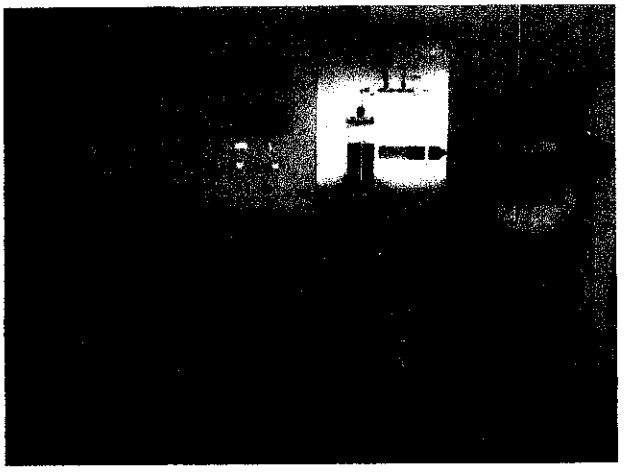

Figure 15: Fix Bed Activation Unit

After impregnation process, the carbonization process was carried out with a laboratory furnace, known as the Fix Bed Activation Unit. This furnace was set to 3 different target temperatures at 400°C, 500°C and 600°C under nitrogen gas flow for

1 hour, 1.5 hour and 2 hours. The produced activated carbon was then cooled off to a room temperature and washed with hot distilled water for several times until pH 6-7 to remove any remaining H3PO4 and residual organic and mineral matters.

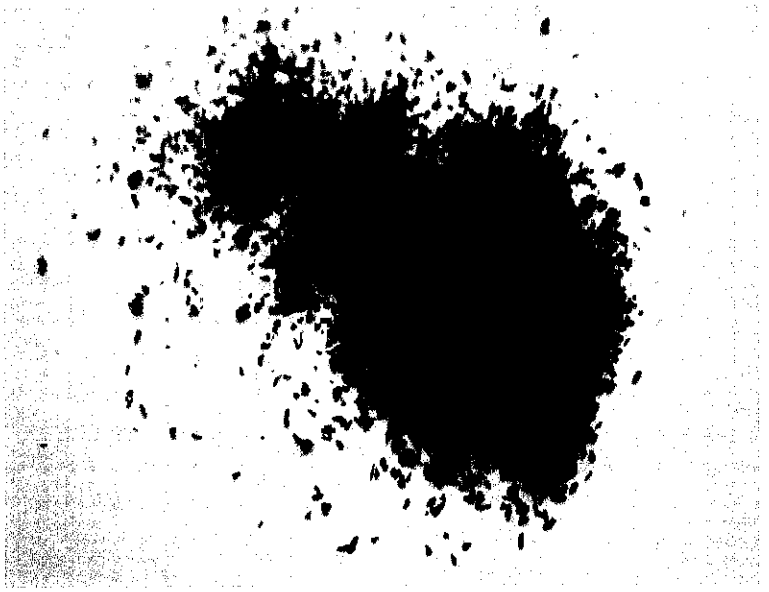

Figure 16: Rubber Seed Shell Activated Carbon

3.3.3 Characterization of the Activated Carbon

The produced activated carbon is then brought to the characterization stage, where we are using the Field Emission Scanning Electron Microscopy and Micromeritics ASAP 2020 surface area and porosity analyzer. The obtained results from these test will contribute in finding which sample to have the higher adsorption capacity based on their active surface area, pore volume and pore diameter.

3.3.4 Adsorption Test

To determine the time required for the adsorbent (rubber seed shell activated carbon) to reach adsorption equilibrium the experiment is been done by batch studies. For this study, the wastewater solution taken from the refinery desalter unit, it acts as the adsorbates. Kinetic test using different predetermined concentrations of the wastewater were performed on rubber seed shell (RSS) activated carbon. The batch technique is being used to obtain a simple and effective data; this is due to time constraints to our project. Initial concentrations of oil and grease in the wastewater

were taken before being tested or mixing with the RSS activated carbon. For each adsorption data point, the mass amount of RSS activated carbon and the predetermined concentration of wastewater solution are as below;

Table 3: Concentration of wastewater and Mass of RSS

Label	Concentration of Wastewater (mg/L)	Mass of RSS activated carbon (g)
1	250	0.19
2	375	0.15
3	500	0.12
4	625	0.08
5	750	0.05

50 ml of each concentration of wastewater was taken and mix with the RSS activated carbon in a conical flask. The conical flasks were placed and shaken mildly manually using hands for 15 minutes everyday to attain equilibrium after about 10 days of contact time. During the whole experiment the samples were covered and filtrate concentration were taken periodically at day 1, 3, 5, 7, 10 and oil and grease concentration is determined using InfraCal TOG/TPH Analyzer Model HATR-T2.

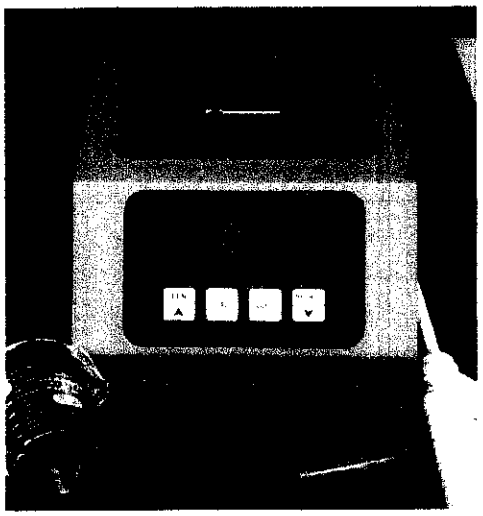

Figure 17: Oil and Grease Analyzer

Figure 18: Sample Bottle

The oil and grease analyser is used to detect the concentration of oil and grease in (mg/l) from the filtrate. The steps for the test are as below:

1. Collect the measured amount of sample
2. Add a measured amount of hexane (1 hexane : 9 filtrate)
3. Shake for two minutes
4. Take 50 μm from the top hexane layer (as figure 19) with a pipette or syringe
5. Eject the hexane onto the centre of the built in sample plate

After selecting the run button, the timer countdown will begin. This will allow the hexane to evaporate, so only the sample hydrocarbon will be measured. When the countdown has completed, the run cycle will automatically begins and less than 30 seconds the results will appear.

The equilibrium adsorption uptake and percentage removal of oil and grease from the aqueous solution, q (mg/g) was determined or calculated using the following relationship:

Amount adsorbed $q = V(Co-C)/W$ (1)
(mg of adsorbate/ g of adsorbent)

Co = initial sorbate concentration (mg/l)
C = equilibrium sorbate concentration (mg/l)
V = volume of solution (l)
W = mass of the adsorbent (g)

Chapter 4

Results and Discussion

Particle size effect on activated carbon adsorption capacity

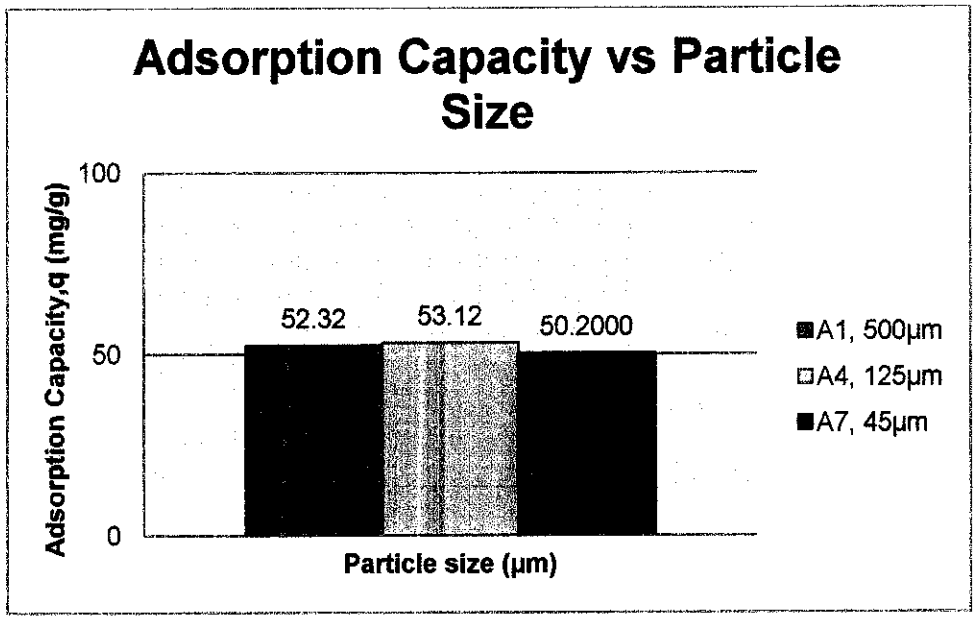

Figure 19 Adsorption Capacity vs Particle Size

The result in figure 19 for the effect of particle size towards RSS adsorption shows not significant because the difference is small. This result show that the adsorption capacities does not determined by the initial particle size of the raw material, instead it is affected by the activation process parameters such as activation time and temperatures as also being proven by (Borhan, 2010). Single Point BET surface area (S_{BET}), total pore volume (V_T) and average pore diameter (D) of the resulting activated carbon are listed in Table 4 below. Initial concentration of oil and grease in each wastewater sample tested for adsorption was measured at 350 mg/l.

Table 4: Preparation condition and results of RSS activated carbon

Sample	Act. Time (hr)	Act. Temp (°C)	Impreg. Ratio	Specific surface area, S_{BET} (m^2/g)	total pore volume, V_T (cc/g)	average pore diameter, D (A)
A1	2 hr	500 °C	1 : 1	**5.694×10^{-1}**	**4.637×10^{-3}**	**1.613×10^{1}**
A4	2 hr	500 °C	1 : 1	**1.020**	**8.587×10^{-3}**	**2.508×10^{1}**
A7	2 hr	500 °C	1 : 1	**1.540**	**1.158×10^{-2}**	**1.611×10^{1}**
A2	2 hr	500 °C	1 : 20	**2.014×10^{-1}**	**1.748×10^{-3}**	**1.613×10^{1}**
A3	2 hr	500 °C	1 : 10	**3.050×10^{-1}**	**2.557×10^{-3}**	**1.612×10^{1}**
C1	2 hr	400 °C	1 : 1	**4.155×10^{-1}**	**3.687×10^{-3}**	**1.091×10^{1}**
E1	1 hr	500 °C	1 : 1	**2.450**	**3.850×10^{-3}**	**6.287×10^{1}**
D1	1.5 hr	500 °C	1 : 1	**5.886**	**7.034×10^{-3}**	**4.781×10^{1}**
A9	1.5 hr	500 °C	1 : 10	**9.932×10^{1}**	**1.621×10^{-1}**	**6.530×10^{1}**

4.2 Impregnation ratio (RSSC : Acid) effect on activated carbon adsorption capacity

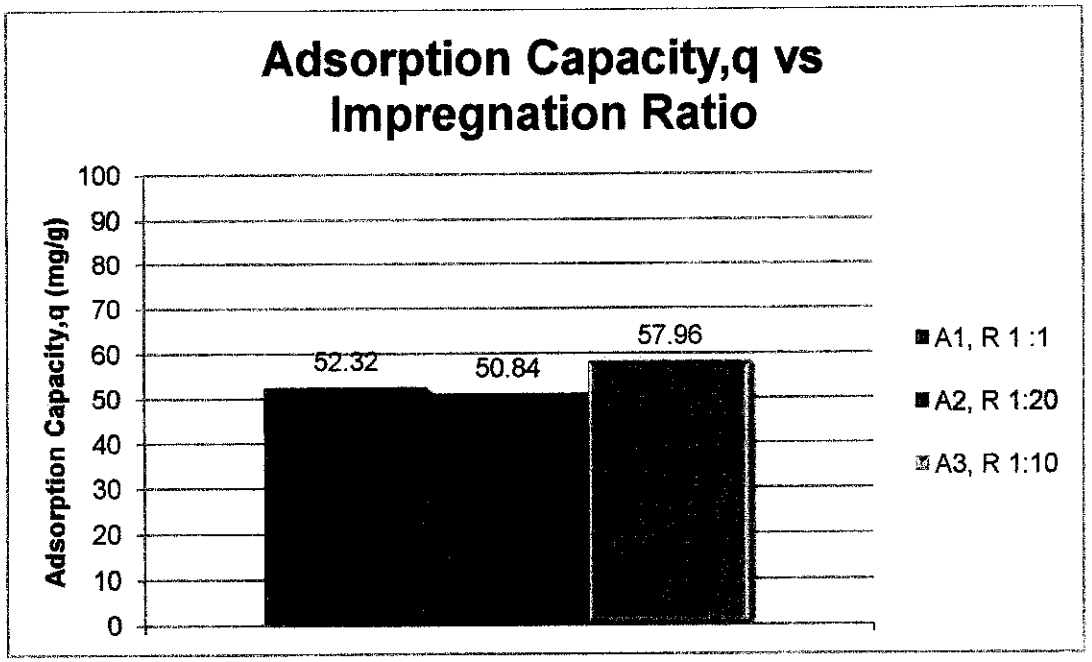

Figure 20 Adsorption Capacity vs Impregnation Ratio

The figure above tells us that higher impregnation ratio between sample A3 and A1 shows a higher adsorption capacity, this maybe come to the effect of higher acid concentration would enhance porosity development. From this also we can see a negative trend on adsorption capacity that shows too high concentration of phosphoric acid can be damaging to the structure or formation of polyphosphate layer acting like a skin covering the pore structure. This also can be observed in the findings of (H. M. Al-Swaidan, 2011).

4.3 Activation Temperature effect on activated carbon adsorption capacity

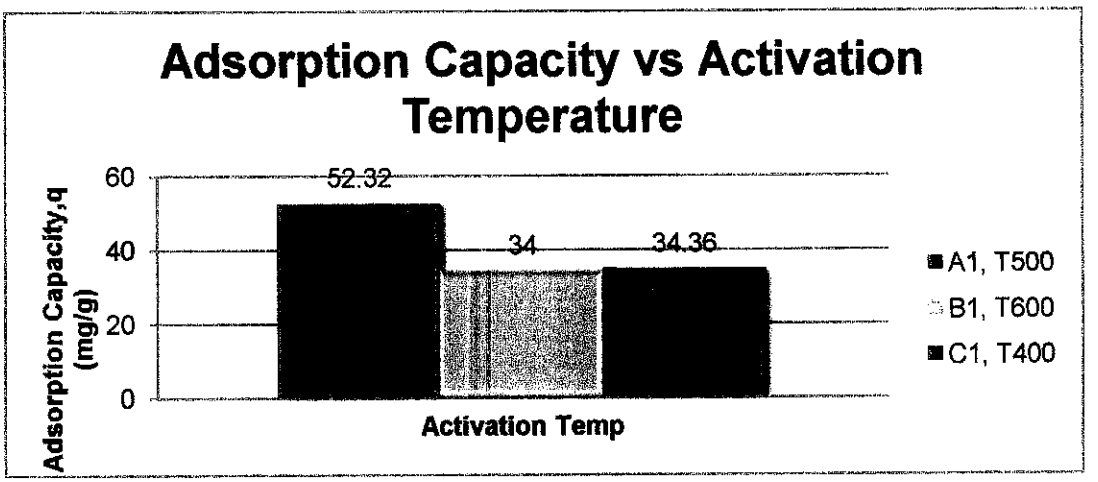

Figure 21 Adsorption Capacity vs Activation Temperature

From figure 21 it shows that at temperature 500°C we get the highest adsorption capacity. We get lower reading at 400°C because at that temperature the RSS activated carbon was prematurely activated, the pores are not fully developed which means most of them are shallow pores. At higher temperature, micropores were enlarged and the walls between pores collapsed and formed mesopores (Sun, 2010). However at 600°C the excessive heat energy given to the carbon resulting in the knocking and breaking of some porous wall, thus blocking the porosity formation (Borhan, 2010). (Ahamadpour, 1996) also finds that the increase in temperature from 500°C -800°C may induce shrinkage in carbon structure, resulting in a reduction in porosity. Therefore the adsorption capacity here shows the lowest among the three samples compared. Specific surface areas, S_{BET} also supports the finding that A1 has a higher value than C1, 5.694×10^{-1}, 4.155×10^{-1} respectively.

4.4 Activation Time effect on activated carbon adsorption capacity

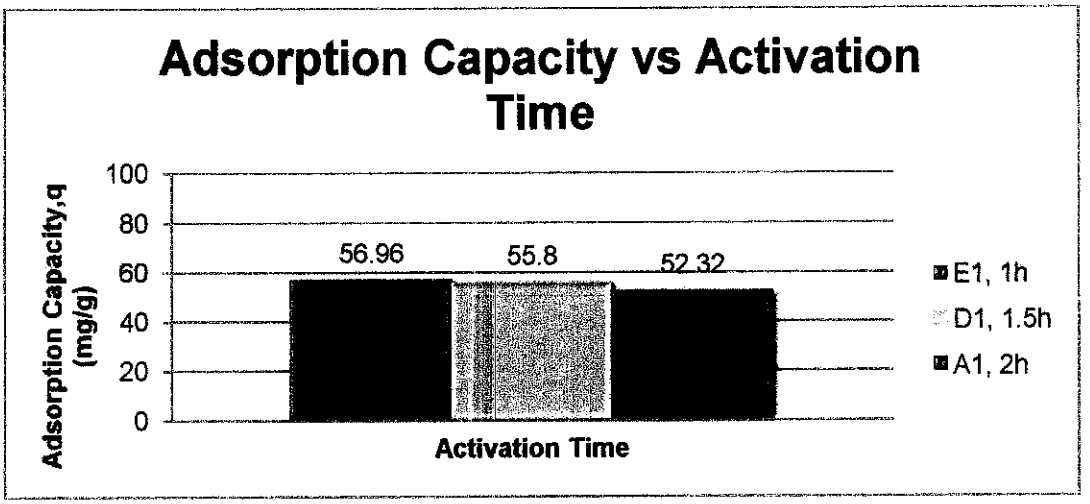

Figure 22: Adsorption Capacity vs Activation Time

It is found in this study that the optimum activation time is 1.5 hours which yield the most Specific surface area, S_{BET} and a larger average pore diameter, D of 5.886 and 4.781×10^{1} respectively on sample D1 in table 4 in comparison with E1 and A1. Although in figure 22 it shows E1 have a slight higher adsorption capacity, this may due to error during adsorption test, where the bottle sample was not properly cleaned with soap before being used for oil and grease test. A higher activation time of 2h shows a much lower reading of adsorption capacity, a study by (Borhan, 2010) proves that prolonged activation also may cause over activation, accelerating surface erosion more than pore formation.

4.5 N2 adsorption and pore size distribuition.

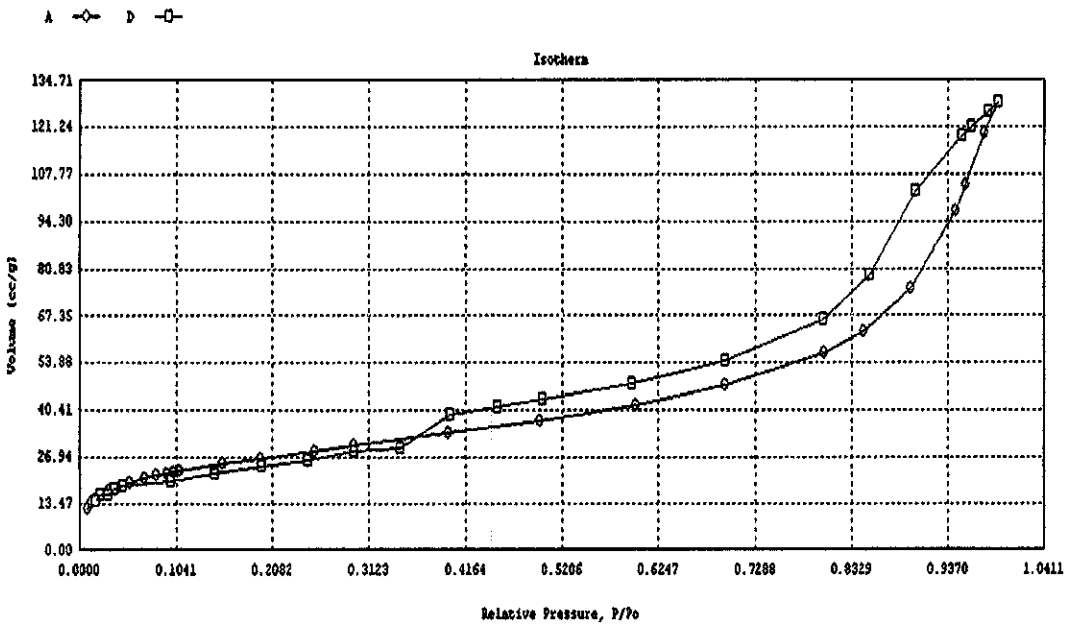

Figure 23: Isotherm

From the isotherm taken from the highest Specific surface area, S_{BET} sample A9, we can see that ranging from 0 to 0.3 is considered as an area rich with micropores which are many in numbers and considered shallow do to its small volume. A significant amount of micropore usually ranging from 50 cc/g and above to be considered as a micropore rich surface. Therefore, from this sample taken, the number of micropores are considered many and concentrated in its distributions. The adsorption isotherm shape is classified as type II. This type of isotherm indicates an indefinite multi-layer formation after completion of the monolayer and is found in adsorbents with wide distributions of pore sizes. Near to the first point of inflexion a monolayer is completed, following which adsorption occurs in successive layers.

Meanwhile ranging from 0.3 to 0.7 relative pressures we can see hysteresis type IV slope of the desorption plot. A hysteresis slope is when there are significant gap between adsorption and desorption plot at the same relative pressure point. These relative pressure ranges mostly are filled or developed with mesoporous pores. A higher volume when desorption is due to during adsorption cold nitrogen gas that enters the pores liquefy due to difference in atmospheric pressure; and during

molecule by molecule desorption it turns as semi liquid which has a bigger volume than nitrogen in gas condition.

For 0.7 and above range it basically shows the pore volume and pore shape of the adsorbent. The figure shows it around 124 cc/g maximum point which translates to a moderate/low and shallow pores. Usually 300 cc/g volume gives a moderate/high and deep pores. From this we can assume the total volume of our adsorbent. The pore shape is generally assumed to be either cylindrical or slit-shaped.

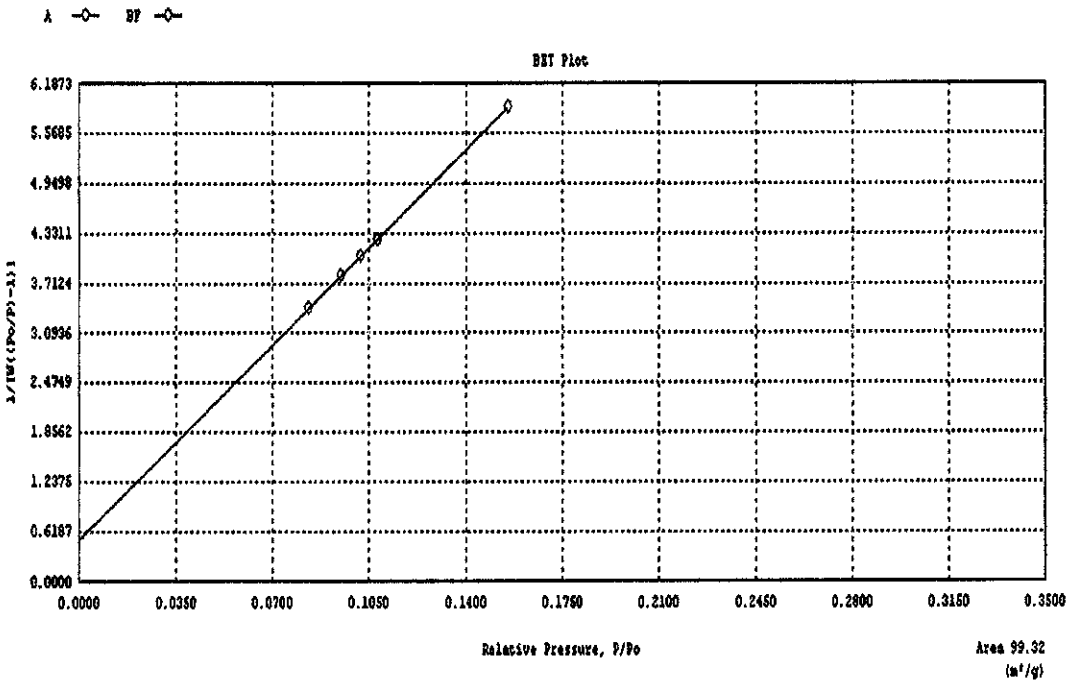

Figure 24: BET Plot

From the BET plot we can get the total surface area of our adsorbent by calculating the gradient value from this plot. While from the while intercept we can get the C value, if its equal to zero means there is none micropores, if its in positive value therefore there are micropores and if the reading is negative means the BET plot is not accurate and needs a redo.

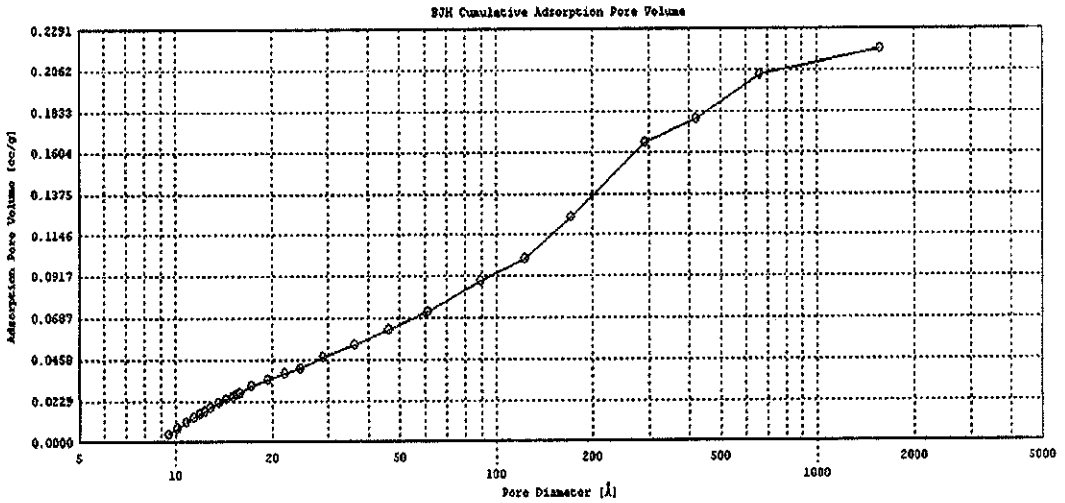

Figure 25: BJH Cumulative Adsorption Pore Volume

The figure shows highly of mesoporous distribution. Pore diameter ranging from 10-20 A shows the micropores area, which are highly congested which will not bring a significant value to the specific surface area. For mesopores range that is between 20-50 A shows a rather small amount and unscattered. A good adsorbent usually needs to be high density, scattered and a lot of pores. The presence of high mesopores and macropores will make a low specific surface area for adsorbent. This hanging graph also shows a number of macropores which again gives no contribution to specific surface area.

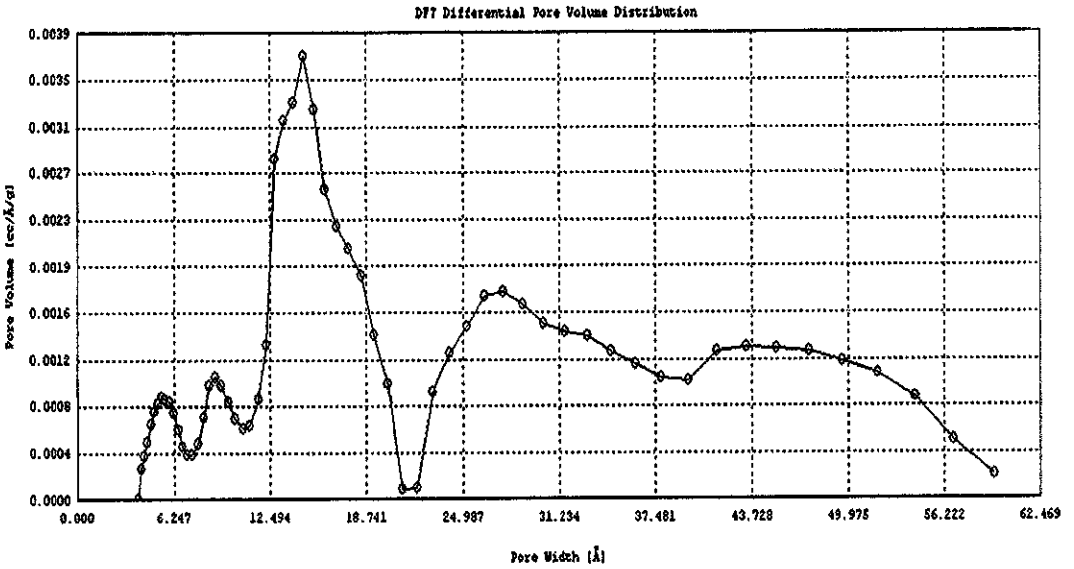

Figure 26: Differential Pore Volume Distribution

From 0-20 A showed a high peak of micropores range which gives us a small amount of micropores with big volume. There are two type of pore size distribution shown here, the sharp shaped is less in distribution but high population of micropores. This is suitable as adsorbent for static effluent usage. Meanwhile, the broad shaped gives a large distribution but a low population of mesopores. A good adsorbent for flowing effluent system is the broad type pore size distribution, because it is easier to trap effluent.

4.6 Field Emission Scanning Electron Microscope

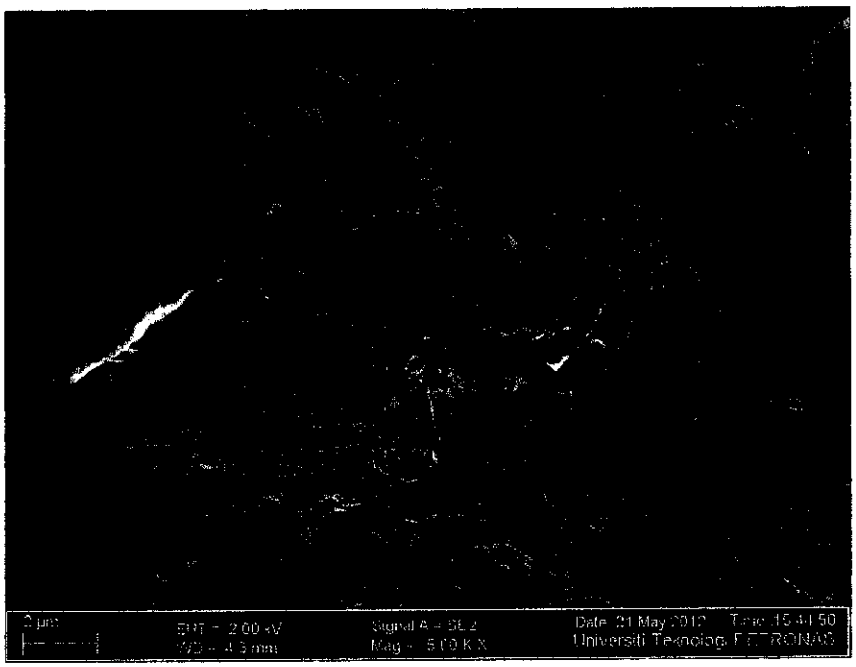

Figure 27: Fresh Rubber Seed Shell (Dried)

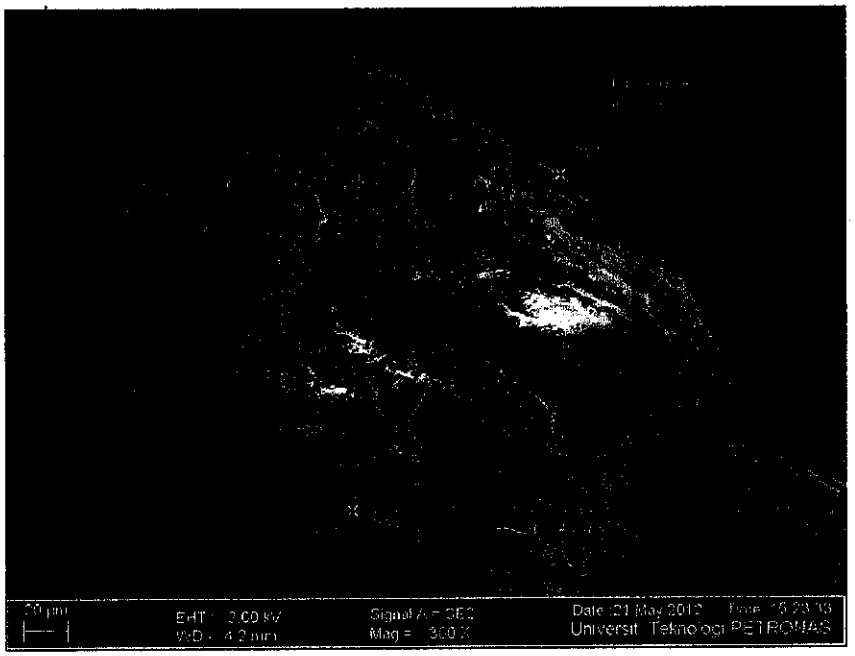

Figure 28: Fresh Rubber Seed Shell (Dried)

The microstructure of the raw rubber seed shell showed clearly the canal structure, pores sites, which is a good and potential texture for preparing activated carbon because activating agents can easily get contact with the inside surfaces.

Table 5 Element composition

Element	Weight%	Atomic%
C K	40.56	48.08
O K	57.22	50.92
Si K	1.37	0.70
K K	0.51	0.19
Ca K	0.33	0.12
Totals	100	

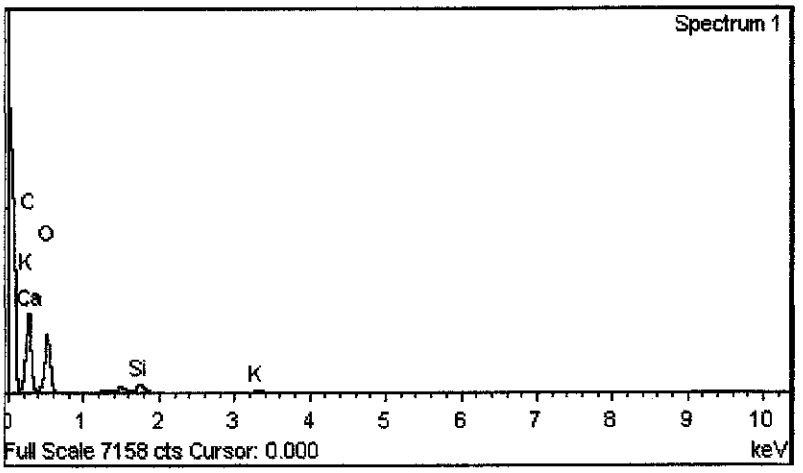

Figure 29: Element composition

Energy dispersive x-Ray (EDX) analysis identified the elemental composition of the raw rubber seed shell as above shows the concentration of each element in the raw rubber seed shell.

Chapter 5

Conclusion and Recommendations

5.1 Conclusion

The main objective is to make full use of rubber seed shell, an agricultural waste and using it as carbon molecular sieve adsorbents on a project to study the removal of oil & grease from the desalter's effluent water. 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. This will also proved to be a mutual situation for the rubber industry also as it can generate more income by adding value to the once considered waste; rubber seed shell.

It is found that phosphoric acid acts as a dehydrating agent that more to cleaning the surface of the activated carbon, it also had promote pore widening effect producing more shallow and wide mesoporous rich sites which makes a low specific surface area, thus low micropores sites. Results also showed that particle size of raw material does not really affect the properties of the activated carbon. While activation time, impregnation ratio and activation temperature gives a significant effect on activated carbon's surface area, total pore volume and as well pore diameters.

Although having a low specific surface area, adsorption studies shows that RSS activated carbon can be used as adsorbents for the removal of oil and grease but ineffective because the contents of oil and grease after adsorption is still above Standard A of not detectable and Standard B at 10 mg/l under the Environmental Quality (Sewage and Industrial Effluents) Regulations 1979. By taking an average of 50.21 mg/l final concentration after adsorption and comparing with 350 mg/l of initial concentration of oil and grease in the wastewater sample, it gives the RSSC 85.7 % effectiveness.

In conclusion the rubber seed shell coat activated carbon is a potential alternative precursor for variants of activated carbon already available and ongoing research

today. Further research on finding the most cost effective and suitable parameters to produce highly distributed concentrated high surface area rubber seed shell activated carbon.

5.2 Recommendations

Below are some recommendations to be implemented for future works in the scope of this project area:

1. For future FYP students and laboratory management to have a web-based system that enables updates and helps both students and lab technician to ease equipment booking, buying chemicals and lab apparatus.
2. To diversify and find more natural source, organic waste that can be benefited by human example grass, hay, dead flowers etc
3. Allow more access to do lab characterization for FYP students.

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APPENDICES

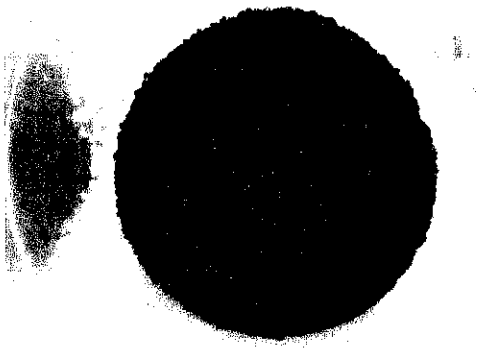

Figure 31: RSS Activated Carbon

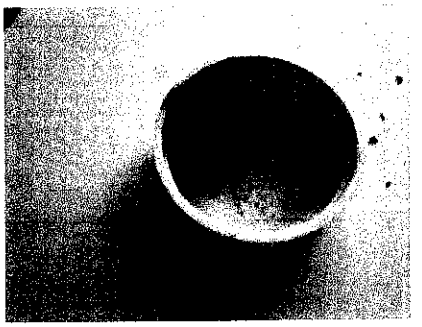

Figure 30: RSS Activated Carbon

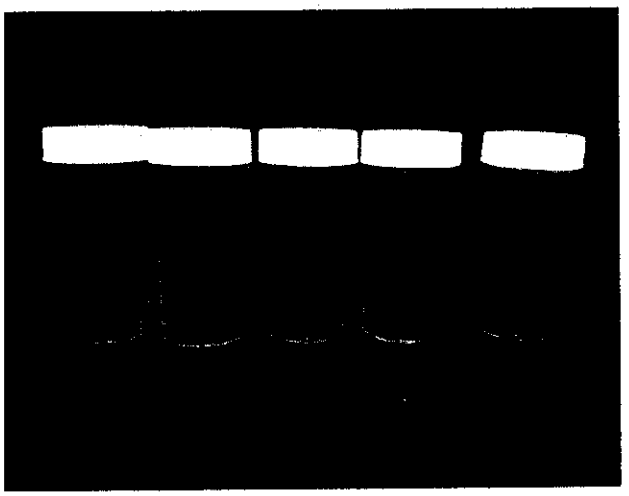

Figure 32: Adsorption Color Change

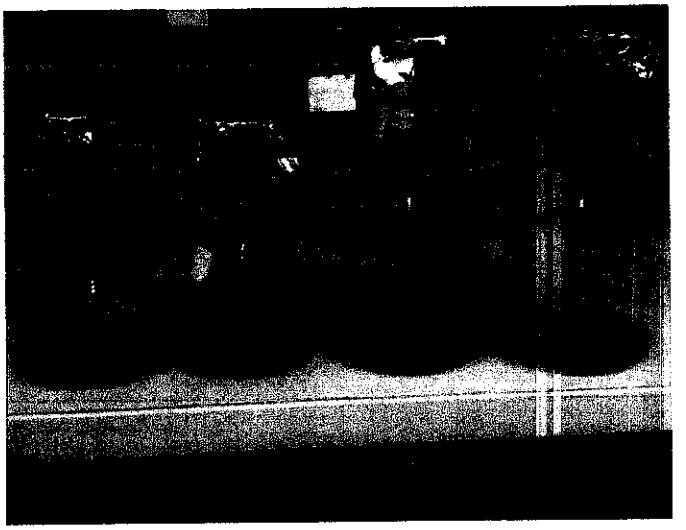

Figure 33: Adsorption

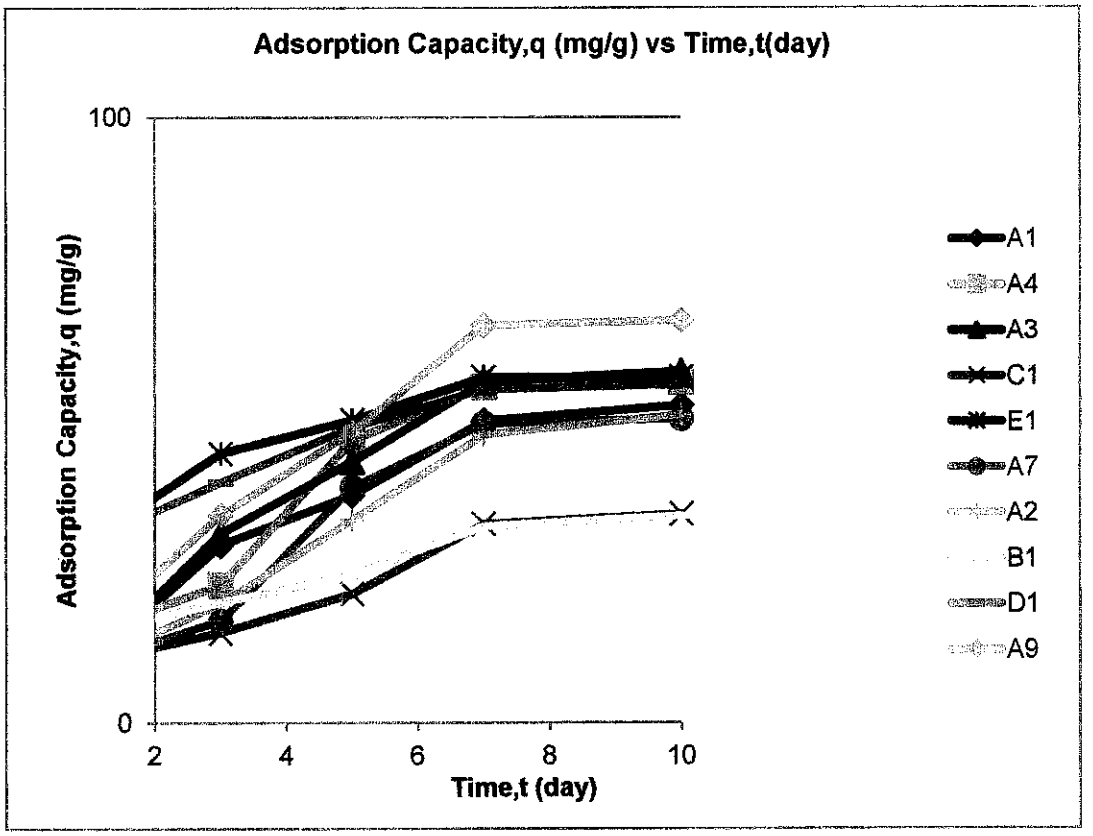

Figure 34 Adsorption Capacity vs Time

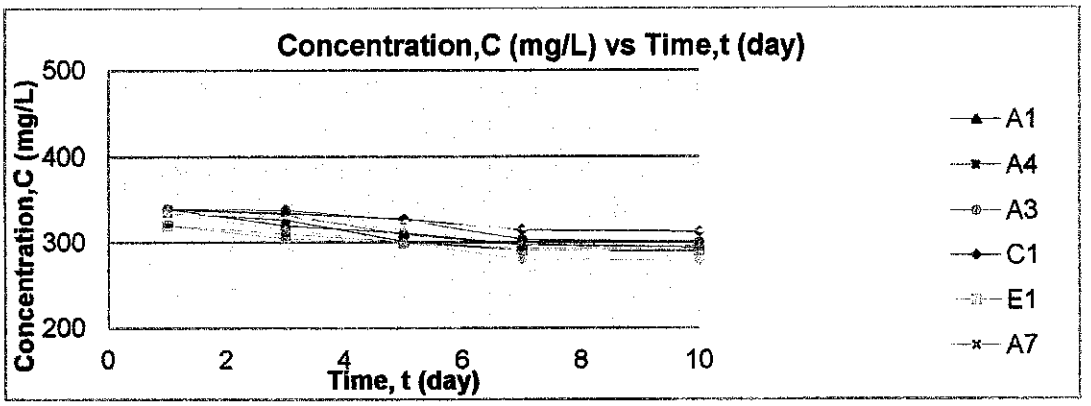

Figure 35 Concentration vs Time

Table 6: Example adsorption calculation for sample A1

First day of experiment:	SAMPLE A1	16-Jun-12	
Sampling time:	DAY 2 Saturday, 18 June 2011: 10:00 AM		
Initial volume of sample:	V=	50	mL
Initila concentration of sample:	C_o=	350	mg/L
Accumulated adsorbed amount:			

day 1

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.1	0.19	50	94.0000	67.3684	67.3684
EQPKS1.2	0.15	50	146.0000	68.0000	68.0000
EQPKS1.3	0.12	50	223.0000	52.9167	52.9167
EQPKS1.4	0.08	50	312.0000	23.7500	23.7500
EQPKS1.5	0.05	50	340.0000	10.0000	10.0000

Sampling time: **DAY 3**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.7	0.19	48.0	81.0000	3.2842	70.6526
EQPKS1.8	0.15	48.0	133.0000	4.1600	72.1600
EQPKS1.9	0.12	48.0	220.0000	1.2000	54.1167
EQPKS1.10	0.08	48.0	300.0000	7.2000	30.9500
EQPKS1.11	0.05	48.0	320.0000	19.2000	29.2000

Sampling time: DAY 5

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.13	0.19	46.0	73.0000	1.9368	72.5895
EQPKS1.14	0.15	46.0	113.0000	6.1333	78.2933
EQPKS1.15	0.12	46.0	209.0000	4.2167	58.3333
EQPKS1.16	0.08	46.0	288.0000	6.9000	37.8500
EQPKS1.17	0.05	46.0	311.0000	8.2800	37.4800

Sampling Time: **DAY7**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	44.0	69.0000	0.9263	73.5158
EQPKS1.20	0.15	44.0	108.0000	1.4667	79.7600
EQPKS1.21	0.12	44.0	202.0000	2.5667	60.9000
EQPKS1.22	0.08	44.0	280.0000	4.4000	42.2500
EQPKS1.23	0.05	44.0	297.0000	12.3200	49.8000

day 10

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)

EQPKS1.19	0.19	42.0	68.0000	0.2211	73.7368
EQPKS1.20	0.15	42.0	107.0000	0.2800	80.0400
EQPKS1.21	0.12	42.0	200.0000	0.7000	61.6000
EQPKS1.22	0.08	42.0	280.0000	0.0000	42.2500
EQPKS1.23	0.05	42.0	294.0000	2.5200	52.3200

Table 7:Example adsorption calculation for sample A2

First day of experiment: SAMPLE A2 16-Jun-12
Sampling time: DAY 2 Saturday, 18 June 2012: 10:00 AM
Initial volume of sample: V= 50 mL
Initila concentration of sample: C_o= 350 mg/L
Accumulated adsorbed amount:

day 1

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.1	0.19	50	100.0000	65.7895	65.7895
EQPKS1.2	0.15	50	150.0000	66.6667	66.6667
EQPKS1.3	0.12	50	223.0000	52.9167	52.9167
EQPKS1.4	0.08	50	312.0000	23.7500	23.7500
EQPKS1.5	0.05	50	340.0000	10.0000	10.0000

Sampling time: **DAY 3**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.7	0.19	48.0	90.0000	2.5263	68.3158
EQPKS1.8	0.15	48.0	133.0000	5.4400	72.1067
EQPKS1.9	0.12	48.0	220.0000	1.2000	54.1167
EQPKS1.10	0.08	48.0	308.0000	2.4000	26.1500
EQPKS1.11	0.05	48.0	333.0000	6.7200	16.7200

Sampling time: DAY 5

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.13	0.19	46.0	87.0000	0.7263	69.0421
EQPKS1.14	0.15	46.0	120.0000	3.9867	76.0933
EQPKS1.15	0.12	46.0	209.0000	4.2167	58.3333
EQPKS1.16	0.08	46.0	290.0000	10.3500	36.5000
EQPKS1.17	0.05	46.0	309.0000	22.0800	38.8000

Sampling Time: **DAY7**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	44.0	84.0000	0.6947	69.7368

EQPKS1.20	0.15	44.0	110.0000	2.9333	79.0267
EQPKS1.21	0.12	44.0	202.0000	2.5667	60.9000
EQPKS1.22	0.08	44.0	280.0000	5.5000	42.0000
EQPKS1.23	0.05	44.0	301.0000	7.0400	45.8400

day 10

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	42.0	83.0000	0.2211	69.9579
EQPKS1.20	0.15	42.0	109.0000	0.2800	79.3067
EQPKS1.21	0.12	42.0	200.0000	0.7000	61.6000
EQPKS1.22	0.08	42.0	278.0000	1.0500	43.0500
EQPKS1.23	0.05	42.0	300.0000	0.8400	46.6800

Table 8:Example adsorption calculation for sample A3

First day of experiment: 16-Jun-12

SAMPLE

Sampling time: A3 DAY 2 Saturday, 18 June 2012: 10:00 AM

Initial volume of sample: V= 50 mL

Initila concentration of sample: C_o= 350 mg/L

Accumulated adsorbed amount:

day 1

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.1	0.19	50	97.0000	66.5789	66.5789
EQPKS1.2	0.15	50	146.0000	68.0000	68.0000
EQPKS1.3	0.12	50	228.0000	50.8333	50.8333
EQPKS1.4	0.08	50	312.0000	23.7500	23.7500
EQPKS1.5	0.05	50	340.0000	10.0000	10.0000

Sampling time: **DAY 3 Tuesday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.7	0.19	48.0	92.0000	1.2632	67.8421
EQPKS1.8	0.15	48.0	133.0000	4.1600	72.1600
EQPKS1.9	0.12	48.0	226.0000	0.8000	51.6333
EQPKS1.10	0.08	48.0	308.0000	2.4000	26.1500
EQPKS1.11	0.05	48.0	339.0000	0.9600	10.9600

Sampling time: DAY 5 Thursday,

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.13	0.19	46.0	73.0000	4.6000	72.4421
EQPKS1.14	0.15	46.0	113.0000	6.1333	78.2933
EQPKS1.15	0.12	46.0	218.0000	3.0667	54.7000
EQPKS1.16	0.08	46.0	292.0000	9.2000	35.3500
EQPKS1.17	0.05	46.0	327.0000	11.0400	22.0000

Sampling Time: **DAY7 Sunday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	44.0	70.0000	0.6947	73.1368
EQPKS1.20	0.15	44.0	110.0000	0.8800	79.1733
EQPKS1.21	0.12	44.0	202.0000	5.8667	60.5667
EQPKS1.22	0.08	44.0	280.0000	6.6000	41.9500
EQPKS1.23	0.05	44.0	303.0000	21.1200	43.1200

day 10

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	42.0	70.0000	0.0000	73.1368
EQPKS1.20	0.15	42.0	109.0000	0.2800	79.4533
EQPKS1.21	0.12	42.0	200.0000	0.7000	61.2667
EQPKS1.22	0.08	42.0	280.0000	0.0000	41.9500
EQPKS1.23	0.05	42.0	300.0000	2.5200	45.6400

Table 9:Example adsorption calculation for sample A4

First day of experiment:	Sample A4	16-Jun-12	
Sampling time:		DAY 2 Saturday, 18 June 2012: 10:00 AM	
Initial volume of sample:	V=	50	mL
Initila concentration of sample:	C_o=	350	mg/L
Accumulated adsorbed amount:			

day 1

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.1	0.19	50	100.0000	65.7895	65.7895
EQPKS1.2	0.15	50	150.0000	66.6667	66.6667
EQPKS1.3	0.12	50	223.0000	52.9167	52.9167
EQPKS1.4	0.08	50	312.0000	23.7500	23.7500
EQPKS1.5	0.05	50	335.0000	15.0000	15.0000

Sampling time: **DAY 3 Tuesday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.7	0.19	48.0	95.0000	1.2632	67.0526
EQPKS1.8	0.15	48.0	133.0000	5.4400	72.1067
EQPKS1.9	0.12	48.0	220.0000	1.2000	54.1167
EQPKS1.10	0.08	48.0	320.0000	-4.8000	18.9500
EQPKS1.11	0.05	48.0	327.0000	7.6800	22.6800

Sampling time: DAY 5 Thursday,

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.13	0.19	46.0	92.0000	0.7263	67.7789
EQPKS1.14	0.15	46.0	113.0000	6.1333	78.2400
EQPKS1.15	0.12	46.0	209.0000	4.2167	58.3333
EQPKS1.16	0.08	46.0	288.0000	18.4000	37.3500
EQPKS1.17	0.05	46.0	301.0000	23.9200	46.6000

Sampling Time: **DAY7**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	44.0	89.0000	0.6947	68.4737
EQPKS1.20	0.15	44.0	110.0000	0.8800	79.1200
EQPKS1.21	0.12	44.0	202.0000	2.5667	60.9000
EQPKS1.22	0.08	44.0	282.0000	3.3000	40.6500
EQPKS1.23	0.05	44.0	291.0000	8.8000	55.4000

day 10

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	42.0	88.0000	0.2211	68.6947
EQPKS1.20	0.15	42.0	109.0000	0.2800	79.4000
EQPKS1.21	0.12	42.0	200.0000	0.7000	61.6000
EQPKS1.22	0.08	42.0	280.0000	1.0500	41.7000
EQPKS1.23	0.05	42.0	290.0000	0.8400	56.2400

Table 10: Example adsorption calculation for sample A7

First day of experiment:	Sample A7	16-Jun-12	
Sampling time:		DAY 2 Saturday, 18 June 2012: 10:00 AM	
Initial volume of sample:	V=	50	mL
Initila concentration of sample:	C_o=	350	mg/L
Accumulated adsorbed amount:			

day 1

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.1	0.19	50	93.0000	67.6316	67.6316
EQPKS1.2	0.15	50	146.0000	68.0000	68.0000
EQPKS1.3	0.12	50	223.0000	52.9167	52.9167
EQPKS1.4	0.08	50	313.0000	23.1250	23.1250
EQPKS1.5	0.05	50	340.0000	10.0000	10.0000

Sampling time: **DAY 3 Tuesday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)

EQPKS1.7	0.19	48.0	86.0000	1.7684	69.4000
EQPKS1.8	0.15	48.0	140.0000	1.9200	69.9200
EQPKS1.9	0.12	48.0	219.0000	1.6000	54.5167
EQPKS1.10	0.08	48.0	299.0000	8.4000	31.5250
EQPKS1.11	0.05	48.0	333.0000	6.7200	16.7200

Sampling time: DAY 5 Thursday,

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.13	0.19	46.0	75.0000	2.6632	72.0632
EQPKS1.14	0.15	46.0	122.0000	5.5200	75.4400
EQPKS1.15	0.12	46.0	208.0000	4.2167	58.7333
EQPKS1.16	0.08	46.0	290.0000	5.1750	36.7000
EQPKS1.17	0.05	46.0	309.0000	22.0800	38.8000

Sampling Time: **DAY7 Sunday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	44.0	69.0000	1.3895	73.4526
EQPKS1.20	0.15	44.0	111.0000	3.2267	78.6667
EQPKS1.21	0.12	44.0	207.0000	0.3667	59.1000
EQPKS1.22	0.08	44.0	286.0000	2.2000	38.9000
EQPKS1.23	0.05	44.0	301.0000	7.0400	45.8400

day 10

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	42.0	68.0000	0.2211	73.6737
EQPKS1.20	0.15	42.0	111.0000	0.0000	78.6667
EQPKS1.21	0.12	42.0	206.0000	0.3500	59.4500
EQPKS1.22	0.08	42.0	287.0000	-0.5250	38.3750
EQPKS1.23	0.05	42.0	300.0000	0.8400	46.6800

Table 11: Example adsorption calculation for sample A9

First day of experiment:	Sample A9	16-Jun-12	
Sampling time:		DAY 2 Saturday, 18 June 2011: 10:00 AM	
Initial volume of sample:	V=	50	mL
Initila concentration of sample:	C_o=	350	mg/L
Accumulated adsorbed amount:			

day 1

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.1	0.19	50	90.0000	68.4211	68.4211
EQPKS1.2	0.15	50	140.0000	70.0000	70.0000

EQPKS1.3	0.12	50	220.0000	54.1667	54.1667
EQPKS1.4	0.08	50	310.0000	25.0000	25.0000
EQPKS1.5	0.05	50	335.0000	15.0000	15.0000

Sampling time: **DAY 3 Tuesday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.7	0.19	48.0	81.0000	2.2737	70.6947
EQPKS1.8	0.15	48.0	133.0000	2.2400	72.2400
EQPKS1.9	0.12	48.0	210.0000	4.0000	58.1667
EQPKS1.10	0.08	48.0	300.0000	6.0000	31.0000
EQPKS1.11	0.05	48.0	315.0000	19.2000	34.2000

Sampling time: DAY 5 Thursday,

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.13	0.19	46.0	73.0000	1.9368	72.6316
EQPKS1.14	0.15	46.0	113.0000	6.1333	78.3733
EQPKS1.15	0.12	46.0	198.0000	4.6000	62.7667
EQPKS1.16	0.08	46.0	288.0000	6.9000	37.9000
EQPKS1.17	0.05	46.0	300.0000	13.8000	48.0000

Sampling Time: **DAY7 Sunday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	44.0	63.0000	2.3158	74.9474
EQPKS1.20	0.15	44.0	100.0000	3.8133	82.1867
EQPKS1.21	0.12	44.0	190.0000	2.9333	65.7000
EQPKS1.22	0.08	44.0	280.0000	4.4000	42.3000
EQPKS1.23	0.05	44.0	280.0000	17.6000	65.6000

day 10

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	42.0	62.0000	0.2211	75.1684
EQPKS1.20	0.15	42.0	100.0000	0.0000	82.1867
EQPKS1.21	0.12	42.0	190.0000	0.0000	65.7000
EQPKS1.22	0.08	42.0	279.0000	0.5250	42.8250
EQPKS1.23	0.05	42.0	279.0000	0.8400	66.4400

Table 12:Example adsorption calculation for sample B1

First day of experiment:	Sample B1	16-Jun-12
Sampling time:		DAY 2 Saturday, 18 June 2012: 10:00 AM
Initial volume of sample:	V=	50 mL

Initila concentration of sample: C_o= 350 mg/L

Accumulated adsorbed amount:

day 1

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.1	0.19	50	93.0000	67.6316	67.6316
EQPKS1.2	0.15	50	150.0000	66.6667	66.6667
EQPKS1.3	0.12	50	224.0000	52.5000	52.5000
EQPKS1.4	0.08	50	312.0000	23.7500	23.7500
EQPKS1.5	0.05	50	335.0000	15.0000	15.0000

Sampling time: **DAY 3 Tuesday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.7	0.19	48.0	81.0000	3.0316	70.6632
EQPKS1.8	0.15	48.0	133.0000	5.4400	72.1067
EQPKS1.9	0.12	48.0	220.0000	1.6000	54.1000
EQPKS1.10	0.08	48.0	300.0000	7.2000	30.9500
EQPKS1.11	0.05	48.0	330.0000	4.8000	19.8000

Sampling time: DAY 5 Thursday,

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.13	0.19	46.0	72.0000	2.1789	72.8421
EQPKS1.14	0.15	46.0	110.0000	7.0533	79.1600
EQPKS1.15	0.12	46.0	209.0000	4.2167	58.3167
EQPKS1.16	0.08	46.0	288.0000	6.9000	37.8500
EQPKS1.17	0.05	46.0	315.0000	13.8000	33.6000

Sampling Time: **DAY7 Sunday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	44.0	68.0000	0.9263	73.7684
EQPKS1.20	0.15	44.0	108.0000	0.5867	79.7467
EQPKS1.21	0.12	44.0	205.0000	1.4667	59.7833
EQPKS1.22	0.08	44.0	270.0000	9.9000	47.7500
EQPKS1.23	0.05	44.0	299.0000	14.0800	47.6800

day 10

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	42.0	68.0000	0.0000	73.7684
EQPKS1.20	0.15	42.0	106.0000	0.5600	80.3067
EQPKS1.21	0.12	42.0	204.0000	0.3500	60.1333
EQPKS1.22	0.08	42.0	273.0000	-1.5750	46.1750
EQPKS1.23	0.05	42.0	295.0000	3.3600	51.0400

Table 13: Example adsorption calculation for sample C1

First day of experiment:	Sample C1	16-Jun-12	
Sampling time:		DAY 2 Saturday, 18 June 2012: 10:00 AM	
Initial volume of sample:		V= 50	mL
Initila concentration of sample:		C_o= 350	mg/L
Accumulated adsorbed amount:			

day 1

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.1	0.19	50	93.0000	67.6316	67.6316
EQPKS1.2	0.15	50	146.0000	68.0000	68.0000
EQPKS1.3	0.12	50	226.0000	51.6667	51.6667
EQPKS1.4	0.08	50	313.0000	23.1250	23.1250
EQPKS1.5	0.05	50	340.0000	10.0000	10.0000

Sampling time: **DAY 3 Tuesday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.7	0.19	48.0	81.0000	3.0316	70.6632
EQPKS1.8	0.15	48.0	132.0000	4.4800	72.4800
EQPKS1.9	0.12	48.0	220.0000	2.4000	54.0667
EQPKS1.10	0.08	48.0	300.0000	7.8000	30.9250
EQPKS1.11	0.05	48.0	335.0000	4.8000	14.8000

Sampling time: DAY 5 Thursday,

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.13	0.19	46.0	72.0000	2.1789	72.8421
EQPKS1.14	0.15	46.0	110.0000	6.7467	79.2267
EQPKS1.15	0.12	46.0	210.0000	3.8333	57.9000
EQPKS1.16	0.08	46.0	287.0000	7.4750	38.4000
EQPKS1.17	0.05	46.0	328.0000	6.4400	21.2400

Sampling Time: **DAY7 Sunday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	44.0	69.0000	0.6947	73.5368
EQPKS1.20	0.15	44.0	109.0000	0.2933	79.5200
EQPKS1.21	0.12	44.0	202.0000	2.9333	60.8333
EQPKS1.22	0.08	44.0	267.0000	11.0000	49.4000
EQPKS1.23	0.05	44.0	315.0000	11.4400	32.6800

day 10

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	42.0	67.0000	0.4421	73.9789
EQPKS1.20	0.15	42.0	108.0000	0.2800	79.8000
EQPKS1.21	0.12	42.0	201.0000	0.3500	61.1833
EQPKS1.22	0.08	42.0	269.0000	-1.0500	48.3500
EQPKS1.23	0.05	42.0	313.0000	1.6800	34.3600

Table 14: Example adsorption calculation for sample D1

First day of experiment: 16-Jun-12

Sampling time: Sample D1 DAY 2 Saturday, 18 June 2012: 10:00 AM

Initial volume of sample: V= 50 mL

Initila concentration of sample: C_o= 350 mg/L

Accumulated adsorbed amount:

day 1

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.1	0.19	50	90.0000	68.4211	68.4211
EQPKS1.2	0.15	50	140.0000	70.0000	70.0000
EQPKS1.3	0.12	50	220.0000	54.1667	54.1667
EQPKS1.4	0.08	50	315.0000	21.8750	21.8750
EQPKS1.5	0.05	50	320.0000	30.0000	30.0000

Sampling time: **DAY 3 Tuesday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.7	0.19	48.0	79.0000	2.7789	71.2000
EQPKS1.8	0.15	48.0	133.0000	2.2400	72.2400
EQPKS1.9	0.12	48.0	210.0000	4.0000	58.1667
EQPKS1.10	0.08	48.0	300.0000	9.0000	30.8750
EQPKS1.11	0.05	48.0	310.0000	9.6000	39.6000

Sampling time: DAY 5 Thursday,

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.13	0.19	46.0	73.0000	1.4526	72.6526
EQPKS1.14	0.15	46.0	110.0000	7.0533	79.2933
EQPKS1.15	0.12	46.0	209.0000	0.3833	58.5500
EQPKS1.16	0.08	46.0	290.0000	5.7500	36.6250
EQPKS1.17	0.05	46.0	300.0000	9.2000	48.8000

Sampling Time: **DAY7 Sunday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	44.0	60.0000	3.0105	75.6632
EQPKS1.20	0.15	44.0	100.0000	2.9333	82.2267
EQPKS1.21	0.12	44.0	202.0000	2.5667	61.1167
EQPKS1.22	0.08	44.0	280.0000	5.5000	42.1250
EQPKS1.23	0.05	44.0	293.0000	6.1600	54.9600

day 10

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	42.0	59.0000	0.2211	75.8842
EQPKS1.20	0.15	42.0	100.0000	0.0000	82.2267
EQPKS1.21	0.12	42.0	200.0000	0.7000	61.8167
EQPKS1.22	0.08	42.0	279.0000	0.5250	42.6500
EQPKS1.23	0.05	42.0	292.0000	0.8400	55.8000

Table 15: Example adsorption calculation for sample E1

First day of experiment:	Sample E1	16-Jun-12	
Sampling time:	DAY 2 Saturday, 18 June 2012: 10:00 AM		
Initial volume of sample:	V=	50	mL
Initila concentration of sample:	C_o=	350	mg/L
Accumulated adsorbed amount:			

day 1

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.1	0.19	50	98.0000	66.3158	66.3158
EQPKS1.2	0.15	50	150.0000	66.6667	66.6667
EQPKS1.3	0.12	50	223.0000	52.9167	52.9167
EQPKS1.4	0.08	50	312.0000	23.7500	23.7500
EQPKS1.5	0.05	50	320.0000	30.0000	30.0000

Sampling time: **DAY 3 Tuesday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.7	0.19	48.0	86.0000	3.0316	69.3474
EQPKS1.8	0.15	48.0	140.0000	3.2000	69.8667
EQPKS1.9	0.12	48.0	210.0000	5.2000	58.1167
EQPKS1.10	0.08	48.0	308.0000	2.4000	26.1500
EQPKS1.11	0.05	48.0	305.0000	14.4000	44.4000

Sampling time: DAY 5 Thursday,

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.13	0.19	46.0	73.0000	3.1474	72.4947

EQPKS1.14	0.15	46.0	120.0000	6.1333	76.0000
EQPKS1.15	0.12	46.0	209.0000	0.3833	58.5000
EQPKS1.16	0.08	46.0	293.0000	8.6250	34.7750
EQPKS1.17	0.05	46.0	299.0000	5.5200	49.9200

Sampling Time: **DAY7 Sunday,**

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	44.0	69.0000	0.9263	73.4211
EQPKS1.20	0.15	44.0	108.0000	3.5200	79.5200
EQPKS1.21	0.12	44.0	202.0000	2.5667	61.0667
EQPKS1.22	0.08	44.0	282.0000	6.0500	40.8250
EQPKS1.23	0.05	44.0	291.0000	7.0400	56.9600

day 10

Sample	Amount of GAC	V (ml)	C (mg/L)	Δq (mg/g)	q (mg/g)
EQPKS1.19	0.19	42.0	68.0000	0.2211	73.6421
EQPKS1.20	0.15	42.0	107.0000	0.2800	79.8000
EQPKS1.21	0.12	42.0	201.0000	0.3500	61.4167
EQPKS1.22	0.08	42.0	280.0000	1.0500	41.8750
EQPKS1.23	0.05	42.0	291.0000	0.0000	56.9600