REMOVAL OF OIL AND GREASE IN WASTEWATER USING PALM KERNEL SHELLS ACTIVATED CARBON

By

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DECLARATION

I declare that this thesis entitled "Removal of Oil and Grease in Wastewater Using Palm Kernel Shells Activated Carbon" is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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Removal of Oil and Grease in Wastewater Using Palm Kernel Shell Activated Carbon

ABSTRACT

Oil and grease wastewater is produced from many sources such as industry and municipal. The discharge of oil and grease in wastewater system causes bad effect towards the environment and also the wastewater system itself. Palm Kernel Shells (PKS), an agriculture waste was chosen to see its ability in removing oil and grease in wastewater. The PKS was carbonized in furnace (400˚C), activated with KOH and carbonized again in furnace (800˚C).The parameter affecting oil and grease removal such as different oil and grease concentration, contact time and flow rate were studied. The oil and grease removal study was conducted by the column adsorption method and followed by the gravimetric method to identify the remaining oil and grease. PKS activated carbon present at 1% oil and grease percentage show the highest removal of 99.93% of oil and grease among all other oil and grease concentrations. The slower flow rate, 1 mL/min shows the overall higher percentage removal of 99.89% of oil and grease. SEM micrograph of PKS activated carbon after adsorption oil and grease shows that oil and grease has covered the pores of the surface of the PKS activated carbon. Overall, the PKS activated carbon has the ability in removing oil and grease which indicates that PKS can be used as a natural adsorbent in removing oil and grease.

Penyingkiran Minyak dan Gris dalam Air Kumbahan mengunakan Shell Kernel Kelapa Sawit Karbon yang Diaktifkan

ABSTRAK

Minyak dan gris dalam air kumbahan dihasilkan dari pelbagai sumber contoh seperti industri dan perbandaran. Pelepasan minyak dan gris di dalam sistem air kumbahan menyebabkan kesan buruk terhadap alam sekitar dan juga sistem air kumbahan itu sendiri. Shell Kernel Kelapa Sawit (PKS), merupakan sisa pertanian yang telah dipilih untuk menyingkirkan minyak dan gris dalam air kumbahan. PKS telah dikarbonkan dengan dibakar (400℃), diaktifkan dengan KOH dan dibakar sekali lagi (800℃). Parameter yang memberikan kesan terhadap penyingkiran minyak dan gris seperti kepekatan minyak dan gris yang berbeza, masa dan kadar aliran telah dikaji. Kajian dijalankan dengan kaedah penjerapan kolum dan diikuti oleh kaedah gravimetric bagi mengenal pasti baki minyak dan gris. PKS yang ada pada kepekatan minyak dan gris 1% menunjukan peratusan penyinkiran minyak dan gris yang tertinggi di antara kepekatan minyank dan gris yang lain iaitu sebanyak 99.93%. Kadar aliran yang lebih perlahan, 1 mL/min menunjukkan penyinkiran peratusan keseleruhan minyak dan gris iaitu sebanyak 99.89%. Pengimejan SEM bagi karbon PKS selepas penjerapan minyak dan gris menunjukkan bahawa minyak dan gris telah memenuhi liang-liang di permukaan karbon PKS. Secara keseluruhan, karbon PKS mempunyai keupayaan untuk menyingkirkan minyak dah gris yang menunujukan bahawa PKS boleh digunakan sebagai penjerap semulajadi dalam penyingkiran minyak dan gris.

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LIST OF ABBREVIATION

$(NH_4)_2S_2O_8$	Ammoniun Persulphate
BET	Brunauer-Emmett-Teller
BOD	Biological Oxygen Demand
C	Carbon
Cf	Final Concentration
Ci	Initial Concentration
DO	Dissolved Oxygen
EAC	Extruded Activated Carbon
GAC	Granular Activated Carbon
H_2O_2	Hydrogen Peroxide
H_3PO_4	Phosphoric Acid
HCl	Hydrochloric Acid
HNO_3	Nitric Acid
$KMnO_4$	Potassium Permanganate
KOH	Potassium Hydroxide
mg/L	Milligram per Liter
mL/min	Flow rate
mm	Millimeter
PAC	Powdered Activated Carbon
PKS	Palm Kernel Shell
RSM	Response Surface Methodology
SEM	Scanning Electron Microscopy
SME	Small and Medium Enterprise

$ZnCl_2$	Zinc Chloride

LIST OF SYMBOL

%	Percentage
˚C	Temperature in Degree Celsius

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Oil and grease contaminate the wastewater by the sources of crude oil productions, oil refinery, petrochemical industry, metal processing, compressor condensates, lubricants, cooling agents, car washing and also restaurants operations (Lan, 2009). Oil and grease wastewater usually consists of toxic substances that bring bad effect towards plants and animals.

The substances such are phenols, petroleum hydrocarbons and polyaromatic hydrocarbons inhibit the growth of plants and animals. Waste generated containing oil and grease is generally classified as hazardous pollutants especially when the waste is being flow into aquatic environment. The oil and grease will form a layer on the water surface it contaminated.

The layer decreases the biological activity of the natural treatment process. The surface of oil and grease will surround the microbes in the water and leads to the decrease in the level of dissolved oxygen level in the water (El-Gawad, 2014). The oxygen molecules will be difficult to be oxidative for the microbial on the hydrocarbon molecules and cause ecological damages (Alade et. al, 2011).

This will cause high toxicity towards the water bodies and affects the aquatic organism and other ecological damage (Mendiolas et. al, 1998). Skimming tanks and oil and grease traps in treatment plants are other conventional techniques in removal of oil and grease. These treatments do not seem to be working as they have low efficiency in removing the oil and grease.

There are also pre-treatment that been done before the wastewater enter the treatment plant such as grease-trap, tilted plate separator and dissolved air flotation system (Cammarota & Freire, 2006). However, there is still oil and grease remains and causes the treatments plants pipes to be clog. This then increase the cost in maintenance and inspection of the pipes (Mueller et. al, 2003).

Activated carbon seems to be the alternative of the high cost conventional techniques in removing oil and grease in wastewater. Removing the oil and grease in the wastewater using activated carbon absorption is a physic-chemical type of treatment (Namasivayam & Kavitha, 2002). Activated carbon has the properties of thermo-stability, high performance, high adsorptive effect, large surface area and well-developed structure (Yahya, 2016). There are many activated carbon produce from variety of other materials such as, orange Peel, banana pith and wood to remove many contaminate exist in wastewater.

For this study, palm kernel shell is chosen as the materials that the activated carbon will be produce from. Palm kernel shell is produce from the palm oil industry and is consider as an agriculture waste. The palm oil industry in Malaysia generates large amount of the palm kernel shell (Onundi et. al, 2010) and this enable the usage of the activated carbon from palm kernel shell as it's easily to be obtain. Apart from that, palm kernel shell has a high percentage of carbon, C. The high amount of carbon content generally will produce high percentage of char and this increase the product yield. In the production of activated carbon it seems like Palm kernel shell will act as a good precursor (Andasa et. al, 2017).

1.2 Problem Statement

The discharge of wastewater containing oil and grease from premise and factory into the wastewater system will later cause few problems if the oil and grease is not treated properly. The discharge will affect the environment if not treated well in the wastewater system but it will also affect the piping system of the wastewater itself. The oil and grease give problem to the piping system of the wastewater system and the piping of the wastewater is needed to be check and fix frequently.

Pipes in the wastewater sewage system will be blocked constantly causing the increase in maintenance cost. Accumulation of the lighter oil in the system of the sewage pumping station will lead to the fouling of electrodes or float system in wastewater system. This will give problem towards the pump controls and may cause failures of function. The oil itself is flammable which can cause hazard towards the treatment process and the treatment plant itself.

The present of oil and grease in the water bodies leads to the formation of layer of oil that caused problems towards the environment. The marine and aquatic organism will face problems as their habitat has been contaminated. When the marine life is affected, degradation of the fishes and other aquatic life will cause effect towards human. Human that consume the aquatic organism such as fishes and other sea foods may obtain health problem. Economic growth in the fisheries sector will be effected by the degradation of the aquatic life causing the economic profit to decreases.

1.3 Expected Outcomes

In this study, the result expected is that the palm kernel shell activated carbon will be able to reduce the concentration of the oil and grease in the wastewater system. With the outcomes of the study, a further study on the usage of palm kernel shell activated carbon as an absorbent in removing of oil and grease can be carried out. This method in removing of oil and grease could be implemented in the sewage plants is Malaysia if the study is carry on further.

1.4 Objectives

1. To determine the ability of palm kernel shell activated carbon to adsorb oil and grease.
2. To determine the optimum parameters affecting column adsorption by different concentration of oil, time and flow rate.

1.5 Scope of Study

In this study, the scope of this study is to remove the oil and grease which is present in wastewater from either industry or municipal discharge. The method use to remove the oil and grease in the wastewater is by the physical-chemical method which using the activated carbon as the adsorbent.

The activated carbon is derived from the palm kernel shell which one of agriculture waste from oil plantation or oil mill. The wastewater was taken from a small and medium enterprise (SME) which conducts food process for its business. The oil and grease will undergo gravimetric process to obtain the percentage of oil and grease before and after the experiment.

The optimum parameters affecting column adsorption by different flow rate, concentration of oil and contact time will be conducted. The Palm Kernel Shell, PKS activated carbon samples will be characterized by using the Scanning Electron Microscopy, SEM.

1.6 Significant of Study

This study has a significant for the community to have a better quality of treated water by making sure that the oil and grease in the wastewater are removing mostly. As the oil and grease cause problem to the treatment sewage, the wastewater system will able to function smoothly without having the worry of the blockage of oil and grease is the pipe system. The authority will not worry about the malfunction of the sewage plant and the cost of maintenance of the pumps and pipe will not be increase.

As nowadays, the environment aspect has become more important to conserve and save them. With this study, one of the sources of oil pollution can be decrease. The removal of oil and grease in the wastewater using activated carbon from palm kernel shell helps the palm oil plantation to use their waste into something that is useful to the community and environment. Palm kernel shell is one of the waste produce from an oil palm plantation activity.

The palm kernel shell is usually burned or dump on the plantation site that causes other environmental problem. As Malaysia is well known on having a large sector of oil palm plantation, the waste from the production and activity of this sector is in large amount. The large amount of waste will contribute more towards environment problems.

With this study, the potential of palm kernel shell waste from oil palm plantation helps reduce the effect toward the environment. It shows that this study will help a lot in terms of increasing environmental health of the area.

CHAPTER 2

LITERATURE REVIEW

2.1 Oil and Grease in Wastewater

2.1.1 Source of Oil and Grease

The largest source of wastewater containing oil and grease is from the production of oil or oil extraction process. This process usually occurs in oil mills and the effluent from the oil mills contains high concentration of oil and grease (Ahmad et. al, 2005). Oily wastewater does also come from domestic sector such as housing and firms. It is reported that higher contributor of oil and grease in domestic grey water is from kitchen grey water, although overall grey water stream do have oil and grease detected in them (Travis et. al, 2008),(Friedler et. al, 2004).

Food processing industries such as meat packing industries, slaughterhouses and dairy production produces effluents containing oil and grease from their industry production (Vidal et. al, 2000), (El-Bestawy et. al, 2005). The production of machine, petroleum refining, steel production, metal cutting and metal forming and textile industries and much other manufacturing industry also produces effluent containing non-vegetable oil (Wake, 2005). Coolants and lubricants applied for cooling the work pieces and machines tools, to reduce friction and wear of tool and improving surface quality of work space in metalworking industries generates effluent containing oil and grease (Alade et. al, 2011).

2.1.2 Effect Oil and Grease in Wastewater

The oily wastewater contains toxic substances such as phenols, petroleum hydrocarbons, polyaromatic hydrocarbons. The substances give harmful effect towards the plants and animal's growth in the water bodies is contaminated. Oil and grease went present in water bodies tend to forms oil layer which causes pollution problems to the marine environment such as reduce in light penetration and photosynthesis. This if prolong leads to the decrease in amount of dissolved oxygen (DO) in the water bodies. This leads to the effect on the survival of aquatic life in the water (Mohammadi, & Esmaelifa, 2005).

2.1.3 Malaysia Effluent Discharge Standard

Malaysia industry has the standard in discharging the wastewater or effluent to the environment. The limitation and standard of these components in the wastewater effluent is listed under the Environmental Quality Act, EQA 1974. Under the EQA the standard discharge falls under the Environmental Quality (Industrial Effluent) Regulations 2009. Oil and grease in wastewater from industry and housing area falls under the Fifth Schedule under Environmental Quality (Industrial Effluent) Regulations 2009 (Environmental Quality (Industrial Effluent) Regulations 2009).

Table 2.1: Acceptable conditions for discharge of industrial effluent or mixed effluent of standard A and B

	Parameter	**Unit**	**Standard A**	**Standard B**
(1)		(2)	(3)	(4)
(i)	Temperature	^oC	40	40
(ii)	pH Value	-	6.0-9.0	5.5-9.0
(iii)	BOD at 20^oC	mg/L	20	50
(iv)	Suspended Solids	mg/L	50	100
(v)	Mercury	mg/L	0.005	0.05
(vi)	Cadmium	mg/L	0.01	0.02
(vii)	Chromium, Hexavalent	mg/L	0.05	0.05
(viii)	Chromium, Trivalent	mg/L	0.20	1.0
(ix)	Arsenic	mg/L	0.05	0.10
(x)	Cyanide	mg/L	0.05	0.10
(xi)	Lead	mg/L	0.10	0.5
(xii)	Copper	mg/L	0.20	1.0
(xiii)	Manganese	mg/L	0.20	1.0
(xiv)	Nickel	mg/L	0.20	1.0
(xv)	Tin	mg/L	0.20	1.0
(xvi)	Zinc	mg/L	2.0	2.0
(xvii)	Boron	mg/L	1.0	4.0
(xviii)	Iron (Fe)	mg/L	1.0	5.0
(xix)	Silver	mg/L	0.1	1.0
(xx)	Aluminium	mg/L	10	15
(xxi)	Selenium	mg/L	0.02	0.5
(xxii)	Barium	mg/L	1.0	2.0
(xxiii)	Fluoride	mg/L	2.0	5.0
(xxiv)	Formaldehyde	mg/L	1.0	2.0
(xxv)	Phenol	mg/L	0.001	1.0
(xxvi)	Free Chlorine	mg/L	1.0	2.0
(xxvii)	Sulphide	mg/L	0.50	0.50
(xxviii)	Oil and Grease	mg/L	1.0	10
(xxix)	Ammoniacal Nitrogen	mg/L	10	20
(xxx)	Colour	ADMI*	100	200

*ADMI-American Dye Manufacturers Institute

Source: Environmental Quality (Industrial Effluent) Regulations 2009

Based on table 2.1 it shows, that the acceptable amount or concentration of oil and grease in wastewater effluent for standard A is 1.0 mg/L and 10 mg/L for standard B.

2.2 Palm Kernel Shells

Palm kernel shells is a by product of an oil mill or an oil plantation site. Palm kernel shell is produce when the palm oil is extracted to obtain the palm oil. The palm kernel shell along with other by product is burned as fuel in the boiler of palm oil mill. The utilization of the palm kernel shell is relatively low, thus causing it to accumulate and increase in number (Olutoge et al, 2012). Palm kernel shell has a potential in becoming a quality charcoal with its characteristic of adequate hardness, low ash content and fairly high fixed carbon, (Elham, 2001).

2.3 Activated Carbon

2.3.1 Characteristic

Activated carbon, is a carbonaceous material that has large internal surface area and has a highly develop porous structure which cause by the high temperature reaction. It is a non-polar adsorbent which is used widely due to its ability in absorb small compounds (Soderberg, 2008). Its characteristic is having large surface area, well developed porosity and tunable surface-containing functional groups (Baker et al. 1992) (Zongxuan et al., 2003). It is compost of about 87% to 97% of carbon but the carbon percentage depends on the raw materials it is derived from (Leimkuehler, 2010).

The porous structure in the activated carbon able it to adsorb materials in the phase of liquid and gas (Jankowska, et. al, 1991). The ability of the activated carbon in adsorbing a compound in a solution depends on parameter such as polarity of the contaminants or compound, contact time between activated carbon and the contaminant, the pH of the solution, temperature and the concentration of the compound (Edalat, 2008). There are different activation methods such as dry and wet methods for easing the introduction of the surface oxygen from functional groups

2.3.2 Activation Methods

Dry and wet oxidizing agents are used as different methods in activation of the activated carbon. Dry oxidation methods involve the reaction of hot oxidizing gas such as steam and carbon dioxide at the temperature of 700℃ (Smisek and Cerney 1970). Wet oxidation methods involve the reaction between carbon surface and the oxidizing agents such as potassium hydroxide KOH, nitric acid HNO_3, potassium permanganate $KMnO_4$, hydrogen peroxide H_2O_2, zinc chloride $ZnCl_2$, ammonium persulphate $(NH_4)_2S_2O_8$ and phosphoric acid H_3PO_4.

2.3.3 Production Process

Activated carbon can be produce from or derived from a wide range of precursor materials including wood, agriculture waste, coal and many more (Sivakumar et. al, 2012). Productions of activated carbon involve carbonization process then followed by the activation of the carbonaceous materials. Carbonization process is a heat treatment at the temperature of 400˚C to 800˚C, which this convert the raw materials into carbon.

This process increases the carbon content in the material and minimized the content of volatile matter. The strength of the material increases through this process and it also creates the porous structure. The porous structure is needed for the carbon to be activated (Leimkuehler, 2010). It is important to choose the correct temperature depends the material used or derived from (Jankowska, et. al, 1991).

This due to the increase of temperature in the process can increase the reactivity but the volume of pores will decrease.

The material is carbonized at a high temperature that increases the condensation and decreases the volume of pores which cause an increase in mechanical strength (Leimkuehler, 2010). After the carbonization which has created the porous structure, an oxidation or activation is carried out in order to create micro pores (Beguin & Frackowiak, 2010). Oxidizing gases or chemical activation is the different type of methods of activation that can be carried out.

For activation by oxidizing gases, the carbon reacts with the oxidizing agent which will produce oxides of carbon. These oxides then diffuse out from the carbon which results to partial gasification. The carbon internal porous structure will further develop and the pores will opens through this process. For chemical activation, high temperature is reacted with the carbon along with a dehydrating agent which removes the majority of oxygen and hydrogen from the carbon structure. Chemical activation process often combines the carbonization and activation process, but these two processes still can be done separately depending on the process.

2.3.4 Types of Activated Carbon

The activated carbon can be classified into three types which are categorized by it size and shape of its particle and each type has its own specific application. Powdered Activated Carbon (PAC) has a large surface area and has ability in adsorbing compounds. PAC has been used in many studies in removing contaminates and organic matter in wastewater by adsorption process (Cecen, Erdincler and Kilic, 2003). PAC is not compatible in removing heavy metals through adsorption but able to absorb substances that have a higher molecular weight and low water solubility.

Granular Activated Carbon (GAC) expected in more efficient in reducing organic fouling which provides the contact time sufficient allowing the substantial adsorption to take place (Wend et al, 2005). GAC is defined as it can be retained on a 50-mesh sieve (0.297 mm) which has relatively larger particle the PAC. GAC is used in water treatment, deodorization and separation of the component or contaminant of flow system.

Next type of activated carbon is Extruded Activated Carbon, EAC. EAC is the combination of powdered activated carbon (PAC) with a binder that helps fused together and extrudes it into a activated carbon block that is cylindrical in shaped. This EAC usually used for application of gas phase contamination removal as they have low dust content, high mechanical strength and low pressure drop (Pradhan, 2011).

2.3.5 Application of Activated Carbon

Activated carbon has wide range of applications which concerned to the removal of contaminates or elements by the absorptions from liquid or gas form (Sivakumar et. al, 2012). Activated carbon is being used as adsorbents widely in removing of metal ions and organic chemicals of environment and economic concern from air, gases and wastewater sectors (El-Hendawy, 2003).

The application of activated carbon has been widely use since ancient times. Egyptians used charcoal as absorbent for medicines and as purifying agents. In 1900's activated carbon is derived from plants and used for decolourization.

Activated carbon is used during World War I, for gas mask to protect from hazardous gas and vapour.

Nowadays, is used to remove contaminates and colours from pharmaceutical and food product processes. It also act as air pollution control devices for automobile exhaust and other industrial and involve in chemical purification and also electrodes in batteries (Leimkuehler, 2010).

2.4 Previous Study in Using Activated Carbon in Removing Oil and Grease in Wastewater

There are few previous study of usage of activated carbon in removal of oil and grease that has been carried out. Table 2.2 show the previous study that has been carried out.

Table 2.2: Previous studies of removal of Oil and Grease using Activated Carbon

Title	Description	Reference
Removal of Oil and Grease from Wastewater using Natural Adsorbents	It uses Sugarcane Bagasse and Banana Pith as its raw material of the activated carbon. the study show that both activated carbon of Sugarcane Bagasse and Banana Pith have the capability of removing oil in the wastewater	(Abdul. et al, 2015)
Removal of Oil and Grease from Wastewater	The activated carbon was extracted from Curry leaf,	(Balaji, Amarnath and

using Natural Adsorbent	Banana pith and Neem leaf. Neem leaf shows the higher effective removal of oil and grease in wastewater.	Balasubramaniyan. A. L., 2018)
The Effectiveness of Activated Carbon from Coconut Shell as Wastewater Pollutant Removal	Activated carbon was derived from Coconut shells to remove pollutants in wastewater where oil and grease is one of the pollutant listed under the Environmental Quality Act, 1974.	(Azhari, 2010)

CHAPTER 3

MATERIALS AND METHOD

3.1 Materials and Reagent

The oil and grease wastewater sample was collected from a local small and medium enterprise (SME) that process fish cracker which is located in Tumpat, Kelantan. N-hexane was used in the experiment as solvent. Palm Kernel Shell (PKS) was collected from Felda Kemahang palm oil plantation mill.

3.1.1 Oil and Grease Wastewater Characterization

Oil and grease wastewater was collected with sampling bottle to conduct the analysis ex-situ. The collected waste water was undergone the gravimetric method analysis to determined the concentration of oil and grease in the sample collected. The wastewater sample was spiked with used cooking oil (1%, 5% and 10%) because the concentration of oil and grease in the wastewater sample was below than the standard discharged which listed under the Environmental Quality Act, 1974.

3.1.2 Wastewater In-situ Analysis

The parameter of temperature, total dissolved solid, pH, salinity and dissolved oxygen (DO) are analysed in-situ. The in-situ parameters were analysed using the YSI Multiparameter.

3.2 Methods

3.2.1 Preparation of Palm Kernel Shell Activated Carbon

The Palm Kernel Shells (PKS) were collected from the local oil palm plantation mill. The PKS was washed, dried under the sun until fully dry and was crushed into smaller pieces. The PKS was crushed by using a blender and was sieved using a 2.36 mm sieve. The sieved PKS was place into crucible and was closed with its lid. The closed crucible was wrapped with aluminium foils.

The wrapped crucible was placed into muffle furnace and was carbonized at 400℃ for 1 hour. 3M of KOH solution was used to soak the carbonized PKS with the ration of 1:1. The KOH was used as an activating agent in the preparation of the PKS activated carbon. The mixture of carbonized PKS and KOH solution was placed on a hot plate at the temperature of 80℃ and was continuously stirred for 2 hours.

After 2 hours, the PKS and KOH solution mixture was filtered using a vacuum pump and was dried overnight in oven at 120˚C. The dried sample was placed in the muffle furnace for 15 minutes to be carbonized again at 800˚C. The dried sample was cooled at room temperature and was washed until the pH7 was obtained using hot distilled water. After obtaining pH7, the sample was dried in the oven at 120˚C. The sample or prepared activated carbon was cooled and stored in the desiccators for further analysis.

3.2.2 Column Adsorption Set up

The column adsorption set up consists of retort stand to hold the column in place and a peristaltic pump to control the flow rate of the wastewater. A tube was connected to the source of the wastewater to the column through the peristaltic pump. The height of the PKS activated carbon was kept at constant height of 10 cm throughout the experiment. A set up without the PKS activated carbon present in the column was also done as a control. Figure 3.1 shows the set up of the column adsorption method.

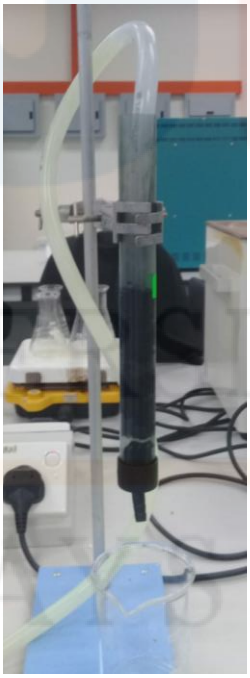

Figure 3.1: Adsorption analysis set up

3.3 Adsorption Analysis

3.3.1 Effect of Different Concentration of Oil and Grease in Different Time

The wastewater was spiked with used cooking oil into 1%, 5% and 10% oil concentration. The spiked wastewater (1% oil concentration) was pump into the column that was filled with PKS activated carbon. The height of the PKS activated carbon was measured at 10 cm and was kept constant throughout the adsorption process. The wastewater flow rate was kept constant at 1 mL/min and the experiment was run 24 hour time. The wastewater that passed through the column was collected at every 2 hours until 24 hours and followed by analysed using gravimetric method. The similar experiment was continued with the different oil and grease concentrations (5% and 10%).

3.3.2 Effect of Different Flow Rate

The oil and grease wastewater with the optimum concentration of oil and grease which was obtained by the experiment conducted from Section 3.3.1 was pump into the column that was filled with PKS activated carbon. The flow rate of the oil and grease wastewater was set into 1 mL/min, 3 mL/min and 5 mL/min. The wastewater that flowed through the column was collected and was analysed using gravimetric method.

3.4 Gravimetric Method

Gravimetric analysis is a set of methods in analytical chemistry for the quantitative determination of analytic based on the mass of a solid. The wastewater that has passed through the PKS activated carbon in the column was extracted with n-Hexane. Hydrochloric acid solution (HCl) was added with the ratio of 1:1 to decrease the pH to 2 or less and act as sample preservation.

The round bottom flask was cleaned with detergent and hot water. The round bottom flask then was rinsed with distilled water and n-Hexane. It is then was dried for 2 hours in the oven at 105˚C. The round bottom flask was cooled to room temperature in 30 minutes in the desiccators. The mass of the round bottom flask was weight immediately using analytic electronic balance.

The extraction process was done three times using a separating funnel and n-hexane was used as a solvent. The extracted solution in the round bottom flask was extracted again to rinse the extraction solution. The final extracted solution was undergone distillation process in order to remove the n-Hexane from the solution. The solution was oven dried at 105˚C overnight to remove water from the round bottom flask.

The residue in the round bottom flask was weighed using analytic electronic balance. To obtain constant weight the round bottom flask was weighed a few times with heating it for 10 minutes in the oven and was left for 30 minutes to cool. The percentage (%) removal of oil and grease in the wastewater will be calculated by the following equation:

$$\text{Percentage removal of oil, } \% = \frac{(\mathrm{Ci}-\mathrm{Cf})}{\mathrm{Ci}} \times 100 \qquad (3.1)$$

Where Ci was represented as the initial reading of the sample, Cf was the final reading of the sample.

3.5 Characterization of Activated Carbon using Scanning Electron Microscopy, SEM

The sample that was conducted under the SEM is the raw blended and sieved PKS, the unused PKS activated carbon and the used PKS activated carbon. Before the samples were characterized, the samples were dried to remove any moisture on the surface of the samples. After the samples were characterized with the SEM images of the surface of the different sample was produced in image form.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Wastewater In-Situ Analysis

The readings of the in –situ analysis of the wastewater is done by using YSI Multiparameter to obtained the parameter stated in Table 4.1.

Table 4.1: The in-situ analysis of the wastewater

Parameter	Readings
Temperature, ˚C	76.43
Total Dissolved Solid, %	1.597
Dissolved Oxygen, %	3.2
pH	5.6
Salinity,	1.29

In comparison to the elements under the discharged standard by the Environmental Quality Act, 1974 the in-situ analysis show that most of the element passed the discharge standard. The total dissolved solid are relatively low at 1.597% which passed the standard A discharge which must be less than 50%. pH passed the standard B of discharged standard with the readings of 5.6.

Oil and grease concentration passed the Standard A of the discharge standard as at the early wastewater analysis show no oil and grease present by gravimetric method. Salinity and dissolved oxygen is not the element listed under the discharged standard (EQA, 1974)

4.2 Adsorption Analysis

4.2.1 Effect of Different Concentration of Oil and Grease with Different Time

Figure 4.1 show the percentage removal of different concentration of oil and grease (1%, 5% and 10%) in 24 hours time. The reading in the graph shows the percentage removal of oil and grease for every 2 hours time.

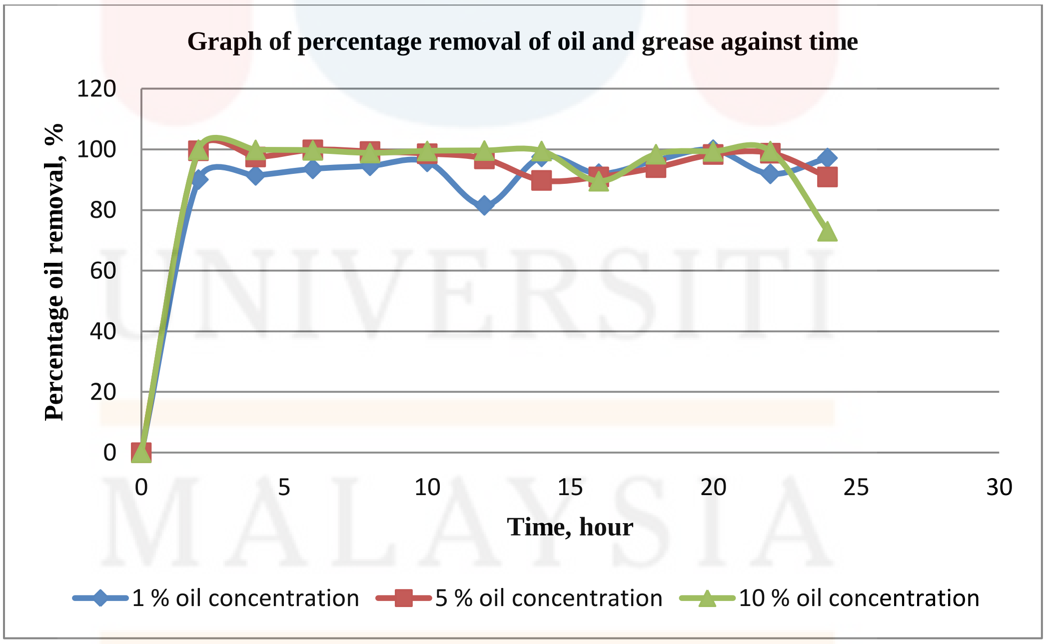

Figure 4.1: Graph of percentage removal of oil and grease, % against time, hour for flow rate 1 mL/min.

For the analysis on the different percentage of oil and grease concentration (1%, 5% and 10%) the flow rate of the column adsorption was kept constant at 1 mL/min and the readings was taken from 0 to 24 hour time of contact. At the early contact time for all 1%, 5% and 10% oil and grease concentration shows high percentage oil and grease removal. This due to at the early time of the wastewater and the PKS activated carbon in the column the most of the oil and grease in the wastewater was stuck on top of the PKS activated carbon. This leads to the decrease in oil and grease concentration in the wastewater collected.

For 1% oil and grease concentration, it show a gradual increase in the percentage removal of oil and grease but then at the time of 10 hours the percentage removal shows a decrease and after the 10 hour time, the percentage removal of oil and grease is not at consistence throughout the 24 hours. The highest percentage removal for the 1% oil and grease concentration was at the time of 20 hour at the 99.93% where the last oil and grease concentration remains in the wastewater was 4 mg/L.

This amount is allowed in discharging the wastewater into the river which falls under Standard B where at a maximum of 10 mg/L is allowed (EQA, 1974). The lowest percentage removal of 1% oil and grease concentration is 81.57% at the 12 hour time. The 20 hour time can be said as the optimum time in removing oil and grease for the 1% oil and grease concentration as it shows the highest percentage removal.

For 5% oil and grease concentration, it shows a constant percentage removal of the oil and grease. After 14 hours, the percentage removal of oil and grease for the 5% oil and grease concentration continues to slightly increase in percentage removal until the time of 22 hour. 5% oil and grease concentration shows more constant readings of percentage removal of oil and grease throughout the 24 hours period.

The highest percentage removal of oil and grease is 99.89% at the time of 6 hour. After that, the percentage removal slightly decreases until at the 14 hour which is the lowest percentage removal for 5% oil and grease concentration. The percentage removal then continues to increase slightly and at the 24 hour time it decreases slightly. The optimum contact time for percentage removal of oil and grease for 5% oil and grease concentration was at 6 hour time as it shows highest percentage removal.

The 10% oil and grease concentration show the constant percentage of oil and grease removal at until at 14. The percentage removal of oil and grease of the 10% oil concentration decreases at the end of the time period. The highest percentage removal of oil and grease is at the time of 2 hours. But to compare among all the different oil and grease concentration, most of the percentage removal at the contact time of 2 hours is high.

This is due to the oil and grease in the wastewater that stick on top of the PKS activated carbon in the column. Throughout the time period 10% oil and grease concentration shows a constant percentage of removal and most percentage is more than 90% or removal. The lowest percentage removal of oil and grease for the 10% oil and grease concentration is at the time of 24 hour with 73.04%. The decrease of percentage removal would indicate that the activated carbon has become saturated with oil and grease as it shows a drastic decrease.

A control set up was also conducted to obtain the exact amount of oil and grease remove by the PKS activated carbon. As oil and grease tends to stick on surfaces, the control experiment is implemented to ensure and to know the real percentage removal of oil and grease is from adsorption of the PKS activated carbon and not from the oil and grease sticking onto the surface of the column. This control experiment was set up by having the same column adsorption set up but without the present of PKS activated carbon in the column.

The control readings obtained is not at a consistent state (Appendix C). At a certain contact time the control percentage removal is higher that the percentage removal where the PKS is present in the column. With these readings, it indicates that there is no removal at all from the PKS activated carbon present as the control reading is higher. But at certain contact time, the control percentage removal readings are much lower than the percentage removal where the PKS is present in the column.

With these, it shows that the PKS activated carbon do remove the oil and grease in the wastewater. These might happen due to the tube that connects and flow the wastewater into the column through the peristaltic pump. The ups and down of the tube accumulated the oil and grease at the top curve of the tube and then when it reaches the column, the oil and grease that goes through the column at one time is not the same. The amount of oil and grease that pass through the column at different time has different amount of oil, grease and wastewater.

Overall, the PKS activated carbon do remove the oil and grease in the wastewater but it is unable to obtained the exact percentage of the removal of oil and grease by the PKS activated carbon. This is due to the inconsistent reading from the control experiment set up.

4.2.2 Effect of Different Flow Rate

Figure 4.2 show the reading of percentage removal of the same concentration of oil and grease (5%) with different flow rate. The different flow rate (1 mL/min, 3 mL/min and 5 mL/min) was adjusted by the peristaltic pump. The experiment readings were collected in 6 hours time period.

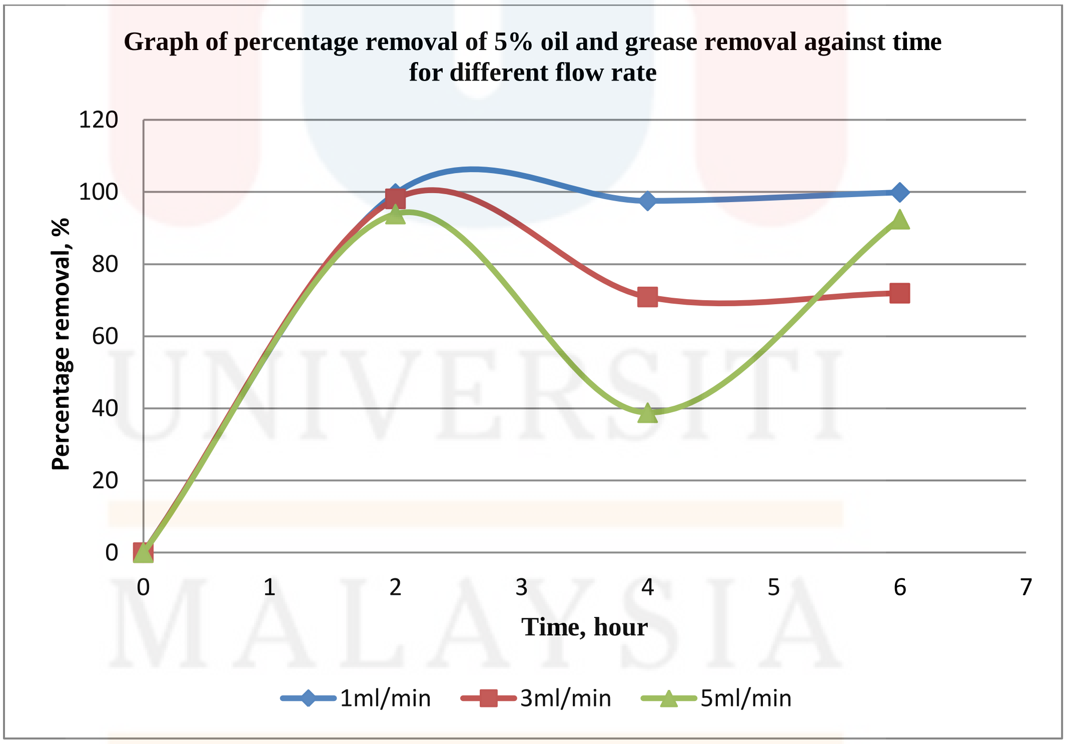

Figure 4.2: Graph of percentage removal of 5% oil and grease concentration with different flow rate.

The flow rate parameter was change in this experiment to see on the effect of flow rate on the column absorption study. The concentration of the oil and grease in the wastewater was kept constant at 5 % concentration. The reading is taken up to 6 hour contact time because at the 5 % oil and grease concentration it shows the highest removal at the 6 hour contact time.

From Figure 4.2, it shows that column flow rate of 1 mL/min show the highest removal among other flow rate. The highest percentage removal is at 6 hour with 99.89% with 68 mg/L oil and grease left. Although 68 mg/l is the lowest concentration of oil and grease obtained but this does not pass the allowed amount of oil and grease concentration according to EPA Standard B which is at maximum of 10 mg/L (EQA, 1974).

To compare all three flow rates, 1 mL/min show the higher percentage removal than 3 mL/min and 5 mL/min which can be seen clearly from Figure 4.2. To see from the overall view of the different flow rate, the slower flow rate in this study is 1 mL/min is more constant and at a higher percentage in removing the oil and grease in the wastewater than the other flow rate. Oil and grease removal is more efficient at slower flow rate to compare with the higher flow rate (Hebbar & Jayantha, 2014).

The control experiment is also conducted to obtain the exact percentage removal of oil and grease which due to the PKS activated carbon and not from loss sticking onto the tube or column surface. Due to the inconsistent reading of the control percentage removal of oil and grease the exact percentage removal from the PKS activated carbon for the different flow rate are unable to be define.

4.3 Characterization of PKS Activated Carbon using Scanning Electron Microscopy, SEM

The PKS activated carbon is characterised by using Scanning Electron Microscopy, SEM. SEM is a tools in providing details surface information by which it trace the sample in a raster pattern using an electron beam (Choudhary and Priyanka, 2017). The SEM is used to study the characterization of the PKS activated carbon. The raw PKS, activated PKS and used PKS are tested under the SEM to see the surface structure. The raw PKS is the sample where no additional chemical are added. The sample is only blended and sieved according to the wanted size. The Figure 4.3 shows that the surface of the raw PKS sample has no visible pores on its surface. The raw PKS show smooth with some rugged surface presence (Amran et al., 2016)

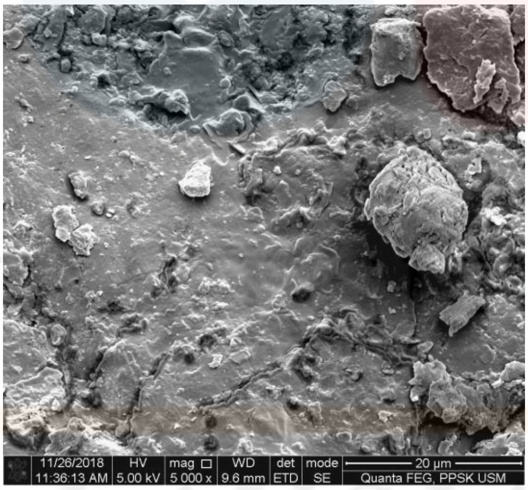

Figure 4.3: The SEM image of raw PKS at the magnification of 5000×

Figure 4.4 show the PKS activated carbon that has been carbonized at high temperature and activated using KOH as the activation agent. The SEM image show the surface of the activated PKS has numbres of pores. The pores are irregular in shaped and some pores are unusually large (Rugayah, Astimar & Norzita,2014).

The surface also show burn out pores with tunnel or honey-comblike structure (Abechi. et al., 2013).

These pores is likely to be appeared by the carbonization and activation process that the raw PKS undergoes. The carbonization at high temperature and activation using KOH leads to the pores structure to be present at the surface of the PKS activated carbon. The well-developed porous structrure with the regular pores size shows the activation has take place (Duran-Valle et al., 2005).

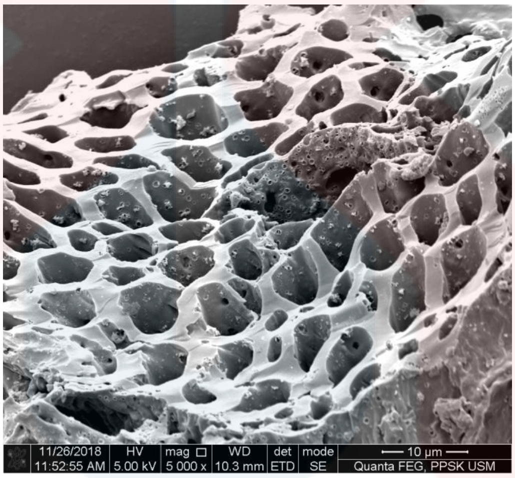

Figure 4.4: The SEM image of activated PKS at the magnification of 5000×

Figure 4.5 show the PKS activated carbon that has been used for the adsorption analysis. The figures show the pore which seems to be the oil and grease has covered the pores on the surface of the PKS activated carbon. It shows the adsorption of the oil and grease onto the surface of the PKS activated carbon surfaces.

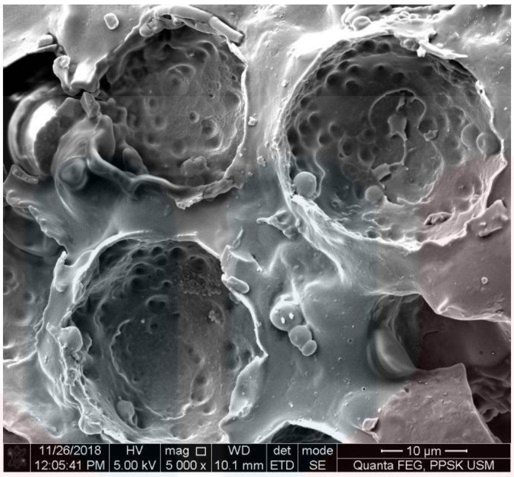

Figure 4.5: The SEM image of activated PKS at the magnification of 5000×

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

This study shows that the present of PKS activated carbon in the column to have an effect in removing the oil and grease in the wastewater. The higher the oil and grease concentration in the wastewater show a shorter period of time for the PKS activated carbon to be saturated. This can be seen at the end of the 24 hour time period the percentage removal of oil and grease for 10% oil and grease concentration is at 73.04% which is the lowest to compare to 1% and 5% oil and grease concentration where at much higher percentage removal. With a further study on contact time the exact saturated period of the 1% and 5% oil and grease concentration can be obtained.

For different flow rate study, it show the slower flow rate, 1 mL/min has a higher percentage removal than the faster flow rate 3 mL/min and 5 mL/min. This is might due to the fact that the slower flow rate gives the activated carbon more time to adsorb the oil and grease that the faster flow rate. In conclusion, the PKS activated carbon does remove the oil and grease that present in the wastewater but the exact percentage of the removal of the oil and grease which is solely caused by the PKS activated carbon is unable to be identified due the inconsistent percentage removal of oil and grease in the control set up.

5.2 Recommendations

To obtain the exact amount of percentage removal of oil and grease from the PKS activated carbon a further study is needed. The column adsorption study is important in wastewater pollutant removal study as the wastewater treatment system in Malaysia uses pipe system which is similar to the column adsorption study. But as seen from this study, the exact percentage removal of oil and grease from the PKS activated carbon is unable to be determined due to the inconsistent reading of the control experiment set. A further study is needed to obtain an exact removal percentage from the activated carbon of other media in the column adsorption methods for oil and grease removal from wastewater.

The parameters used in the removal of oil and grease in the wastewater can be optimized through applying Response Surface Methodology (RSM). RSM consist of group of mathematical and statistical techniques which based on the fit of empirical models the experimental data obtained in relation to experimental design (Bezerra. et al, 2008). Linear or square polynomial functions will be employed to describe the system and to explore more on the experimental conditions until it is optimized (Teofilo, 2006). Through RSM, the parameter of the study that should be used can be calculated mathematically based on the fit of a polynomial equation of the experiment data. More parameter could be added to obtain the best result in removal of oil and grease in the wastewater using the PKS activated carbon.

For further study of the characterization Palm Kernel Shells (PKS) activated carbon there are few techniques or technologies can be used. Further physical characterization can be determined with the use of Brunauer-Emmett-Teller (BET). BET can helps in observing the surface area of the activated carbon by a physical absorption of gas molecules. Through this technique, characteristic such porosity can be studied and observed. The BET technique helps in increasing the understanding the physical characteristic of the activated carbon. With a higher understanding of the activated carbon's physical characteristic, its parameter such as amount of activated carbon needed, or contact time can be adjusted to find the best parameter in removing oil and grease in the wastewater.

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APPENDIX A

TABLES OF DIFFERENT OIL AND GREASE CONCENTRATION FOR 1 ML/MIN OF FLOW RATE.

Table A1: 1% oil and grease concentration, 1mL/min flow rate

Time, hour	Mg/L or PPM	Percentage removal, %
0	5544	0
2	550	90.08
4	474	91.45
6	354	93.61
8	298	94.62
10	226	95.92
12	1022	81.57
14	136	97.55
16	442	92.03
18	198	96.43
20	4	99.93
22	446	91.96
24	156	97.19

Table A2: 5% oil and grease concentration, 1mL/min low rate

Time, hour	Mg/L or PPM	Percentage removal, %
0	63788	0
2	270	99.58
4	1578	97.53
6	68	99.89
8	430	99.33

10	888	98.61
12	1978	96.90
14	6508	89.80
16	5776	90.95
18	3824	94.01
20	1046	98.36
22	786	98.78
24	5780	90.94

Table A3: 10% oil and grease concentration, 1 mL/min flow rate

Time, hour	Mg/L or PPM	Percentage removal, %
0	430228	0
2	284	99.93
4	550	99.87
6	1252	99.71
8	4944	98.85
10	2048	99.52
12	1498	99.65
14	2144	99.50
16	45062	89.53
18	6680	98.45
20	2656	99.38
22	2282	99.47
24	115990	73.04

APPENDIX B

TABLE OF 5% OIL AND GREASE CONCENTRATION FOR DIFFERENT FLOW RATE

Table B1: 5% oil and grease, 1 mL/min flow rate

Time,	**Mg/L or PPM**	**Percentage removal, %**
0	63788	0
2	270	99.58
4	1578	97.53
6	68	99.89

Table B2: 5% oil and grease, 3 mL/min flow rate

Time,	**Mg/L or PPM**	**Percentage removal, %**
0	63788	0
2	1210	98.10
4	18570	70.89
6	17902	71.94

Table B3: 5% oil and grease, 5 mL/min flow rate

Time,	**Mg/L or PPM**	**Percentage removal, %**
0	63788	0
2	3896	93.89
4	39040	38.80
6	4808	92.46

APPENDIX C

TABLE OF CONTROL EXPERIMENT OF DIFFERENT OIL AND GREASE CONCENTRATION, 1 ML/MIN FLOW RATE

Table C1: Control experiment of 1% oil and grease concentration, 1 mL/min flow rate

Time, hour	Mg/L or PPM	Percentage removal, %
0	5544	0
1	270	95.13
4	236	95.74
8	2754	50.32
12	452	91.85
16	536	90.33
20	962	82.65
24	440	92.06

Table C2: Control experiment of 5% oil and grease concentration, 1 mL/min flow rate

Time, hour	Mg/L or PPM	Percentage removal, %
0	63788	0
1	19970	68.69
4	2096	96.71
8	6814	89.32
12	1766	97.23
16	1500	97.65
20	336	99.47
24	7980	87.49

Table C3: Control experiment of 10% oil and grease concentration, 1 mL/min flow rate

Time, hour	Mg/L or PPM	Percentage removal, %
0	430228	0
1	13504	96.86
4	45615	89.40
8	12689	97.05
12	12117	97.18
16	1257	99.71
20	3871	99.10
24	56530	86.86

APPENDIX E

PHOTO GALLERY

Figure D1: The fish crackers factory.