REMOVAL OF OIL AND GREASE IN INDUSTRIAL 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 Industrial 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.

Signature :________________________

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

ABSTRACT

The wastewater contaminated with oil and grease which discharged from industries and factories into the wastewater system will lead to a serious problem to the environment and human health. In this study, palm kernel shells (PKS) were choose as a natural absorbent in removing oil and grease in the wastewater system as the PKS become one of the most agricultural waste in Malaysia. The ability of PKS activated carbon in reducing the oil and grease concentration were studied. The PKS were carbonized at 400℃ in furnace, activation with KOH, furnace again at 800℃ and adjusted the pH to neutral using the distilled water. The parameter that were studied in affecting the removal oil and grease in wastewater were the effect of different contact time with initial concentration of oil and grease and the effect of different flow rate. Column adsorption method were conducted in identify the affecting parameters followed by the gravimetric method in determine the remaining oil and grease. At the 20 hours period of contact time, the removal efficiency of oil and grease were 99.18%. The PKS activated carbon shows the highest removal of oil and grease at 98.81% at 1 mL/min of flow rate. The functional group and the chemical structure of the PKS activated carbon before and after the absorption method were analysed. Generally, PKS activated carbon do has the ability in removing the oil and grease in the industrial wastewater.

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Penyingkiran Minyak dan Gris dalam Air Kumbahan Industri mengunakan Tempurung Kelapa Sawit Karbon yang Diaktifkan

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ABSTRAK

Air kumbahan yang tercemar dengan minyak dan gris yang dibuang oleh industri dan kilang ke dalam sistem air kumbahan akan membawa kepada masalah yang serius kepada alam sekitar dan kesihatan manusia. Dalam kajian ini, tempurung kelapa sawit (PKS) telah dipilih sebagai penyerap semulajadi dalam mengeluarkan minyak dan gris dalam sistem air kumbahan kerana PKS merupakan salah satu sisa pertanian yang paling banyak di Malaysia. Keupayaan PKS karbon yang diaktifkan dalam mengurangkan kepekatan minyak dan gris dikaji. PKS telah dibakar pada suhu 400℃, pengaktifan dengan KOH, dibakar sekali lagi pada suhu 800℃ dan pH diselarakan ke neutral menggunakan air sulingan. Parameter yang dikaji dalam mempengaruhi penyingkiran minyak dan gris dalam air kumbahan adalah kesan terhadap masa yang berbeza dengan kepekatan awal minyak dan gris dan kesan terhadap kadar aliran yang berbeza. Kaedah penjerapan kolum telah dijalankan untuk mengenal pasti parameter yang mempengaruhi diikuti oleh kaedah gravimetrik dalam menentukan minyak dan gris yang tersisa. Di jam ke 20, kecekapan penyingkiran minyak dan gris adalah 99.18%. PKS karbon yang diaktifkan menunjukkan penyingkiran minyak dan gris tertinggi pada 98.81% pada kadar aliran 1 mL / min. Kelompok berfungsi dan struktur kimia PKS karbon yang diaktifkan sebelum dan selepas kaedah penyerapan dianalisis. Secara amnya, PKS karbon diaktifkan mempunyai keupayaan untuk mengeluarkan minyak dan gris dalam air kumbahan perindustrian.

UNIVERSITI
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KELANTAN

TABLE OF CONTENT

FYP FSB

FYP FSB

UNIVERSITI MALAYSIA KELANTAN

FYP FSB

UNIVERSITI MALAYSIA KELANTAN

LIST OF TABLES

FYP FSB

UNIVERSITI MALAYSIA KELANTAN

LIST OF FIGURE

FYP FSB

UNIVERSITI

MALAYSIA

KELANTAN

LIST OF ABBREVIATION

$(NH_4)_2S_2O_8$	Ammoniun Persulphate
AOPs	Advanced Oxidation Processes
BET	Brunauer-Emmett-Teller
BOD	Biological Oxygen Demand
C	Carbon
Cf	Final Concentration
Ci	Initial Concentration
DAF	Dissolved Air Flotation
DO	Dissolved Oxygen
EAC	Extruded Activated Carbon
EDXS	Energy Dispersive X-Ray Spectroscopy
FT-IR	Fourier Transform-Infrared Spectroscopy
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
MF	Micro-Filtration
mg/L	Milligram per Liter
mL/min	Flow rate
mm	Millimeter
NF	Nano-Filtration
PAC	Powdered Activated Carbon
PKS	Palm Kernel Shell
RO	Reverse Osmosis
SEM	Scanning Electron Microscopy
SME	Small and Medium Enterprise
UF	Ultra-Filtration
$ZnCl_2$	Zinc Chloride

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LIST OF SYMBOL

%	Percentage
℃	Temperature in Degree Celsius
Å	Measurement in length (10^{-10} meters)

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The wastewater can be define as flow of the discharge of the used water by all effluent of domestic household, meat packing industries, slaughter houses, institutions, restaurants, hospitals and industries (Aquastat, 2014). The wastewater also can cause adverse effect to the environmental and human health due to formation of carcinogenic substance (Wu et al., 2009).

The oil and grease such as the motor oil can constrain and kill the growth of plants. The motor oil is popular in making a pesticide of weed killer (Bailey, 2019). The wastewater that contaminated with oil and grease can be categorised as hazardous waste if the waste generated enter into the water bodies without any proper treatment. It can cause the adverse effect to the aquatic and marine life by lowering the biochemical oxygen demand (BOD) level of the water bodies due to formation of layer on the water surface (El-Gawad & S, 2014).

The removal of pollutant in wastewater are considered important nowadays. The needed of water resources either in developed, developing or undeveloped country are very high demand. In developed and developing countries, they sustain their water quality resources by the water treatment applications.

The treatment plant normally used to treat the oil and grease in wastewater such as skimming tanks methods and oil and grease traps methods are known as the

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conventional techniques. These both techniques worked but taking a long time due to its low efficiency of removal (El-Gawad & S, 2014).

Activated carbon or widely known as activated charcoal can be defined as a carbon-based materials that been produced to increase the tolerance in adsorptive medium and yielding higher adsorbent materials. Activated carbon is one of the high cost conventional techniques that can be used in wastewater treatment plant. Removing the oil and grease in the wastewater using activated carbon absorption is a physicochemical type of treatment (Chinnayai & Kavitha, 2002). The treatment of physicochemical is the treatment that can change the physical state of colloidal particles by using the chemical to make them more stable and coagulable for further treatment (Chokhavatia Associates, 2019).

Activated carbon has the properties of thermo-stability, high performance, high adsorptive effect, large surface area and well-developed structure (Yahya, 2018). There are quite a lot of types of activated carbon that were produced from the agricultural wastes such as the corn cob, risk husk, palm trunk, coconut shells and wood that can be used in remove the residues in wastewater (Tamer Alslaibi, 2012).

1.2 Problem Statement

The wastewater that contaminated with oil and grease that were discharged by the industries and factories into the wastewater system will cause a serious problems to the environment if the oil and grease is not treated properly. The wastewater of oil and grease that were directly discharged by the vehicle workshop into the environment will lead to the contamination of surface water and to the ground water system.

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The present of oil and grease in water bodies will led to the formation of oil layer that can cause the reduction of oxygen in the water bodies which leads to dead of marine and aquatic life (El-Gawad & S, 2014). The wastewater that contaminated with oil and grease will need to be treated properly so that the issues towards environmental can be reduced.

In this study, the palm kernel shell from the agricultural waste have been chosen as the main materials of activated carbon in removal of oil and grease in industrial wastewater system. The palm kernel shells can be collected from the palm industries because it is considered as one of agricultural waste. The oil palm industry in Malaysia has been known as one of the biggest producers and exporters of the oil palm to the worldwide (Journal of Oil Palm Environment & Health, 2012).

1.3 Expected Outcomes

In this study, the expected outcome is the ability of the palm kernel shells as a activated carbon to adsorb and reduce the oil and grease concentration in wastewater system. The further study will be conducted to know the ability of palm kernel shells activated carbon in wastewater treatment plant that polluted with oil and grease.

1.4 Objectives

The objectives of this experiment are as follows:

1. To determine the optimum parameters affecting column adsorption by using different contact time, flow rate and initial concentration of oil and grease in wastewater.
2. To characterize palm kernel shells activated carbon using FTIR.

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1.5 Scope of Study

The scope of study in this study is to remove the oil and grease that existed in wastewater from the industrial sector. The method that will used in removal the oil and grease in wastewater is by the physical-chemical method which using the activated carbon as the adsorbent.

The activated carbon were collected from the palm kernel shells which is one of the agriculture waste from the palm plantation or palm industry. The wastewater that contaminated with oil and grease were collected from vehicle workshop. The wastewater of oil and grease were undergo gravimetric process to obtain the percentage of the oil and grease concentration before and after the experiment.

The optimum parameters affecting column adsorption by different flow rate, contact time and initial concentration of oil and grease in wastewater were conducted. The palm kernel shells (PKS) activated carbon samples of raw, unused and used will be characterized by using Fourier Transform Infrared Spectroscopy (FTIR)

1.6 Significant of Study

The significance of this study is to have a better quality of water for the community consumption by treating the discharge of wastewater from all sources including from the businesses, hospital, domestic households, industries and institutions that are contaminated with oil and grease. The oil and grease can remain in the pipes system of the sewage treatment. The remaining of oil and grease can become a blockage and next will damage the pipes of the sewage treatment. The treated wastewater from the sewage treatment will not cause damage to the pipes system of the wastewater treatment plant due to earlier treatment at the sewage.

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In this day and age, the awareness of the important to have a better environment to the human health have been increase by conservation and sustain it. In this study, the oil pollution toward water can be reduce. The widely used of the activated carbon in removal of oil and grease can increase the water quality for the environmental and human health. The activated carbon that are produced by palm kernel shells can decrease the agricultural waste of the palm kernel shells and also can increase the economy which are generated from waste to money.

This study will help to reduce the environmental issues at once will generate income to the economy of Malaysia. The uses of palm kernel shells before to be known can be give benefits to human and environment, it will be burned or dumped the palm kernel shells on the plantation site. This practices will cause an environmental issues such as air pollution due to open burning of the palm kernel shells.

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CHAPTER 2

LITERATURE REVIEW

2.1 Oil and Grease in Wastewater

2.1.1 Sources of Oil and Grease

The largest source of the wastewater that contaminated with oil and grease are from the production of oil and the extraction of oil 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). The discharge of oil and grease in wastewater also can be found in domestic household such as cooking, cosmetics, repairing that using oil and grease, slaughter houses, textiles and automobiles industries. The 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, Weisbrod, & Gross, 2008).

The wastewater from the automobile workshop is one of the highest contributor to the oil and grease in wastewater and also contribute to the water pollution (Manilal, 2019). 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).

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2.1.2 Effect of Oil and Grease in Wastewater

The several of toxicity are different among the types of oils and greases. The crude oil are less toxicity than the refined oil (Adams et al. 2017). The adverse impact to the human health problems can be affected by the various hydrocarbons which existed in the fuels. For example, it can lead to the liver and kidney damage, cancer and anemia from mutagenesis (El-Gawad & S, 2014).

Next, the adverse impact to the environment can decrease the ability for aquatic organisms to reproduce and survive due to the oil pollution. The studies have been conducted by the previous research indicated that 0.3 - 0.6 mg/L of certain aromatic hydrocarbon can be poisonous to the aquatic organisms (Stenstrom, 2019).

Then, oil and grease also can give effect to the sewer and the pipelines under the ground. The oil and grease can cause the blockages to the pipelines during the oil and grease in wastewater passes through it. The oil and grease are insoluble particles in the water, which it need the different treatment from other. If the oil and grease are not be treated properly, the oil and grease will accumulates and stick to the pipelines and cause the clogs to the pipelines and to the sanitary sewer system.

2.1.3 Malaysia Effluent Discharge Standard

The guidelines of the amount of oil and grease and other types of wastes that can be discharge to the water flow has been listed in the Environmental Laws of Malaysia, Environmental Quality Act 1974 and the Malaysia Environmental Quality (Industrial Effluent) Regulation 2009. Oil and grease in wastewater from industry and housing area falls under the Fifth Schedule under Environmental Quality (Industrial

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Effluent) Regulations 2009 (Environmental Quality (Industrial Effluent) Regulations 2009).

Based on the Table 2.1, the amount for the oil and grease that can be discharged is less than 1.0 for Standard A while less than 10 for Standard B. Standard A is applicable to discharge into any inland waters within catchment areas listed in the Third Schedule, while Standard B is applicable to any other inland waters or Malaysian waters.

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

	Parameter	**Unit**	**Standard**	
			A	**B**
	(1)	(2)	(3)	(4)
(i)	Temperature	℃	40	40
(ii)	pH Value	-	6.0-9.0	5.5-9.0
(iii)	BOD at 20ºC	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

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(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

2.2 Palm Kernel Shells

Palm kernel shells is the product of the plantation or agricultural waste from the palm plantation. The palm kernel shells is one of the waste that will be burned or dumped along with others by product of the palm plantation. The palm kernel shell are known as a natural high grade solid biofuel and has high energy density. The palm kernel shells are widely be used as a biomass resource. According to Zafar (2019) in the Palm Oil industries, the left-over of by-products and the application rate of these products are low. As the palm kernel shells has lower moisture content compared to other biomass residues with different sources between 11% and 13% of the moisture content (Zafar, 2019).

The application of the palm kernel shell is relatively low, thus causing it to accumulate and increase in number (Olutoge et al, 2012). There are another application of palm kernel shell which it can be used as alternative fuel for biomass-based

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combination of heat and power plants to produce heat and power for industrial boiler, furnace and foundries, bricks kilns, residential and commercial heating and light weight aggregate for concrete (Global Green Synergy, 2008). The features of the palm kernel shell are high calorific value, low sulphur content, excellent year-round availability and no species variation (Global Green Synergy, 2008).

2.3 Activated carbon

2.3.1 Characteristic

Activated carbon is well known as an activated charcoal which it has a different characteristic depend on the composition of the initial raw materials. The activated charcoal is a carbonaceous material that has a large internal surface area and high develop porous structure which cause by the high temperature reaction. The previous research stated that the large surface area within 500 m^2g and 1500 m^2g and electrical charge effectively adsorb a wide range of polar compounds, notably phenols and their derivatives (Jackson, 2014).

According to Ebbenis (2019), there are three main types of activated carbon that were produced which are powdered activated carbon (PAC), granular activated carbon (GAC) and extruded activated carbon (EAC). The PAC is in the powdered state on activated carbon which in range of the size 5 to 150 Å, while GAC is in a granular particles which in range of size 0.2 mm to 5 mm. meanwhile, the EAC is in cylindrical pellet in range of size 1.0 mm to 5.0 mm. There are also several additional types of activated carbon which are bead activated carbon, impregnated carbon, polymer coated carbon, activated carbon cloths and activated carbon fibers.

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The ability of higher adsorption and chemical reactions is because of its large specific surface area and the large develop porous structure of the activated carbon. The ability of the porous structure in the activated carbon is to adsorb materials in the phase of liquid and gas. The adsorption in the liquid-phase is much easier than in the gas-phase because of the experimental protocols of simply mixing the two phases (Dipendu Saha, 2017). The parameters such as polarity of the contaminants or compound, the pH of the solution, the contact time between activated carbon and the contaminant, temperature and the concentration of the compound are the ability of the adsorption of activated carbon (Edalat, 2008). Besides, the varieties of the characteristic of activated carbon should be include as well. For example, the pore structure, hardness or abrasion, adsorptive properties, apparent density, moisture, ash content, pH value and the particle size (Ebbenis, 2019).

2.3.2 Fourier Transform Infrared Spectroscopy, FTIR

The Fourier Transform Infrared Spectroscopy (FTIR) or also known as FTIR analysis is an analytical technique. Commonly, the FTIR analysis were used to identify the organic and polymeric materials. Under the specific circumstances, the FTIR analysis also can be used in identify the inorganic materials (Laboratories, 2015).

The sample of the raw blended and sieved PKS, the unused PKS activated carbon and the used PKS activated carbon will be conducted under the FTIR. The FTIR is used to gain the information on the chemical structures and the functional groups of the prepared PKS activated carbon by measures the absorption of infrared radiation by the sample material versus wavelength. The infrared absorption bands identify molecular components and structures (Materials Evaluation and Engineering,

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2019). The samples will be characterized after the sample were dried to remove the excesses moisture of the samples.

Laboratories (2015) stated that in the final result, the FTIR analysis of the PKS activated carbon will able to identify and characterize the unknown materials either in films, solids, powders or liquids. Next, the FTIR will able to identify the contamination on or in a material either in particles, fibers, powders or liquids. Lastly, to identify additives after extraction from a polymer matrix and identifications of oxidation, decomposition, or uncured monomers in failure analysis investigations of the PKS activated carbon.

2.3.3 Activation Method

The methods in activation are the activation of the material or known as carbonization process of the material. In this experiment, the carbonization process will be applied on the palm kernel shells (PKS) in making the PKS activated carbon. The methods of the activated carbon can be categories into dry and wet oxidizing agents which both are the different methods. 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). The dry oxidation methods involve the reaction of hot oxidizing gas such as steam and carbon dioxide at the temperature of 700°C (Kathiraser, 2015).

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2.3.4 Application of Activated Carbon

The application of the activated carbon is a worldwide range application in removing the contaminants or pollutants in waster or wastewater by the adsorptions in liquid or gas form (Sivakumar, Kannan, & Karthikeyan, 2012). The use of activated carbon can improve the environmental quality and human health due its harmless properties and reactions toward the environment and human.

The activated carbon has been widely used in ancient times as the absorbent in medicines and as a purifying agents. The activated carbon also has been used during the World War I as a gas mask to prevent and protect from the hazardous gas and vapors (Jacoby, 2014).

In current time, the activated carbon is one of the alternative agent in water treatment plants or wastewater treatment plant. Besides, the application of activated carbon also widely used as a beauty product, in pharmaceutical and food product. The utilization of the activated carbon as the corrosion prevention in the pipelines that help protect systems and piping from corrosive chemical species (Jacobi Carbon Group, 2019).

According to Ebbenis (2019), basically the application of the activated carbon were applied in the water purification industries, air purification, metals recovery industries, foods and beverages and in the medicinal sectors.

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2.4 Previous Study in Removing Oil and Grease in Wastewater using Activated carbon

Based on the Table 2.2, these are the previous study of the removal of oil and grease using the activated carbon.

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 shows 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 using Natural Adsorbent	The activated carbon was extracted from Curry leaf, Banana pith and Neem leaf. Neem leaf shows the higher effective removal of oil and grease in wastewater.	(Balaji. et al, 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)

2.5 Other Techniques that were Used in Removal of Oil and Grease in wastewater

In removal of oil and grease in the wastewater, there are several techniques that can be applied in this treatment. According to the Ariana Pintor (2016), there are three treatment that can be used in removal of oil and grease in wastewater. The primary treatment, secondary treatment and tertiary treatment that are shown in Figure 2.1.

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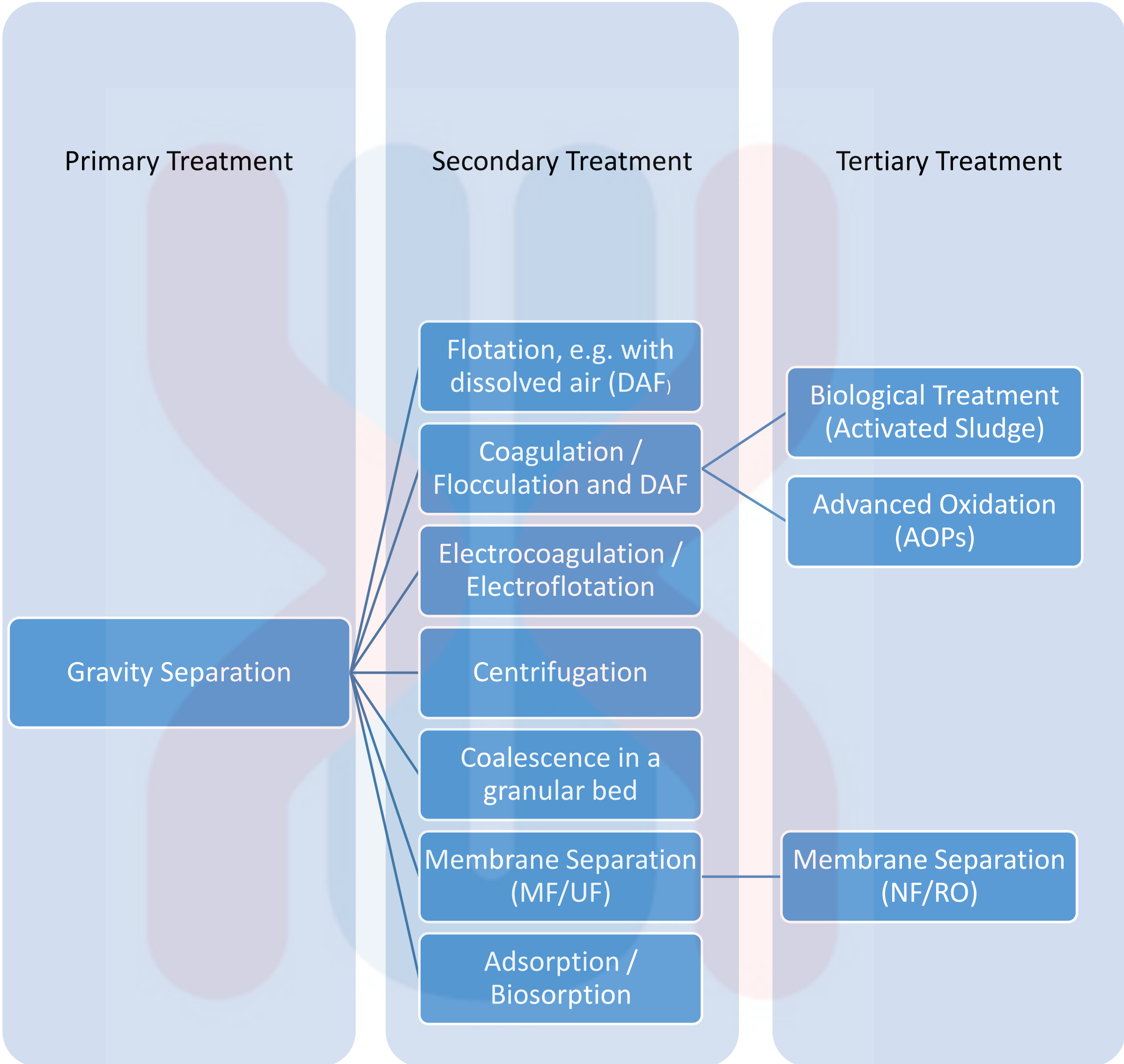

Figure 2.1 The other techniques that can be used in treatment of oil and grease in wastewater.

In this study, the technique that were used in removal of oil and grease in wastewater is by adsorption. The adsorption is a technique that are widely used in removal of the low concentration of inorganic pollutant in the wastewater such as in removal the oil and grease in the wastewater (VITO, 2015). The primary treatment is the gravity separation which used in removal of free oil. The treatment that using the chemical, electrical and physical methods such as coagulation/flocculation, Dissolved Air Flotation (DAF), electrocoagulation/flotation and membrane separation either in Multi-Filtration (MF), Ultra-filtration (UF), Nano-Filtration (NF)

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or Reverse Osmosis (RO). In the tertiary treatment or also known as other secondary treatment involves tighter membranes and Advanced Oxidation Processes (AOPs) (Ariana Pintor, 2016).

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CHAPTER 3

METHODOLOGY

3.1 Materials and Reagent

The wastewater sample of oil and grease were collected at the local industry of vehicle and motorcycle workshop that located in Kota Bharu, Kelantan. Palm Kernel Shells (PKS) were collected from the factory of Palm Plantation in Felda Kemahang, Kelantan. The chemical reagent of N-hexane were used in the experiment as a solvent.

3.2 Wastewater of Oil and Grease Characterization

The wastewater that contaminated with oil and grease were collected using the sampling bottle to conduct the ex-situ analysis. The collected waste water were undergo the gravimetric method analysis to determine the concentration of oil and grease in the collected sample.

3.2.1 Gravimetric Analysis of Oil and Grease

Gravimetric methods depend on the weight as a means of quantifying an analyte for some final determination. Gravimetric analysis are the oldest techniques that are widely used in environmental analysis which requires long time and tiresome to complete it. There are four fundamental types of gravimetric analysis which are physical gravimetric, thermogravimetry, precipitation gravimetric analysis and

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electrodeposition (Department of Chemical Engineering, 2004). Each four fundamental types of the gravimetric analysis are different.

3.3 Wastewater In-Situ Analysis

The in-situ analysis were conducted using the YSI Multiparameter. The parameter of temperature, total dissolved solid (TDS), pH, salinity and dissolved oxygen (DO) were analysed by in-situ method.

3.4 Methods

3.4.1 Preparation of Palm Kernel Shells Activated Carbon

The palm kernel shells (PKS) were collected from the palm plantation factory. The PKS were washed, dried under the sun until fully dry and were crushed into smaller pieces. The PKS were crushed by using a blender and were sieved using a 2.0 mm sieve. The sieved PKS were placed into crucible and were closed with its lid. Next, the closed crucible were wrapped with aluminium foils.

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

After 2 hours, the PKS and KOH solution mixture were filtered using a vacuum pump and was dried overnight in oven at 120 ℃. The dried sample were placed in the

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muffle furnace for 15 minutes to be carbonized again at 800 °C. The dried sample were cooled at room temperature and were washed until the neutral pH were obtained using hot or cold distilled water. After obtaining neutral pH, the sample were dried in the oven at 120 °C. The sample or prepared activated carbon were cooled and stored in the desiccators for further analysis.

3.4.2 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 were connected to the source of the wastewater to the column through the peristaltic pump. The height of the PKS activated carbon were maintained the height of 5 cm throughout the experiment. A set up without the PKS activated carbon present in the column were done as a control experiment.

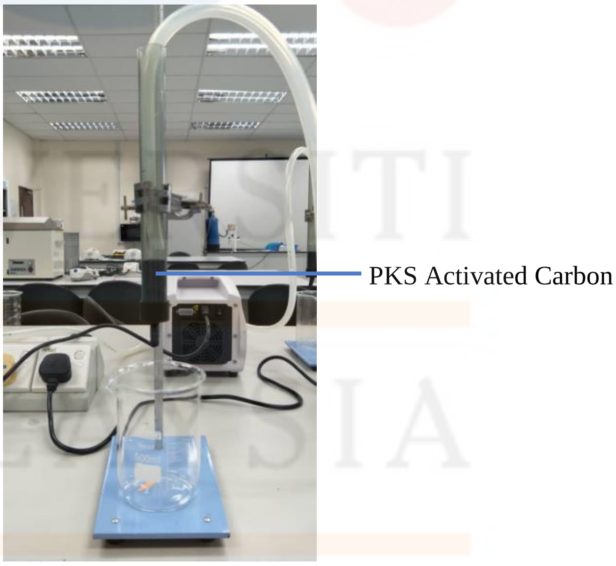

Figure 3.1 Column Adsorption Set-Up

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3.5 Adsorption Analysis

3.5.1 Effect of Oil and Grease in Different Contact Time

The wastewater of oil and grease will be pumped into the column that were filled with PKS activated carbon. The height of the PKS activated carbon was measured at 5 cm and were kept constant throughout the adsorption process. The wastewater flow rate were kept constant at 1 mL/min and the contact time of 24 hour. The wastewater that passed through the column were collected every 2 hours in 24 hours and were followed by analysed using gravimetric method.

3.5.2 Effect of Different Flow Rate

Flow rate is the volumetric fluids that passes through per unit time (Study.com, 2018). The oil and grease wastewater with the optimum concentration of oil and grease which will be obtained by the experiment conducted from Section 3.5.1 were pumped into the column that were filled with PKS activated carbon. The flow rate of the oil and grease wastewater will set into 1 mL/min, 3 mL/min and 5 mL/min. The wastewater that flowed through the column were collected every 2 hours for 6 hours and were analysed using gravimetric method.

According to Mishra (2015), the concentration of the fluid were affected by the flow rate. As the flow rate of the increase, the concentration of the oil will increase. The final result were obtained in a form of a graph which represent the optimum concentration of oil and grease vs. flow rate.

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3.6 Gravimetric Analaysis

In this study, the wastewater that has passed through the PKS activated carbon in the column were extracted with n-Hexane. Hydrochloric acid solution (HCl) were added with the ratio of 1:1 to decrease the pH to 2 or less and act as sample preservation.

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

The extraction process were done three times using a separating funnel and 5 ml of n-hexane were used as a solvent. The extracted solution in the round bottom flask were extracted again to rinse the extraction solution. The final extracted solution were undergone distillation process in order to remove the n-Hexane from the solution. The solution were dried in the oven at 105 ℃ overnight to remove water from the round bottom flask.

The residue in the round bottom flask were weighed using analytic electronic balance. To obtain constant weight the round bottom flask were weighed a three 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 were calculated by the following equation:

$$\text{Removal Percentage of oil, \%} = \left(\frac{(Ci - Cf)}{Ci}\right) \text{x } 100 \qquad (3.1)$$

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Where Ci represented as the initial reading of the sample and Cf is the final reading of the sample.

3.7 Characterization of activated Carbon using Fourier Transform Infrared Spectroscopy, FTIR

The sample of raw PKS, unused PKS activated carbon and used PKS activated carbon were prepared to be analysed under the FTIR. All the samples were fully dried under the sun after the adsorption analysis and were crushed into powder. FTIR can analysed the material in the state of solid, liquid or powder to obtain the actual graph for the material. The wave number that were used to analyse are between 4000 – 500 cm^{-1}. The brand of FTIR that were used to analyse the raw PKS, unused PKS activated carbon and used PKS activated carbon is 8400S of Shimadzu.

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CHAPTER 4

RESULT AND DISCUSSION

4.1 Wastewater In-Situ Analysis

The result of in-situ analysis for wastewater sample are shown in Table 4.1. All the parameters were followed based on Water Quality Standard (WQI) guidelines to achieve the discharging of wastewater by industries.

Table 4.1 The result of in-situ analysis for the wastewater

Parameter	Reading
Temperature (℃)	25.3
Total Dissolve Solid, TDS (%)	2.0
Dissolved Oxygen, DO (%)	2.3
Salinity	1.1
pH	6.5

Based on the Table 4.1, the reading for the parameter were collected using the YSI Multiparameter. The temperature that were obtain were 25.3 ℃ which are the normal temperature of wastewater discharge into the drain system. According to the guidelines in Table 2.1, the temperature for the industrial discharge both for Standard A and Standard B are below 40 ℃. The total dissolved solid (TDS) that were contained in the wastewater were 2.0 % which below the values in guidelines in Table 2.1. This is due to the wastewater were contaminated with all the waste from

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other sources such as cigarettes and dust. The dissolved oxygen (DO) were 2.3 % or 0.19 mg/L. The dissolved oxygen were lower in percentage because of the presence of oil and grease in the wastewater. The presence of oil and grease prevent the oxygen from the atmosphere dissolved in the water. Meanwhile, the data for salinity and the pH of the wastewater that were analysed were 1.1 % and 6.5 which can be considered as normal range for neutral pH. The presence of salinity in the wastewater of oil and grease were because of the foods and drinks that were thrown in the wastewater along with the discharged of the oil and grease. The pH value in Table 2.1 stated that the condition for the industrial discharge or mixed effluent of Standard A and Standard B are between 6.0 – 9.0 and 5.5 – 9.0.

4.2 Adsorption Analysis

4.2.1 Effect of Oil and Grease in Different Contact Time

Figure 4.1 shows the removal efficiency of oil and grease for initial concentration for every 2 hours in 24 hours contact time. The data for this graph were collected through the gravimetric process. The final weight of the oil and grease after completing the gravimetric process were collected three times to get the average weight of the oil and grease. The removal efficiency of the oil and grease were calculated using the formula in Section 3.6.

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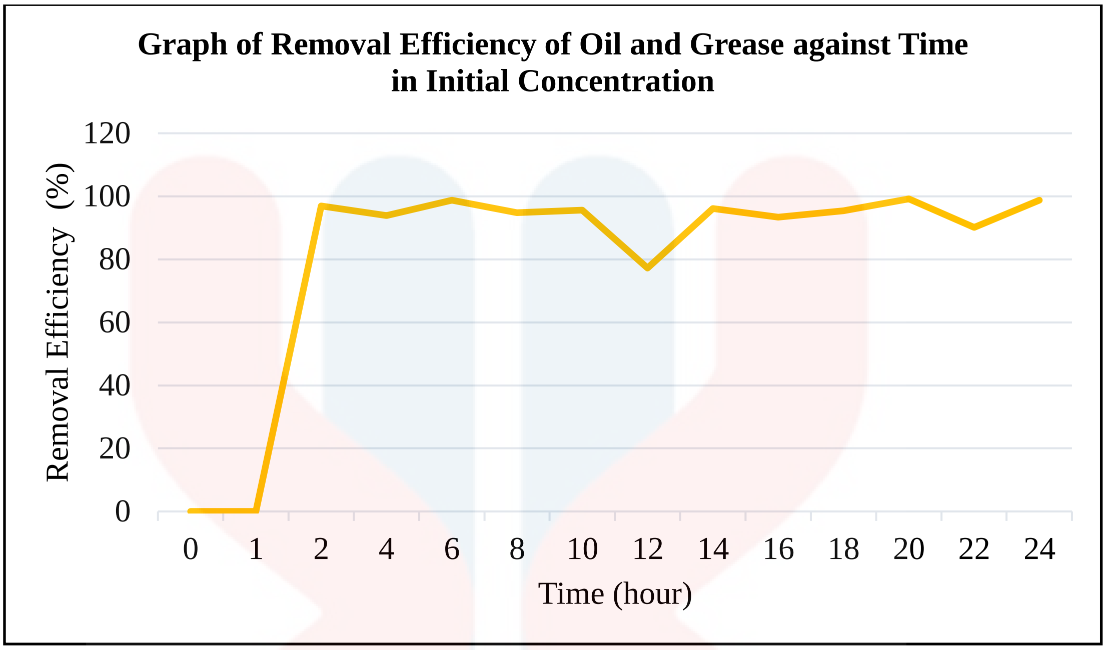

Figure 4.1 Graph of removal efficiency of oil and grease (%) against time (hour) for flow 1mL/min

The result from the Figure 4.1 were analysed on the initial concentration of oil and grease with the same flow rate at 1 mL/min for the column adsorption while the reading were taken from 0 hour until 24 hours in the contact time. At the beginner of experiment which is at 0 hour, the initial concentration of the oil and grease was obtained at 57500 mg/L. At the 0 hour, the gravimetric method was conducted to obtain the initial weight of the oil and grease from the wastewater source to identify the initial concentration of the oil and grease.

The graph shows the initial concentration of the oil and grease for the next 2 hours was obtained at high percentage of 96.90 % removal of the oil and grease in the industrial wastewater. The high percentage of removal efficiency at the first 2 hours is because of it was the first reaction between the contact time of the wastewater source and the PKS activated carbon in the adsorption column. Besides, in the first 2 hours period, most of the oil and grease was trapped on the top layer of the PKS

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activated carbon in the adsorption column which make the longer period to absorb the oil and grease.

The concentration of oil and grease at the first 4 hours contact time until the first 6 hours period of the contact time shows the increment of the 4.91 % of the removal efficiency of oil and grease which each are at 93.90 % and 98.81 %. However, at the 12 hours period of the contact time, the removal efficiency was drop at 77.27 % of the removal efficiency of oil and grease in the wastewater source. The data that were collected for the 24 hours period of contact time shows the unstable of the removal efficiency of oil and grease in the wastewater source.

Meanwhile, the removal efficiency of oil and grease was increased again at the period of 20 hours of contact time. This is the highest percentage the removal efficiency of oil and grease in the wastewater source which are at 99.18 % and then it decreased again at 90.13 % of the removal efficiency of oil and grease. This is because the longer period of the contact time with of the PKS activated carbon has become more saturated that been filled with the concentrated of oil and grease.

According to the Khaled Okiel (2010) when the length of the column increase, the removal of the oil and grease increase and the concentration of the oil and grease is decrease. The final result of this effect of concentration of oil and grease in different contact time will be produced in a form of a graph shows the relationship between the initial concentration of oil and grease vs. contact time.

Figure 4.2 shows that the result that were obtain in the control experiment. The control experiment was conducted to obtain the actual data of the removal efficiency of oil and grease in the wastewater source. The experiment control were conducted with the same set-up adsorption analysis but with absence of PKS

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activated carbon in the adsorption column. The purpose in conducting this control experiment is to obtain the actual data for the removal of oil and grease in the wastewater as they tend to stick around the walls during the adsorption process and the main purpose is to gain the exact data that were not trapped on the PKS activated carbon during conducting the experiment control.

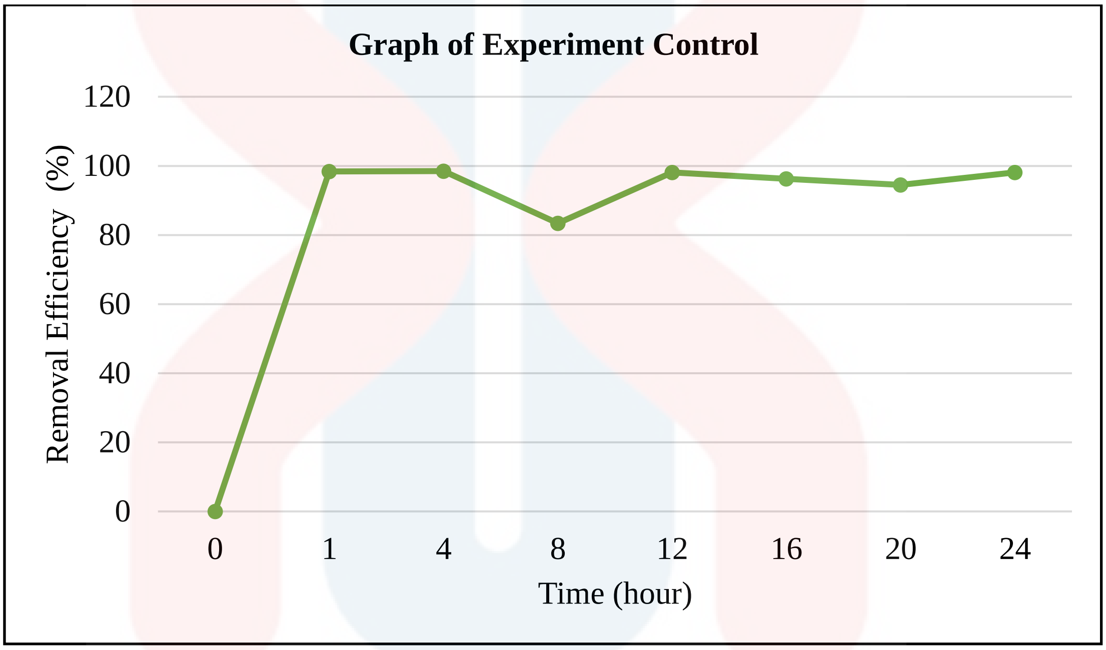

Figure 4.2 Graph of the experiment control

The data for the control experiment that were collected in every 4 hours period of the contact time in 24 hours. The result shows that at the first 4 hours of contact time, the wastewater of oil and grease that were obtain are 98.49 % which are higher than the result in the actual experiment. The increment between removal efficiency of oil and grease and in the experiment control are 4.59 % of removal efficiency.

The comparison between the result in both control experiment and the actual experiment had the unstable data for the removal efficiency of oil and grease. The data of the removal efficiency of oil and grease in the control experiment are greater than the data in actual experiment. This because of the absent of the PKS activated carbon in the adsorption process which the PKS activated carbon act as the filter as it adsorb the oil and grease in the wastewater when it flows on it.

Generally, in removal efficiency of oil and grease in the wastewater, the PKS activated carbon do act as a filter in removing the oil and grease in the wastewater sources. According to Okoroigwe et. al. (2013), PKS activated carbon works effectively as the water treatment material. The period of soaking in the activation process and the carbonization period affected the level of the water purification (Okoroigwe et. al., 2013)

4.2.2 Effect of Different Flow Rate

Figure 4.3 shows the effect of different flow rate which are 1 mL/min, 3 mL/min and 5 mL/min with the selected concentration of oil and grease that were obtained in the previous experiment in Section 4.2.1 which is the initial concentration of oil and grease. The reading of the removal efficiency were collected in every 2 hours for 6 hours started from 0 hour to 6 hours period of contact time. The different flow rate for 1 mL/min, 3 mL/min and 5 mL/min were adjusted using the peristaltic pump.

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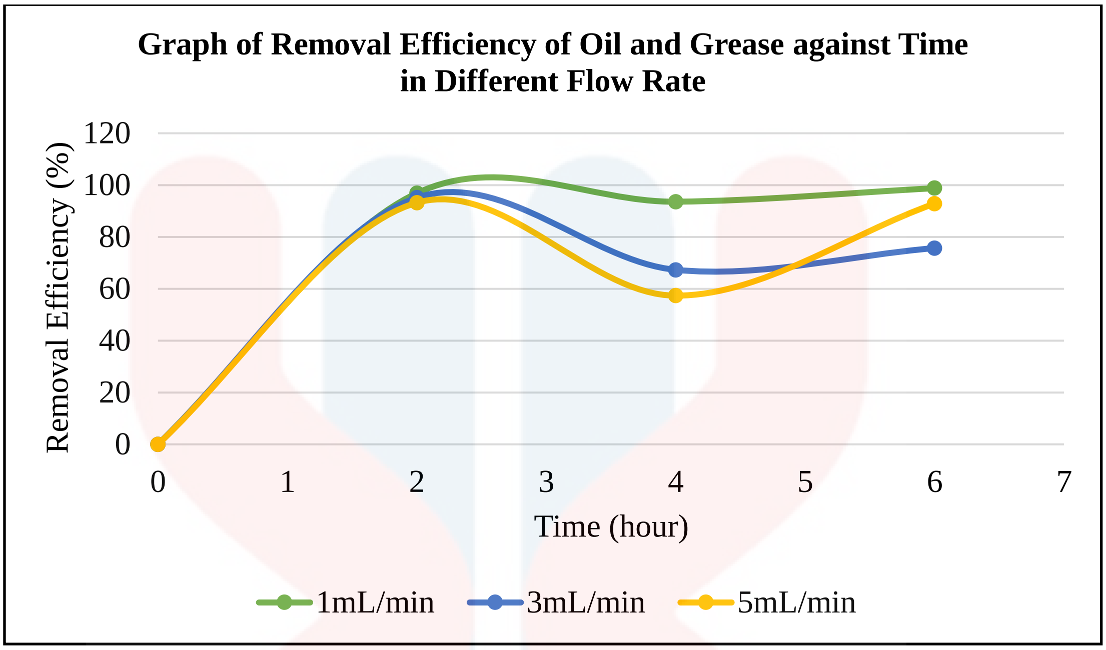

Figure 4.3 Graph of different flow rate with the initial concentration of oil and grease

The result in the graph of different flow rate at 1 mL/min, 3 mL/min and 5 mL/min with the initial concentration of oil and grease were examine to identify the effect of the different flow rate against the initial concentration of oil and grease in the wastewater. Based on the Figure 4.3, the highest removal efficiency of the oil and grease in the wastewater was at 1 mL/min of the flow rate in the 6 hours periods of contact time with 98.81 % of the removal efficiency of oil and grease. As the flow rate decrease, the removal efficiency of the oil and grease in the wastewater increase. At the first 2 hours period of contact time in 1 mL/min of flow rate, the removal efficiency of oil and grease are at 96.90 %. The efficiency of removal of oil and grease decreased with increased of the flow rate (Hebbar & Jayantha, 2013). This is due to the time taken of the PKS activated carbon to absorb the oil and grease that were passed through it were longer with the slowest flow rate. The slower flow rate have longer period of contact time during the adsorbent process (Hebbar & Jayantha, 2014).

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For the initial concentration of oil and grease at the 5 mL/min of flow rate, the data that were obtained are slower than the data at the 1 mL/min for the first 2 hours with 93.21 % of the removal efficiency. The overall result that were obtained shows that the higher of flow rate, the lower of the removal efficiency of oil and grease in wastewater.

The control experiment for the different flow rate with initial concentration were conducted to see the changes of the removal efficiency of the oil and grease in wastewater.

4.3 Characterization of PKS Activated Carbon using Fourier Transform Infrared Spectroscopy, FTIR

The PKS activated carbon were characterized under the Fourier Transform Infrared Spectroscopy, FTIR. FTIR is known as an analytical technique that determine the chemical structure and the functional group of the element. To determine its chemical structure and its functional group, the certain wavenumbers were choose which between 4000 - 500 cm^{-1}. The x-axis represent the wave numbers of infrared spectrum between 4000 – 500 cm^{-1} which in category of the mid-range wave numbers. While the y-axis represent the amount of infrared being transmitted or absorbed by the analysing of elements.

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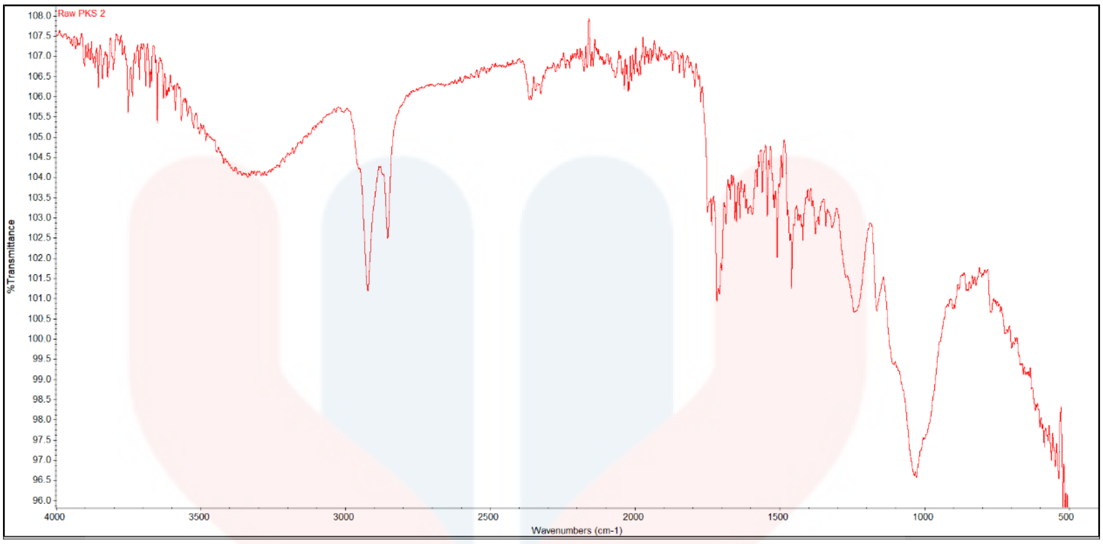

Figure 4.4 The functional group for raw PKS under FTIR

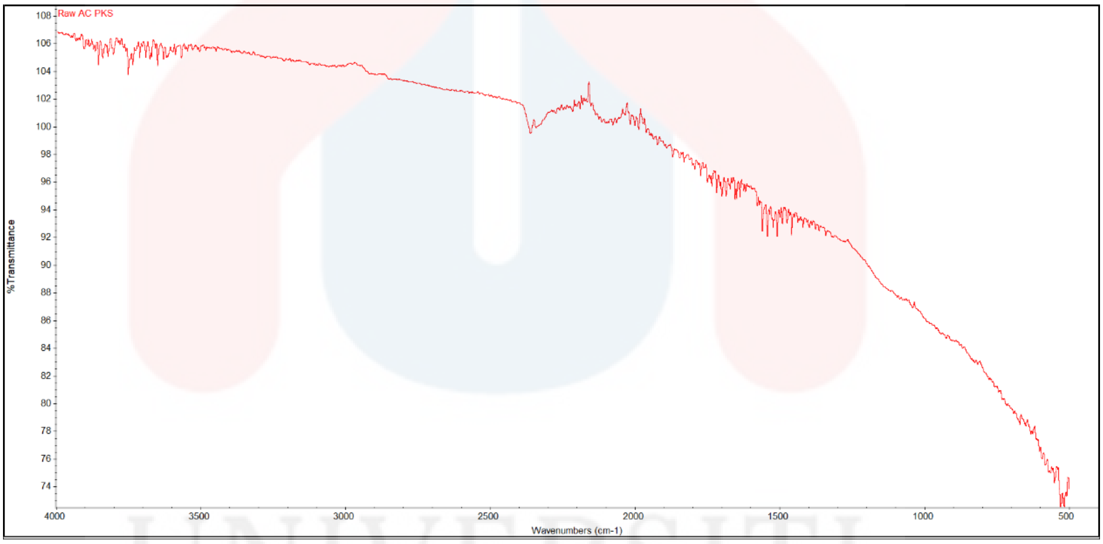

Figure 4.5 The functional group for unused PKS activated carbon under FTIR

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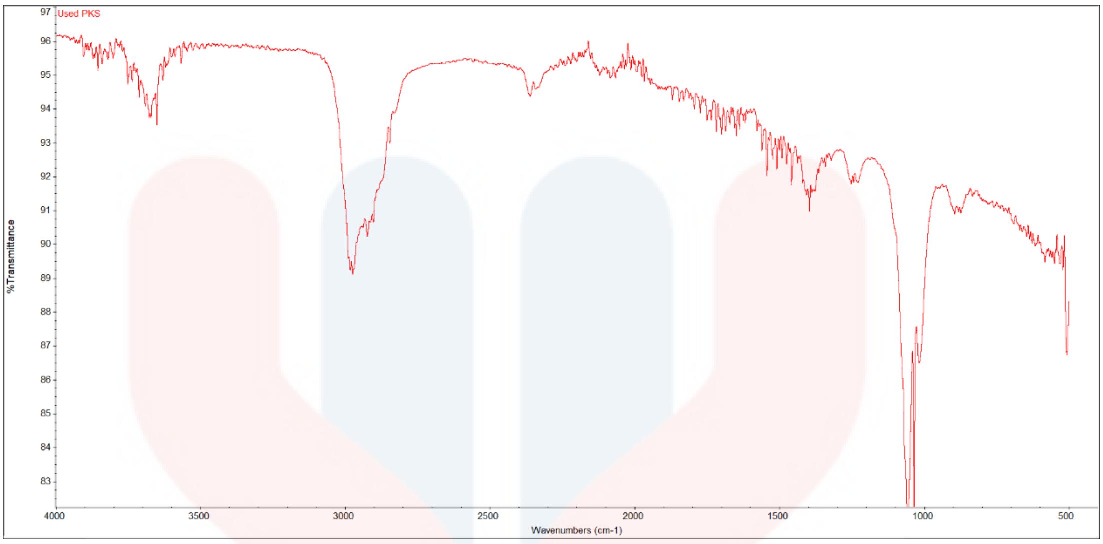

Figure 4.6 The functional group for used PKS activated carbon under FTIR

Based on the Figure 4.4, Figure 4.5 and Figure 4.6 shows the raw images of the raw PKS, unused PKS activated carbon and used PKS activated carbon were analysed under FTIR. The each figures shows different functional group of the PKS. In Figure 4.4 shows that the functional group is in the inorganic phosphate. Inorganic phosphate are salt in phosphoric acid with the metal ion which present in all living organisms include human, plants and animals (Alfa Aesar, 2019).

Meanwhile, the functional group for unused PKS activated carbon are Hydrocarbon as shown in Figure 4.5. The differences between Figure 4.5 and Figure 4.6 were the present of the moisture content. In Figure 4.5, the moisture content such as water content were not presented and dried caused by the carbonization process and produced the less peak than Figure 4.6.

The functional group for the used PKS activated carbon are the Aliphatic Hydrocarbon as shown in Figure 4.6. Hydrocarbon can be divided in two classes which are aromatic compound and non-aromatic compound. The Aliphatic Hydrocarbon or also known as non-aromatic compound are less harmful to human and environment than the aromatic compound. As the aromatic compound have

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double chain compared to the non-aromatic compound with single chain which why the Aliphatic Hydrocarbon fall under the non-aromatic compound.

The amount of infrared that were transmitted or absorbed by the raw PKS were at 107.5 %, by the unused PKS activated carbon were at 106.8 %, and 96.1 % were transmitted or absorbed by the used PKS activated carbon. Based on the Figure 4.4, the raw PKS obtained the highest percentage of the infrared that were transmitted or absorbed at the 4000 cm^{-1} of the wave number under the FTIR analysis. The difference percentage of the transmittance or adsorbent of the infrared for raw material and the prepared activated carbon that were analysed under FTIR were affected by the activation method (Hesas et al., 2013). The activation method in prepared activated carbon reduced a significant amount of the hydrogen which decrease the number of the transmittance or adsorbent of the infrared (Hesas et al., 2013).

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CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

This study shows that the PKS activated carbon do act as the wastewater purification as it can remove the oil and grease in the wastewater source. In the column adsorption analysis, the longer period of the contact time, the slower removal efficiency of the oil and grease. This is because of the longer contact time, the PKS activated carbon become more saturated with the oil and grease. This can be seen at the 12 hours of contact time, the value of the removal efficiency of oil and grease were decreased to 77.27 % from the wastewater sample.

Next, the study of the PKS activated carbon with the different flow rate column adsorption analysis for 1 mL/min, 3 mL/min and 5 mL/min shows that its can affect the removal efficiency of the oil and grease in the wastewater treatment. As the result that obtained in 6 hours period of contact time, the slower flow rate, the greater removal efficiency of oil and grease. The flow rate at 1 mL/min has the higher removal efficiency of oil and grease compared to the flow rate of 3 mL/min and 5 mL/min.

In conclusion, the used of the PKS activated carbon in the wastewater purification or wastewater treatment does act as a filter that remove the pollutants from it. The further study need to be conducted in order to achieve the consistency value of the removal efficiency of oil and grease.

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5.2 Recommendation

The improvement for the study of the removal efficiency of oil and grease by using the PKS activated carbon need to have a further study on it. The concentration of oil and grease, the period of the contact time, the flow rate and the size and length of the adsorption column need to be determine with precise in order to obtain an accurate result of the removal efficiency of oil and grease.

For further study in characterization of the Palm Kernel Shells (PKS) activated carbon can be enhanced. There are a few technologies and instruments that can be used in determine the characterization of the activated carbon. The physical method that can be use is the Energy Dispersive X-Ray Spectroscopy (EDXS). According to Materials Evaluation and Engineering (2019), EDSX is a chemical microanalysis technique used in combining with the Scanning Electron Microscopy (SEM). The characterization of the elemental composition in selected volume of analysing by the EDSX technique can detect the x-rays that emitted from the sample by an electron beam during the penetration. The analytical information can be obtained by qualitative analysis, quantitative analysis, elemental mapping and line profile analysis which provide more accurate in determine the presence of an element in the sample.

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APPENDIX A

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

Table A1 Initial concentration of oil and grease, 1mL/min flow rate

Time (hour)	Mg/L or PPM	Percentage removal (%)
0	57500	0
2	1750	96.90
4	3510	93.90
6	682	98.81
8	2996	94.79
10	2528	95.60
12	13072	77.27
14	2254	96.08
16	3780	93.42
18	2637	95.41
20	471	99.18
22	5677	90.13
24	682	98.81

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APPENDIX B

TABLE OF OIL AND GREASE CONCENTRATION FOR DIFFERENT FLOW RATE

Table B1 Initial concentration of oil and grease, 1 mL/min flow rate

Time (hour)	Mg/L or PPM	Percentage removal (%)
0	57500	0
2	1750	96.90
4	3510	93.90
6	682	98.81

Table B2 Initial concentration of oil and grease, 3 mL/min flow rate

Time (hour)	Mg/L or PPM	Percentage removal (%)
0	57500	0
2	2788	95.15
4	18790	67.32
6	13975	75.70

Table B3 Initial concentration of oil and grease, 5 mL/min flow rate

Time (hour)	Mg/L or PPM	Percentage removal (%)
0	57500	0
2	3903	93.21
4	24520	57.36
6	4110	92.85

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APPENDIX C

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

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

Time (hour)	Mg/L or PPM	Percentage removal (%)
0	57500	0
1	925	98.39
4	870	98.49
8	6874	83.35
12	1092	98.10
16	2136	96.29
20	3175	94.48
24	1079	98.12

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APPENDIX D

PHOTO GALLERY

Figure D1 The workshop

Figure D2 The raw PKS before blend

Figure D3 The 2mm of the raw PKS