UTILISING OIL PALM BIOMASS AS AN EFFICIENT BIOADSORBENT FOR TREATING FINAL DISCHARGE OF PALM OIL MILL EFFLUENT

By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

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Chair : Prof Ts Suraini Abd-Aziz, PhD
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The abundance of agricultural waste from Malaysia palm oil industry, as the world's second-largest palm oil producer, presents an opportunity to convert it into value-added products. However, the discharge of palm oil mill effluent (POME), a substantial liquid waste, poses a challenge to achieving a sustainable palm oil industry. Treating POME is difficult for palm oil mill owners due to the stringent discharge limits set by the Department of Environment (DOE) Malaysia regarding pollutants and colour levels. This study focuses on the utilisation of oil palm kernel shell activated carbon (OPKS-AC), derived from oil palm biomass, as a tertiary treatment for POME final discharge through adsorption. The results indicated a successful reduction of pollutants and colour within the POME final discharge, achieving up to 91% reduction in biochemical oxygen demand (BOD), 87% reduction in chemical oxygen demand (COD), 74% reduction in total suspended solids (TSS), and 97% reduction in colour after treatment with OPKS-AC. The isotherm and kinetic analyses demonstrated that OPKS-AC exhibited significant adsorption capabilities for removing pollutants and colour from POME final discharge, making it a promising renewable bioadsorbent alternative to commercial activated carbon. The isotherm data of BOD, COD, TSS, and colour adsorption onto OPKS-AC are fitted well to the Langmuir model with R^2=0.996, 0.994, 0.999, and 0.993 and χ^2=3.964x10^{-4}, 0.034, 7.430x 10^{-4}, and 0.495. Meanwhile, the kinetic adsorption of BOD, COD, TSS, and colour onto OPKS-AC can be modelled by using the pseudo-second order (PSO) model, (R^2 = 0.991, 0.983, 0.982, and 0.994) and (χ^2= 0.7x10^{-4}, 0.012, 0.009 and 0.188). In addition to the batch adsorption method, the study explored a continuous adsorption system, which is crucial for assessing the suitability of OPKS-AC as an alternative tertiary treatment for POME management. The continuous adsorption approach offers flexibility for larger-scale experiments and requires less time. Meanwhile, the batch adsorption method helps to understand the adsorption mechanism of adsorbates onto OPKS-AC. Process optimisation was carried out using Design Expert 11.0 software to evaluate the significant operational variables and their interactive effects on pollutant (COD and TSS) and colour removal from POME final discharge. The optimal operational conditions for pollutant and colour removal were determined as a flow rate of approximately 30.31 ml min^{-1}, an adsorbent quantity of 2.13 kg, and a treatment duration of 3.62 hr resulting in optimal removal percentage of 98.05, 98.80, and 99.20% for COD, TSS and colour from POME final discharge, respectively. In conclusion, the use of OPKS-AC as a bioadsorbent for treating POME final discharge offers significant potential for reducing pollutants and colour levels, contributing to the sustainability of the palm oil industry in Malaysia. The continuous adsorption system, in combination with the batch

adsorption method, provides valuable insights into the adsorption process and enables optimisation of operational variables for enhanced treatment efficiency.

Keywords: activated carbon, adsorption, oil palm kernel shells, optimisation, palm oil mill effluent

SDG: GOAL 12: Responsible Consumption And Production

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

MENGGUNAKAN BIOJISIM KELAPA SAWIT SEBAGAI BIOPENJERAP EFISIEN UNTUK MERAWAT PELEPASAN AKHIR AIR SISA KILANG KELAPA SAWIT

Oleh

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Limpahan sisa pertanian daripada industri minyak sawit Malaysia sebagai pengeluar minyak sawit kedua terbesar di dunia, meningkatkan peluang untuk penukaran kepada produk nilai tambah. Walau bagaimanapun, pelepasan air sisa kilang kelapa sawit (POME), sejenis sisa cecair yang banyak, menimbulkan cabaran untuk mencapai industri minyak sawit yang mampan. Rawatan POME adalah sukar bagi pemilik kilang kelapa sawit disebabkan had pelepasan yang ketat yang ditetapkan oleh Jabatan Alam Sekitar (JAS) Malaysia berhubung tahap bahan pencemar dan warna. Kajian ini memberi tumpuan kepada penggunaan karbon teraktif tempurung kelapa sawit (OPKS-AC), diperolehi daripada biojisim kelapa sawit sebagai rawatan tertiari untuk pelepasan akhir POME melalui penjerapan. Hasil kajian menunjukkan kejayaan pengurangan bahan pencemar dan warna dalam pelepasan akhir POME, mencapai sehingga 91% pengurangan dalam keperluan oksigen biokimia (BOD), 87% pengurangan dalam keperluan oksigen kimia (COD),

74% pengurangan dalam jumlah pepejal terampai (TSS), dan 97% pengurangan warna, selepas rawatan dengan OPKS-AC. Analisis isoterma dan kinetik menunjukkan bahawa OPKS-AC mempamerkan keupayaan penjerapan yang ketara bagi menyingkirkan bahan pencemar dan warna daripada pelepasan akhir POME, menjadikannya biopenjerap yang dijamin keberkesanan, boleh diperbaharui, pengganti kepada karbon teraktif komersial. Data isoterma penjerapan BOD, COD, TSS, dan warna pada OPKS-AC sesuai dengan model Langmuir dengan R^2=0.996, 0.994, 0.999, dan 0.993 serta χ^2=3.964x10^{-4}, 0.034, 7.430x 10^{-4}, dan 0.495. Sementara itu, penjerapan kinetik BOD, COD, TSS, dan warna pada OPKS-AC boleh dimodelkan dengan menggunakan model pseudo-tertib kedua (PSO), (R^2 = 0.991, 0.983, 0.982, dan 0.994) dan (χ^2= 0.7x10^{-4}, 0.012, 0.009 dan 0.188). Sebagai tambahan kepada kaedah penjerapan kelompok, kajian ini meneliti sistem penjerapan berterusan, yang penting untuk menilai kesesuaian OPKS-AC sebagai rawatan tertiari alternatif untuk pengurusan POME. Pendekatan penjerapan berterusan menawarkan fleksibiliti untuk eksperimen berskala lebih besar dan memerlukan masa yang lebih sedikit. Sementara itu, kaedah penjerapan kelompok membantu untuk memahami mekanisme penjerapan bahan menjerap pada OPKS-AC. Pengoptimuman proses telah dijalankan menggunakan perisian Design Expert 11.0 untuk menilai pembolehubah operasi penting dan kesan interaktifnya terhadap penyingkiran bahan pencemar (COD dan TSS) dan warna daripada pelepasan akhir POME. Keadaan operasi yang optimum untuk penyingkiran bahan pencemar dan warna dikenal pasti apabila kadar aliran kira-kira 30.31 ml min^{-1}, kuantiti penjerap berjumlah 2.13 kg, dan tempoh rawatan selama 3.62 jam

menghasilkan peratusan penyingkiran optimum sebanyak 98.05, 98.80, dan 99.20% untuk COD, TSS dan warna daripada pelepasan akhir POME, masing-masing. Kesimpulannya, penggunaan OPKS-AC sebagai biopenjerap untuk merawat pelepasan akhir POME menawarkan potensi besar untuk mengurangkan tahap bahan pencemar dan warna, menyumbang kepada kelestarian industri minyak sawit di Malaysia. Sistem penjerapan berterusan, digabungkan dengan kaedah penjerapan kelompok, memberikan kefahaman berharga mengenai proses penjerapan dan memperbolehkan pengoptimuman pembolehubah operasi untuk keberkesanan rawatan yang ditambah baik.

Kata Kunci: air sisa kilang kelapa sawit, karbon teraktif, pengoptimuman, penjerapan, tempurung kelapa sawit

SDG: *GOAL 12: Responsible Consumption And Production*

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LIST OF SYMBOLS

Symbol		Unit
C	Intraparticle diffusion constant associated to the thickness of the boundary layer	mg g^{-1}
C_0	Initial concentration	mg L^{-1}
b	Langmuir constant associated with the adsorption energy	L g^{-1}
C_e	Concentrations at equilibrium	mg L^{-1}
C_t	Concentrations at time	mg L^{-1}
K_1	Rate constant of Pseudo-first order kinetic model	min^{-1}
K_2	Rate constant of Pseudo-second order kinetic model	g (mg min)$^{-1}$
K_3	Rate constant of Intraparticle diffusion kinetic model	mg (g min)$^{-0.5}$
K_F	Freundlich equilibrium constant related to the degree of the adsorption capacity	mg g^{-1} (L mg $^{-1}$) 1/n
n	Freundlich equilibrium constant	
R^2	Coefficient of determination	
R_L	Equilibrium parameter	
q_{cal}	Calculated data	mg g^{-1}
q_e	The amount of adsorbate adsorbed at equilibrium	mg g^{-1}
q_{exp}	Experimental data	mg g^{-1}
Q_o	Maximal adsorption capacity	mg g^{-1}
q_t	The amount of adsorbate adsorbed at time	mg g^{-1}

α	Initial adsorption rate	(mg g^{-1}) min
β	Initial desorption rate	g mg^{-1}
χ^2	Pearson's Chi-square	
β_0	Constant coefficient	
β_i	Linear coefficients	
β_{ii}	Quadratic coefficients	
β_{ij}	Interaction coefficients	
ε	Statistical error	

LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
AOP	Advanced oxidation process
BET	Brunauer-Emmett-Teller
BOD	Biochemical oxygen demand
CCD	Central composite design
CH_4	Methane
CO_2	Carbon dioxide
COD	Chemical oxygen demand
CPO	Crude palm oil
CSPO	Certified Sustainable Palm Oil
DO	Dissolved oxygen
DOE	Department of Environment
DoE	Design of Experiment
EDLC	Electrochemical double-layered capacitor
GHG	Greenhouse gas
HTC	Hydrothermal carbonisation method
IPD	Intraparticle diffusion
MF	Microfiltration
MPOB	Malaysian Palm Oil Board
MSPO	Malaysian Sustainable Palm Oil
NF	Nanofiltration
O&G	Oil and grease
OFAT	One Factor at Time

•OH	Hydroxyl radical
OPEFB	Oil palm empty fruit bunches
OPF	Oil palm frond
OPFFB	Oil palm fresh fruit bunches
OPKS	Oil palm kernel shell
OPKS-AC	Oil palm kernel shell-activated carbon
OPMF	Oil palm mesocarp fibre
OPT	Oil palm trunk
PFO	Pseudo-first order
POME	Palm oil mill effluent
PSO	Pseudo-second order
PC	Pseudocapacitor
RO	Reverse osmosis
RSM	Response surface methodology
RSPO	Roundtable on Sustainable Palm Oil
SEM	Scanning Electron Microscope
TC	Thermal carbonisation
TSS	Total suspended solid
UF	Ultrafiltration
3D	three-dimensional

CHAPTER 1

INTRODUCTION

1.1 Research background

The adsorption process is a surface phenomenon that occurs when substances come into contact with a solid surface. The solid surface where the substances attached is termed as the adsorbent while the adsorbate is referring to the adsorbed substance accumulated on the adsorbent (Theodore et al., 2017). This approach is one of the most popular methods and is currently considered as an effective, efficient, and economic method for the wastewater purification including for the palm oil mill effluent (POME) final discharge treatment, the massive wastewater released from the palm oil industry. One of the most extensively employed adsorbent material is the activated carbon as it possesses abundant microporous structure, large specific surface area and high hydrophobicity (Hu & Xu, 2020). The commercial activated carbon is expensive, and is commonly derived from non-renewable materials such as coal (bituminous, anthracite, and lignite), and peat (Menya et al., 2018).

The common preparation method for activated carbon involves either chemical or physical treatment. Physical or "thermal" treatment typically includes the activation process using steam or carbon dioxide following the carbonisation process. As for the chemical activation, the precursors are impregnated with an activating reagent and then subjected to a heating process under an inert atmosphere (Yahya et al., 2015). Physical activation offers a notable advantage over chemical activation by avoiding the incorporation of impurities from the chemical agents. Besides, chemically activated carbon has a considerable amount of inorganic content, which make the physical activation is preferable in terms of environmental safety, due to its exclusion of chemical reagents (Zhou et al., 2018).

In sustainable development and the circular economy, extensive research has focused on making activated carbon from renewable and cost-effective sources like agricultural waste and lignocellulosic materials (Abuelnoor et al., 2021). Jiunn Boon Yong et al. (2022) reported that, throughout the production process, coal-based activated carbon consumes 35% more cumulative energy demand than the biomass-derived activated carbon, leading to a more significant environmental footprint. The biomass waste from the palm oil industry seems to be a suitable candidate for the production of renewable bioadsorbent for POME final discharge treatment. Malaysian Palm Oil Council (MPOC) had reported that Malaysia as the world's second largest palm oil producer and exporter, had accounted for 25.8% and 34.3%, respectively, of global palm oil production and exportation in 2021 (Nusrat Aman et al., 2023). The tremendous production of palm oil products inevitably creates a large amount of biomass waste as only 10% of the total biomass is accountable for palm oil products (Lee et al., 2021). This study aims to assess the capability oil palm kernel shell-activated carbon (OPKS-AC) to be an outstanding bioadsorbent for the treatment of POME final discharge wastewater. Instead of using a two-steps process, this study utilised the two in one carbonisation-activation system, in which the physical activation was continuously conducted in the same reactor as the carbonisation process without interrupting the operation. Without compromising on the yield and the quality of activated carbon produced, this system is cost-efficient and capable of reducing both time and energy consumption (Nahrul Hayawin et al., 2018).

The environmental impact of wastewater typically will be evaluated by the level of chemical oxygen demand (COD) and biochemical oxygen demand (BOD) contents inside the water. POME wastewater is always associated with high BOD, COD, total suspended solid (TSS) and oil and grease (O&G) level of 10,250-43,000 mg L^{-1}, 15,000-100,000 mg L^{-1}, 46,304-63,192 mg L^{-1} and 130-18,000 mg L^{-1}, respectively. Apart from the afomentioned contaminants, the high colour intensity of POME will negatively affect the penetration of the sunlight into the water course. A minimised sunlight penetration process will reduce photosynthetic activities and eventually cause oxygen depletion in the aquatic ecosystem (Mohammad Saleh et al., 2020). Thus, before POME final discharge can be released into the environment, the contaminants and colour level of this wastewater need to be reduced.

1.2 Problem Statement

Palm oil industry requires a vast water consumption to integrate basic unit operations for palm oil production, particularly the palm oil milling process. To generate a tonne of crude palm oil (CPO), approximately 5.0-7.5 tonnes of water are required with more than half of the used water will be released as

POME wastewater (Dashti et al., 2022). The most common treatment for POME wastewater is by using the open ponding system which were applied for almost 85% of palm oil mills across Malaysia. The ponding system involved several types of ponds to remove pollutants from the wastewater. However, the POME from the final discharge pond is still cannot comply to the discharge standard regulated by the Department of Environment (DOE) Malaysia.

This work attempts to treat the POME final discharge by applying the adsorption approach exploiting the palm oil industry biomass waste, oil palm kernel shell (OPKS). It is a necessary move as this process offers a dual benefit to the palm oil industry by producing a value-added resource and at the same time encouraging a sustainable and eco-friendly oil palm biomass waste disposal. The OPKS-AC produced from the two in one carbonisation-activation reactor had proven to be effective in removing the pollutants and colour from POME final discharge (Nahrul Hayawin et al., 2018). However, the studies of the kinetic and equilibrium measure of adsorption onto OPKS-AC are still limited.

It is pretty challenging to completely get the most out of any adsorption process; therefore, by performing the design of experiments (DoE), process optimisation can be achieved. Experimental design can provide a better insight on how an adsorption performance is influenced by operational factors. Many process optimisations are driven by the interactive effects of operational factors. Some critical interactions may go unnoticed if the factorial design is ignored, and overall optimisation will not be achieved (Yahiaoui & Aissani-

Benissad, 2010). Thus, this study utilised a two-level full factorial design to study the significant experimental factors and performed the CCD of response surface methodology (RSM) for the process optimisation of pollutants and colour removal from POME final discharge by employing the continuous adsorption system. The study of OPKS-AC as a bioadsorbent is conducted not only to evaluate its adsorptive capability, but also to discover its potential to be one among many feasible POME wastewater treatments available.

1.3 Research Objectives

This research aims to treat the POME final discharge by removing pollutants such as BOD, COD, TSS and colour while simultaneously exploring the credibility of OPKS-AC as a good bioadsorbent. The specific objectives to achieve the main goal are:

1. To determine the optimum operational parameters for batch adsorption method by using OPKS-AC in treating POME final discharge.
2. To analyse the adsorptive mechanism of OPKS-AC by performing the isotherm and kinetic studies.
3. To optimise the operational factors of a continuous adsorption system utilising OPKS-AC through the RSM approach.

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