POME POLISHING TREATMENT USING PALM KERNEL SHELL ACTIVATED CARBON PRODUCED VIA INDUSTRIAL KILN

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To all my family who gave me encouragement, everlasting love and support throughout my study years

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ABSTRACT

Malaysia is one of the largest producers and exporters of palm oil products in the Asian region. To manage the waste generated from palm oil processing is not easy as some of palm oil mills still fail to comply with the standard discharge limit set by Department of Environment even though biologically treatment unit has been applied. Hence, in this study polishing treatment unit was proposed using adsorption process that improved the 3-days biological oxygen demand (BOD_3), chemical oxygen demand (COD), suspended solid (SS) and colour removing efficiency so that the standard discharge limit can be achieved. The potential of palm kernel shell (PKS) based activated carbon (AC) as adsorbent in removal of organic matters in palm oil mill effluent (POME) was investigated. The precursor was prepared in a large scale environmental charcoal kiln by carbonization under controlled atmosphere and physically activated using large scale rotary kiln using the optimum parameter (steam injection at 5L h^{-1}; temperature 900-1000 ^{o}C). The largest Brunauer-Emmett-Teller (BET) surface area of palm kernel shell activated carbon (PKS AC) obtained was 607.8 m^2/g with the pore volume of 0.25 cm^3/g. The optimum condition to reduce BOD_3 to 17 mgL^{-1} was 50 g/250 mL PKS AC, 24 h mixing time, 150 rpm and at pH 2 whereas COD, SS, and colour value obtained after polishing treatment were 203 mg/L, 145 mg/L and 2640 Pt-Co, respectively. The adsorption equilibrium was best represented by Langmuir and Freudlich isotherm model whilst the kinetics of adsorption was well described by pseudo-first order model. Regeneration study was carried out by thermal treatment. The adsorption capacity for organic matters was 6.08-4.36 mg/g, after five adsorption-regeneration cycles. This study has identified that PKS AC has a potential to be used as a precursor in the preparation of AC to achieve BOD_3 of less than 20 mgL^{-1} in POME polishing treatment.

ABSTRAK

Malaysia merupakan salah satu pengeluar dan pengeksport terbesar produk minyak sawit di rantau Asia. Untuk menguruskan bahan buangan yang dihasilkan daripada pemprosesan minyak sawit bukanlah mudah memandangkan segelintir kilang minyak sawit masih gagal mematuhi had pelepasan standard yang ditetapkan oleh Jabatan Alam Sekitar walaupun unit rawatan biologi telah digunakan. Oleh itu, unit kajian rawatan penggilap menggunakan proses penjerapan telah dicadangkan untuk memperbaiki nilai keperluan oksigen biokimia 3-hari (BOD_3), keperluan oksigen kimia (COD), pepejal terampai (SS) dan kecekapan penyingkiran warna supaya had pelepasan standard boleh dicapai. Potensi karbon teraktif berasaskan tempurung kelapa sawit (PKS) sebagai penjerap untuk menyingkiran bahan organik di dalam bahan buangan sisa minyak sawit (POME) telah dikaji. Pelopor ini telah disediakan di dalam tanur arang mesra alam berskala besar di bawah atmosfera terkawal dan diaktifkan secara fizikal menggunakan tanur berputar skala besar di bawah parameter optimum (suntikan stim pada 5 Lh^{-1}; suhu 900-1000 ^{o}C). Luas permukaan Brunauer-Emmett-Teller (BET) terbesar karbon teraktif tempurung kelapa sawit (PKS AC) dapat dicapai sehingga 607.8 m^2/g dengan jumlah liang 0.25 cm^3/g. Keadaan optimum untuk mengurangkan BOD_3 kepada 17 mgL^{-1} adalah 50 g/250 ml PKS AC, 24 jam masa pencampuran, 150 rpm dan pada pH 2 manakala bacaan akhir COD, SS dan nilai warna yang diperoleh selepas rawatan penggilap adalah masing-masing 203 mgL^{-1}, 145 mgL^{-1} dan 2640 Pt-Co. Keseimbangan penjerapan adalah terbaik dipadankan oleh kedua-dua model isoterma Langmuir dan Freudlich manakala kinetik penjerapan dihuraikan oleh model kinetik pseudo pertama. Kajian penjanaan semula telah di laksanakan dengan rawatan terma. Kapasiti penjerapan bahan organik didapati sebanyak 6.08-4.36 mg/g walaupun selepas lima kitaran penjerapan-penjanaan semula. Kajian ini telah mengenal pasti bahawa PKS AC mempunyai potensi untuk digunakan sebagai pelopor dalam penyediaan AC untuk mencapai BOD_3 kurang daripada 20 mgL^{-1} dalam rawatan penggilapan POME.

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LIST OF SYMBOLS AND ABBREVIATONS

ACs	-	Activated carbons
ASTM	-	American Society for Testing and Materials
BET	-	Brunauer, Emmett and Teller
BOD	-	Biochemical oxygen demand
BOD_3	-	3-days Biochemical Oxygen Demand
CAC	-	Commercial Activated Carbon
Cd	-	Cadmiun
Ce	-	equilibrium concentration
cm	-	centimeter
Co	-	initial concentration
CO_2	-	Carbon dioxide
COD	-	Chemical oxygen demand
CPO	-	Crude palm oil
Cu	-	Cuprum
DOE	-	Department of Environmental
EFB	-	Emplty Fruit Bunches
Fe	-	Ferum
FFA	-	Free fatty acids
FFB	-	Fresh fruits bunches
FTIR	-	Fourier Transform Infra-red
g	-	gram
H_2SO_4	-	Sulphuric acid
H_3PO_4	-	Phosphoric acid
HCl	-	Hydrochloric acid
HRT	-	Hydrolic retention time
IUPAC	-	International Union of Pure and Applied Chemistry

K	-	Kelvin
KOH	-	Potassium hydroxide
L	-	Liter
L/mg	-	liter per miligram
MB	-	Methylene blue
MDF	-	Medium density fiber board
mg L^{-1}	-	Milligram per liter
mg/mg	-	miligram per miligram
Mn	-	Manganese
MO	-	Moringa Oleifera
mol / g	-	mol per gram
MPOB	-	Malaysian palm oil board
MT	-	Metric tonnes
NaOH	-	Sodium hydroxide
NTU	-	Nephelomeric unit
NTU	-	Nephelometric Turbidity Unit
oC	-	Degree celcius
OPF	-	Oil palm frond
OPT	-	Oil palm trunk
PACl	-	Polyaluminum chloride
PAM	-	Polyacrilamide
PKS AC	-	Palm Kernel Shell Activated Carbon
PNP	-	P-nitrophenol
POME	-	Palm oil mill effluent
PU	-	Polyurethane
RO	-	Reverse osmosis
RSM	-	Response surface methodology
S_{BET}	-	BET surface area
SEM	-	Scanning electron microscopy
SEM	-	Scanning electron microscopy
SS	-	Suspended Solid
t	-	time
TGA	-	Thermal gravimetric analyzer

TSS	-	Total suspended solid
UF	-	Ultrafiltration
V_T	-	Total pore volume
wt%	-	Weight percent
$ZnCl_2$	-	Zinc chloride

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CHAPTER 1

INTRODUCTION

1.1 Overview

We are proud that our country have brought oil palm trees from the small scale crop plantation to become the largest commodity product in the world. Malaysia has produced about 17.3 million tons of crude palm oil, and exported the bulk of its products and securing for the country RM44498 million in revenue by 2014 (Malaysia Palm Oil Board, 2014). Oil palm tree is the major crop plantation in Malaysia and the palm oil industry has had an exceptional growth for about four decades ago as the top agricultural industry. Presently, almost half of the agricultural land in Malaysia is under oil palm and the plantation area is still expanding from time to time. Today, 44% of world exports and 39% of world palm oil production were monopolized by Malaysia. Between 1990 and 2015, the palm oil production almost tripled from 6,094,622 to 19,666,953 tonnes per year, ranking Malaysia as the second biggest palm oil producer throughout the world after Indonesia (Malaysia Palm Oil Council, 2015). As the producer and exporter of palm oil and palm oil products, Malaysia plays an important role to fulfill the growing global needs for oils and fats in general.

Throughout the rapid development of the palm oil industry in the country, oil palm tree, its product and also the by-product produced have always been connected to the environment. In 2014, it was estimated that 93.38 million tonnes of empty fruit bunches (EFB), 21.03 million tonnes of oil palm fibre (OPF), and 4.46 million tonnes of oil palm shell (OPS) all together totalled about 118.9 million tonnes of solid biomass were produced (Loh, 2016). The fast and rapid development both upstream and downstream would result to an unmanageable environmental pollution directly.

Currently, palm kernel shell (PKS) is less being exploited other than being used as a secondary fuel for boiler particularly during starting up of burning (Yap and Menon, 2012). The remaining of PKS is usually left abandoned at the landfill site near to the processing mill. For that reason, PKS which is less utilized in the current market is preferred as the raw material for the production of activated carbon.

Similar to other agricultural and industrial activities, oil palm cultivating, milling and processing are also regulated by environmental legislation that aims to conserve and protect the natural environment. These rules and regulations play an important role to minimize the degradation of the atmospheric, soil as well as water in the environment (Fairhurst and Mutert, 1999). To produce palm oil products, large amount of water is consumed, and consequently, it will generate tremendous volume of wastewater. Palm oil mills are the main source of palm oil wastewater other than palm oil refineries. The water pollution known as palm oil mill effluent (POME) is generated mainly from the sterilization process during crude oil production. POME water pollution is very synonymous to palm oil processing. Approximately, 30 million tonnes of POME are generated annually from more than 300 palm oil mills in Malaysia. An outstanding water pollution in the environmental system, enforcement must to be placed in its treatment system.

In 1978, the Environmental Quality Regulations which described the discharge standards for POME had been enacted. Several specifications for the discharge limit of POME had been implemented and one of the parameter was the Biochemical Oxygen Demand (BOD). Initially,the BOD discharge from the mill was from 25000 ppm of the untreated POME, the value was later reduced in the first generation of discharge standard to 5000 ppm and was further reduced to the present BOD of 100 ppm (Malaysia, 1977).

1.2 Background of Research

The POME is brownish in colour and has a colloidal suspension containing 95-96% water, 4-5% total solids, and 0.6-0.7% oil and grease. It also contains high BOD and voluminous industrial waste. Over half of the total solids are dissolved solids, while the other half is a mixture of various form of inorganic and organic suspended solids. It is a thick liquid with a temperature of 80-90°C and acidic nature with a pH value around 4.0-5.0 during discharge in the cooling pond. With this characteristic, together with a high BOD value and low pH, makes this liquid waste not only difficult to treat by conventional methods but also highly polluting the environment. According to Bhatia et al., (2007b), the production of 985,603 tonnes of crude palm oil requires 1,477,595m^3 of water to produce 38,797m^3 of POME. Thus, effective POME treatment technologies are required to meet DOE discharge limits i.e. 400ppm TSS and 100ppm BOD to control water stream pollution.

Currently, there are several treatment methods that have been used by palm oil mill industries in Malaysia. Most of them are using the facultative anaerobic ponds, tank digestion and mechanical aeration, facultative ponds and tank digestion, facultative ponds and decanter, biological and physicochemical treatment. Apparently, this treatment method had also tried out several methods including membrane technology, up flow anaerobic sludge fixed film bioreactor, up flow anaerobic filtration, and aerobic sludge blanked. Presently, according to Vijayaraghavan et al., (2007) about 85% of the POME treatment method use the anaerobic and facultative ponding technology followed by the open tank digester together with aeration in the pond.

From previous research studies (Ahmad et al., 2003; Ahmad et al., 2005a, Ahmad et al., 2006; Ariffin et al., 2005; Mohamed and Lau, 1987; Ng et al., 1987; Bhatia et al., 2007b), it was reported that, most of the POME treatments used the physicochemical method (i.e. coagulation/flocculation method). However, most of the researches that were conducted focused more on the treatment of POME in the primary stage.

In the current situation, most of the treatment plants use the anaerobic digestion to treat POME followed by an aerobic oxidation in facultative and maturation or algae ponds. Thus, in this research study, it is intended to reduce the COD, BOD_3, SS and colour. The polishing part using activated carbon as an adsorbent will be used at the end of the treatment so that the value of the parameters could meet the Department of Environmental (DOE) standards. The polishing treatment is also aimed to bring the BOD_3 level to below 20 mgL^{-1}. The efficiency of the activated carbon adsorbent will be assessed by treating the final discharge of the effluent obtained from the treatment plant located at Labu, Negeri Sembilan.

1.3 Problem Statement

Through extensive research on the biological POME treatment technology, it was found that many treatment plants failed to achieve the standard discharge limits established by DOE. The final treated effluent discharged into the water courses still contains a high BOD_3, COD, SS and colour value which can certainly cause problems to the environment (Wu et al., 2009). For that reason, an alternative for POME treatment system is highly required to meet standard discharge limits. It is believed that tertiary treatment is able to improve the quality of the effluent since there is an attempt to enforce 20 mg/L BOD_3 discharge limit on crude palm oil (Liew et al., 2015). Therefore, in this study, the absorption process from activated carbon as an option to treat biologically treated POME will be investigated. This study will explore the possibility of using activated carbon as a polishing step in POME treatment for reducing BOD_3 to below 20 mg/L.. The production of PKS AC in a large scale using environmental kiln for carbonization and rotary kiln for activation will also be practiced.

1.4 Objectives of Research

This research project is designed to achieve the following objectives

i. To synthesize and characterise AC from PKS produced from industrial kiln.
ii. To study the effect of the synthesized PKS AC towards POME polishing treatment by assessing the removal of BOD_3, COD, SS and color.
iii. To study the equilibrium and kinetic of the adsorption process.
iv. To perform the regeneration study of the PKS AC.

1.5 Scope of Research

The steps and the scopes leading to the objectives are:

1. Characterization of raw PKS collected from Felcra Berhad Sungai Melikai Oil Palm Mill Mersing Johor, PKS charcoal and PKS AC on proximate (moisture, volatile matter, ash and fixed carbon), ultimate analysis (C, H, N, O, S content), lignocellulosic analysis (cellulose, hemicellulose and lignin), thermogravimetric analysis (TGA), Fourier

Transform Infrared Spectroscopy (FTIR), pore development, and morphology using Scanning Electron Microscopy (SEM).

2. To characterize biologically treated POME sample by finding the value of BOD_3, COD, SS and color for every different sample taken from the mill.

3. To investigate the optimum dosage (0-125g), mixing time (20-140 hours), mixing rate (45-200rpm), and pH (2-12) of PKS AC in reducing the pollutant load (BOD_3, COD, SS and color).

4. To analyse the equilibrium (Langmuir and Freundlich isotherm method) and kinetics study (Pseudo-first order & Pseudo-second order) of PKS AC sorption.

5. To compare the performance of PKS AC with commercial AC available in the market.

6. To perform saturation and regeneration studies (5 cycles) of PKS AC and compare to CAC.

1.6 Significance of Research

After the enforcement of the rules and regulation according to the standard discharge limit for crude palm oil industry under Environmental Quality (Prescribed Premises) (Crude Palm Oil) Regulations 1977, Malaysia and other crude palm oil industries are dealing with the challenge of sustainable development, palm oil economic feasibility, and most important is the balancing the environmental protection. Focusing on this country, the number of palm oil mills effluent discharge failed to meet the required standard limit established by the DOE, even biological treatment methods has been employed. This research study is purposely done to make an up-grading of the existing wastewater treatment plants in order to comply with the standards stipulated by the authorities. By applying the adsorption process in the final part at the treatment plant is expected will significantly improve the treatment systems and thus reduce the liability of the effluent discharge to the environment.

1.7 Thesis Outline

This thesis is divided into 5 main chapters. Chapter 1 explains the introduction of this whole research work. This chapter consists of research background, problem statements, objectives, scopes and significance of this research. Literature Review are given in Chapter 2 which explains in detail about the previous researches that are related to POME treatment and production of activated carbon as well as researches that are relevant to this area. Chapter 3 has been described as the experimental procedures such as PKS AC preparation, POME characterization, analytical methods involved to evaluate the efficiency of PKS AC as well as the comparison between commercial AC and finally the regeneration of PKS AC. The next chapter is Chapter 4, where it explains in detail the results and discussions for the whole study. Finally, Chapter 5 concludes the findings and significance of this study. Recommendations are also suggested in assurance the positive outlook of this research area.

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