UNIVERSITI PUTRA MALAYSIA

UTILIZATION AND OPTIMIZATION OF INDUSTRIAL BOTTOM ASH FOR PALM OIL MILL EFFLUENT DECOLOURIZATION

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By

SYAHIN BT MOHAMMAD SALEH

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

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Chair : Assoc. Prof. Ir. Wan Azlina Wan Abdul Karim Ghani, PhD
Faculty : Engineering

The increasing demand of crude palm oil (CPO) has resulted in the enormous increases in palm oil mills operation in Malaysia and hence the palm oil mill effluent (POME) discharges. Thus, existing treatment facilities have difficulties to comply the discharge limit for this excess POME from the running plants. In this case, colour of POME was one of the major concerns as it will be incorporated in the current regulation. Various adsorbents (i.e POMBA, treated POMBA, CBA, treated PKS) had been screened for POME decolourization. Coal bottom ash (CBA) showed highest colour removal percentage among others and hence selected for the consequence investigations. The raw CBA were then chemically (ACBA) and physically treated (PCBA) for comparison study. Furthermore, response surface methodology (RSM) was used for optimization. As results, the optimum parameters of ACBA and PCBA for POME colour adsorption proposed the followings: 14.20 hr and 15.37 hr contact time, 13.16 g and 13.84 g adsorbent dosage, 27.97% and 57.52% POME concentration, pH 6.24 and pH 6.04, respectively. Both predicted and experimental percentage removal of POME colour for ACBA and PCBA were significantly correlated with the R^2 values for ACBA and PCBA were 0.9793 and 0.9755, respectively. Physico-chemical characterizations of both treated CBA were performed using BET for surface area, FTIR for its surface chemistry, SEM for morphology and EDX for elemental analysis. It was shown that the isotherms for adsorption of colour from POME onto treated CBA were well fitted by Freundlich model. Furthermore, pseudo-second order was identified to be governing mechanism for both treated CBA. Other pollutants i.e COD, BOD_3 and NH_3-N removal are also evaluated. It was found that percentage removal of COD, BOD_3 and NH_3-N using ACBA were 61.33%, 58.33% and 61.43%, respectively. Meanwhile, percentage removal of COD, BOD_3 and NH_3-N using PCBA were 38.39%, 33.33% and 31.10%, respectively. Upon these findings, CBA has potential not only in POME decolourization also in POME treatment as adsorbent.

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PENGGUNAAN DAN PENGOPTIMUMAN ABU MENDAP DARI INDUSTRI UNTUK MENYAHWARNA SISA KILANG MINYAK KELAPA SAWIT

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Pengerusi : Profesor Madya Ir. Wan Azlina Wan Abdul Karim Ghani, PhD
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Peningkatan permintaan minyak kelapa sawit mentah (CPO) meningkatkan operasi kilang minyak kelapa sawit di Malaysia dan begitu juga penjanaan sisa kilang minyak kelapa sawit (POME). Oleh itu, kemudahan rawatan yang sedia ada mempunyai kesulitan untuk mematuhi had pelepasan untuk POME yang berlebihan keluar daripada kilang yang sedang beroperasi. Dalam kes ini, warna POME adalah salah satu kebimbangan utama kerana ia akan dimasukkan ke dalam peraturan semasa. Pelbagai penyerap (i.e POMBA, POMBA dirawat, CBA, PKS dirawat) telah diskrin untuk menyahwarnakan POME. Abu mendap arang (CBA) menunjukkan peratusan pernyingkiran warna yang paling tinggi antara yang lain dan dengan itu ia dipilih untuk akibat penyelidikan. CBA mentah dirawat kimia (ACBA) dan dirawat secara fizikal (PCBA) untuk kajian perbandingan. Tambahan pula, metodologi tindak balas permukaan (RSM) digunakan untuk pengotimuman.Sebagai hasilnya parameter optimum ACBA dan PCBA untuk menyahwarnakan POME mencadangkan: 14.20 jam dan 15.37 jam masa sentuhan, 13.16 g dan 13.84 g dos penjerap, 27.97% dan 57.52% kepekatan POME, pH 6.24 dan pH 6.04. Kedua-dua ramalan dan percubaan penyingkiran warna POME untuk ACBA dan PCBA berkorelasi dengan nilai R^2 untuk ACBA dan PCBA masing-masing adalah 0.9793 dan 0.9755. Pengelasan fiziko-kimia kedua-dua CBA yang dirawat telah dibuat dengan menggunakan BET untuk menganalisa luas permukaan, FTIR untuk kimia permukaannya, SEM untuk morfologi dan EDX untuk analisis unsur. Telah ditunjukkan bahawa isotem untuk menyahwarnakan POME pada CBA yang dirawat telah sesuai dengan model Freundlich. '*Pseudo second order*' telah dikenalpasti sebagai mekanisme yang dikawal bagi kedua-dua CBA yang dirawat. Penghapusan bahan pencemar lain seperti COD, BOD_3 dan NH_3-N juga dinilai. Telah didapati bahawa penghapusan COD, BOD_3 dan NH_3-N menggunakan ACBA masing-masing adalah 61.33%, 58.33% dan 61.43%. Manakala, penghapusan COD, BOD_3 dan NH_3-N menggunakan PCBA masing-masing adalah 38.39%, 33.33% dan 31.10%. Atas penemuan ini, CBA mempunyai potensi bukan sahaja dalam penyahwarna POME malah juga dalam rawatan POME sebagai penjerap.

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LIST OF ABBREVIATIONS

A.P.	Adequate precision
AAm	Acrylamide
ABA	Acid treated BA
AC	Activated carbon
Adj. R^2	Adjusted R^2
ADMI	American Dye Manufacturers Institute
ANOVA	Analysis of variance
AnPOME	Anaerobically treated effluent
APS	Ammonium persulfate
As	Arsenic
BET	Brunauer-Emmett-Teller
BOD	Biochemical oxygen demand
C	Final concentration
C.V.	Coefficient of variance
CaO	Calcium oxide
CBA	Coal bottom ash
CBZ	Carbamazepine
CCD	Central composite design
C_e	Concentration at equilibrium
C_o	Initial concentration
CO_2	Carbon dioxide
COD	Chemical oxygen demand
CPO	Crude palm oil
C_t	Concentration at t
DOE	Department of environment
DOE	Design of experiment
DW	Distilled water
EDX	Energy Dispersive X-Ray
FA	Fly ash
$FeCl_3$	Iron chloride
FFB	Fresh fruit bunches
FTIR	Fourier Transform Infrared
H_2O_2	Hydrogen peroxide
H_2SO_4	Sulphuric acid
H_3PO_4	Phosphoric acid
HBC-POMBA	Hydrogel biochar palm oil mill boiler ash composite
HCl	Hydrochloric acid
HNO_3	Nitric acid
k	Number of variables
K_1	First order rate constant
K_2	Second order rate constant
K_F	Constant related to the overall adsorption capacity
K_L	Langmuir isotherm constant
KOH	Potassium hydroxide
M	Molar concentration
m	Mass
MBA	N,N’-Methylenebisacrylamide

$MgCl_2 \cdot 6H_2O$	Magnesium chloride hexahydrate
MgO	Magnesium oxide
N	Normality
N_2	Nitrogen gas
NaOH	Sodium hydroxide
NH_3-N	Ammonical-nitrogen
P (AAm)	Poly(acrylamide)
Pb	Lead
PBA	Pyrolysis treated BA
PES	Polyethersulphone
PKS	Palm kernel shell
PKSAC	Palm kernel shell activated carbon
POMBA	Palm oil mill boiler ash
POME	Palm oil mill effluent
Pre. R^2	Predicted R^2
Pt-Co	Platinum-cobalt
PVDF	Polyvinylidene fluoride ultrafitration
q_e	Amount of adsorbate adsorbed by adsorbent at equilibrium
q_{max}	Maximum amount of adsorbate adsorbed by adsorbent
q_t	Amount of adsorbate adsorbed at time t
R^2	Coefficient of determination
R^2	Correlation coeffiecient
rpm	Rotation per minute
RSM	Response surface methodology
SEM	Scanning Electron Mscroscope
SMX	Sulfamethoxazole
t	Time
TiO_2	Titanium dioxide
UBA	Untreated BA
V	Volume of solution
x_i	Independent variables
x_j	Independent variables
Y	Predicted response
ZIO	Zinc-iron oxide
Zn	Zinc
$ZnCl_2$	Zinc Chloride
α_0	Coefficient of constant
α_i	Coefficient of linear
α_{ii}	Coefficient of squared
α_{ij}	Coefficient of cross-product
ε_0	Measurement error.

CHAPTER 1

INTRODUCTION

1.1 Research Background

Water crisis is an alarming issues worldwide due to clean water is getting deteriorate. Disposal of wastewater from industrial and commercial sources grab a lot of attention from people and government because it may contain pollutants at levels that could have an impact on environmental conditions of receiving waters. It may cause adverse impacts such as water pollution, creating unpleasant smells and potential health hazard. Commonly, the water discharging from industrial wastes have the most intense coloring i.e. textile industry, paper industry, food industry, and palm oil industry. Colour of surface water is a sign of pollution by the public (Zahrim *et al*., 2014). Colour in water may appear faulty to human because the colour makes it unappealing to drink. It also affects aquatic plant due to the colour reduces photosynthesis activity and dissolved oxygen (Ratpukdi *et al*., 2012). In general, suspended and dissolved particles in water influence colour which may be the result of natural causes or/and human activity. Low accumulation of dissolved materials appears as transparent water and showing low productivity. Moreover, dissolved organic matter, such as peat, humus or decaying plant matter may produce a yellow or brown colour (Perlman, 2016). Different colour of surface water influenced by different sources for instance tannin contributes to the brown colour of the water caused by organic matter coming out from leaves and roots. Palm oil industry is commonly discharging brown colour due to lignin and tannin producing from plants such as oil palm fruits, leaves, root and etc. It was reported that the content of lignin in waste palm shells is 50.7% (Kong *et al*., 2014). Colour of POME still remains even after biological treatment, both aerobic and anaerobic process (Mohammed *et al*., 2014). Even, new and advanced technologies available in the market which are ultrafiltration, membrane bioreactor, coagulation treatment and sequencing batch reactor still the water discharged is brownish and visible in water stream (Jalani *et al*., 2016). Colour pigments are highly stable and resistant to light and moderate oxidative agents, thus they cannot be fully removed by a conventional biological treatment such as activated sludge and anaerobic digestion (Vimonses *et al*., 2009). The palm oil industry has felt uncomfortable tackling issue on this colour removal from POME since it is difficult to be removed. This has drawn the attention of researchers to study on removing them from POME.

Effluent standards regarding to colour level have been applied under Environmental Quality (Industrial Effluent) Regulation 2009. According to the fifth schedule under Environmental Quality (Industrial Effluent) Regulation 2009, level of colour cannot be discharged more than 100 American Dye Manufacturers Institute (ADMI) units (Environmental Quality Act 1974). If the level of the colour discharge exceeds the standard, then it is considered as water pollution. Clean and safe water is needed to be the concern on in order to avoid any environmental and human health impact and of course to save it for future generation as well as gain greener image for our country. Otherwise, polluted water is ingested by people may ill and with prolonged exposure, it may develop cancers or bear children with birth defects. The major water pollutants are chemical, biological, or physical materials which degrade water quality. There are several methods to treat wastewater such as adsorption (Popuri *et al*., 2016), flocculation and coagulation (Madhavi *et al*., 2014), ozonation (Mustapha, 2015), oxidation (Hassaan *et al*., 2017), membrane filtration (Zielińska and Galik, 2017) and microbial biodegradation (Selvakumar *et al*., 2013). Among these methods, adsorption technique seems to be an effective alternative for removal of water pollutants. Activated carbon has been used widely as the adsorbent in adsorption of organic substances including dyes and colours due to its high adsorption capacity and high surface area (Tan *et al*., 2015). Despite its prolific use in water and wastewater industries, commercial activated carbon application is still limited due to its high cost and difficulty in regeneration (Sun *et al*., 2012). This has led to a search for low cost materials as an alternative adsorbent. Several studies reported on alternative low cost adsorbents for colours removal from wastewater i.e. activated carbon prepared from coconut shell (Sia *et al*., 2017), orange peel (Popuri *et al*., 2016), palm kernel shell (Jalani *et al*., 2016), almond gum (Bouaziz *et al*., 2015), fly ash (Asadullah and Rathnasiri, 2015) and sepiolite (Duman *et al*., 2015). Based on the usage of widely available and low-cost materials recovered from waste, study becomes more interesting that has been eco-friendly approach of ‘‘using waste to treat the waste” by adsorption for removal of hazardous pollutants from solution, instead of using expensive adsorbents (e.g. ion exchange resins or commercial activated carbon).

Coal bottom ash is a waste material produced from combustion of solid fuels. It is incombustible waste material of thermal power plant collected from the bottom of the incinerator furnace (Mittal *et al*., 2013). Recently, a number of studies have shown that bottom ash is used as potential low cost adsorbent to remove many types of dyes such as vertigo blue dyes (Kusmiyati *et al*., 2017), basic dyes (Gandhimathi *et al*., 2013), eosin yellow dye (Mittal *et al*., 2013), congo red (Saleh *et al*., 2012) and crystal violet (Nisheesh *et al*., 2012). This is due to the characteristic of CBA which its particle size span, high porosity and large surface area (Cahan *et al*., 2013). There is no literature on the removal of colour from POME so far by using CBA as an adsorbent. This suggests that CBA may have the potential to remove colour from POME under different pH, initial POME concentration, adsorbent dosage and contact time. It is abundantly available since it is an incombustible waste material of thermal power plant (Mittal *et al*., 2013). Disposal of CBA becomes an issue because it requires a larger area for disposal due to increasing generation of CBA waste (Jayaranjan *et al*, 2014). Therefore, the practical use of CBA shows a great contribution to waste minimization as well as resources conservation.

Besides that, a few studies have paid attention to chemical/physical treatment for the adsorbent to enhance the adsorption capacity of adsorbents. This is because adsorbents come from different raw material will give different adsorption capacity. Treatment is necessary in order to achieve optimum adsorption. Treatments of low-cost adsorbent those have been studied includes steam activation of palm kernel shell (Jalani *et al*., 2016), acid activation of fly ash (Asadullah and Rathnasiri, 2015), acid activation of coal bottom ash (Jarusiripot, 2014) and alkaline treatment of biomass fly ash (Pengthamkeerati *et al*., 2010). Some of the studies, chemical and physical treatment was combined to activate the adsorbent such as microwave and chemical activation of palm kernel shell and empty fruit bunch (Mohammed *et al*, 2013), and alkali activation followed by pyrolysis of peanut shell, corncob and cotton stalks biochars (Liu *et al.*, 2016).

1.2 Problem Statement

Coloured water when it is discharged into surface water, the natural colour of the water will change. It does not only reduce water clarity, making aquatic life difficult to find food, also prevent the transmission of sunlight through the water. This causes aquatic plants such as sea-grasses and seaweed do not have enough sunlight for their photosynthesis process which their function is served as nurseries for many important fish species. The problem arises when a lot of industrial wastewaters contain substances that are difficult to remove through conventionally secondary treatment.

The increasing production of major oil palm products generates a high amount of palm oil mill effluent (POME) as a potential environmental pollutant requiring urgent attention due to its considerably “stubborn” color characteristics. This is because even after tertiary treatment, colour still remains. The effluent colour is primarily due to lignin and its degraded products, which chemically stable and resistant to biological degradation. Besides, the treatment conducted by using membrane bioreactor has successfully removed the heavy organic component of POME, however, the discharged water remains coloured due to its by-products (Facta *et al.*, 2010). Another study has mentioned that a high COD can be removed using membrane anaerobic system, however, the permeate shows a high colour with low turbidity (< 10 NTU) due to dissolved solids content with molecular weights < 200,000 g/mol (Wu *et al.*, 2010). Figure 1.1 show the final discharge of POME at Maran, Pahang. Even after ponding treatment, the dark brown colour still remained.

Figure 1.1: Final discharge of POME in Maran, Pahang

Coal bottom ash disposal is abundant as tonnes of CBA are generated daily and end up in near the surrounding land either as wet or dry form. It is produced approximately 25% from coal combustion and it is not widely utilized in Malaysia (Marto and Tan, 2016). The massive generation of CBA results in handling and storage problems such as inadequate facilities or spaces to hold the residues, hence contributes to high capital and operating cost (Rashidi and Yusop, 2016; Jayaranjan *et al*, 2014). It also became authorities concern since its disposal makes the land infertile and is considered unsuitable for agricultural utilization (Nisheesh *et al*., 2012). Power plants stations that located near to urban cities will face difficulties in finding appropriate disposal sites. Thus, an attempt to find an alternative way of CBA utilization is necessary in order to reduce waste disposal and avoid the consumption of increasingly scarce landfill space.

1.3 Research Objectives

The objective of this study is to find an effective and low-cost adsorbent, and to evaluate its potential to remove colour from palm oil mill effluent (POME). The specific objectives of this research include:

1. To assess the physico-chemical properties of coal bottom ash (CBA) and treated CBA as a potential adsorbent. (SEM, EDX, FTIR, BET)
2. To evaluate and optimize treated CBA in colour removal from POME using response surface methodology.
3. To perform the adsorption isotherm and kinetics study of treated CBA adsorption for POME colour removal using Langmuir and Freundlich; Pseudo-first-order and Pseudo-second-order.

1.4 Scope of Research

This research was carried out to evaluate the potential of adsorbent that able to remove colour from POME. Various adsorbents had been screening in order to select the best performance for POME decolourization. Coal-fired plant bottom ash was selected as a potential adsorbent for POME decolourization and was further discussed in this study including adsorbent treatment and optimization. Raw material which was CBA was taken from the local power plant, Stesen Janakuasa Sultan Azlan Shah, Manjung, Perak. CBA was pretreated by rinsing with distilled water several times and drying in an oven at 70℃ for two days. CBA was treated by physical and chemical treatment, respectively. CBA was pyrolyzed with the flowing of nitrogen gas (N_2) at 700 ℃ for 1 hr and was denoted as PCBA. Meanwhile, for chemical treatment, CBA was treated with acid by stirring it with 0.1 M HCl for 24 hours and was left for overnight, later it was rinsed with an excess of distilled water to obtain pH 7. It was denoted as ACBA. Both treated CBA was dried in an oven at 70˚C for two days. Later, samples of CBA were taken into analysis for characterization. The analysis that had been done was Braunauer-Emmeh-Teller (BET) surface area, Scanning Electron Microscope (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and Energy Dispersive X-Ray analysis (EDX). POME sample was prepared by filtering it through filter paper to remove solids following the APHA platinum-cobalt standard method. The sample of POME was taken from Kilang Sawit Sri Senggora, Maran, Pahang. The performance of colour adsorption onto treated CBA was analyzed using spectrophotometer (HACH DR2700) in unit PtCo by measuring the percentage removal. It was analyzed before and after the adsorption process. Optimization was performed using response surface methodology (RSM) to find optimal values to achieve maximum removal. Isotherm and kinetic study were also studied. Other pollutants removal (i.e. COD, BOD_3 and NH_3-N) by using ACBA and PCBA, respectively were also evaluated following the optimization condition of colour removal.

1.5 Significant Contribution

The main contribution of this research is to observe the ability and potentiality of treated coal-fired plant bottom ash to decolourize palm oil mill effluent (POME). This study targets for POME pollution abatement to promote a greener image of the palm oil industry. It also offers the palm oil industry an insight for better effluent management. Besides, the utilization of CBA through this study may contribute to reducing the waste disposal. It has been eco-friendly approach of "using waste to treat another waste" instead of using expensive adsorbent.

1.6 Limitation of Study

Limitation of this study is there is no literature on POME decolourization by CBA as potential adsorbent and there is also limited information on adsorption behavior of POME colour onto the adsorbent. Thus, the properties and characteristic of CBA contributing to adsorption of colour from POME are evaluated throughout this study. This study focuses on utilization of CBA as a potential adsorbent for POME decolourization. Colour of wastewater is quite complicated since it is composed of various components. Hence, this study focuses on colour reduction of POME using CBA by measuring the colour before and after treatment following APHA platinum-cobalt standard method.

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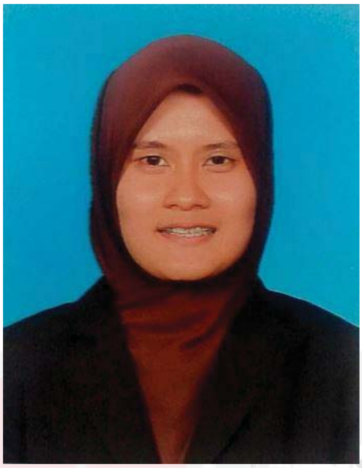

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LIST OF PUBLICATION

A. Published

Saleh, S., Kamarudin, K. B., Ghani, W. A. W. A. K., & Kheang, L. S. (2016). Removal of organic contaminant from aqueous solution using magnetic biochar. *Procedia Engineering,* 148: 228 – 235.

B. Accepted

Saleh, S., Ghani, W. A. W. A. K., & Kheang, L. S. (2019). Treated coal bottom ash for palm oil mill effluent (POME) decolourization. *Journal of Physical Science,* 30(3).

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