

**REMOVAL OF MERCURY (II) ION FROM  
INDUSTRIAL WASTEWATER USING PALM  
OIL FUEL ASH**

**IMLA SYAFIQAH BINTI MOHD SALLEH**

**Master of Science**

**UNIVERSITI MALAYSIA PAHANG**



### **SUPERVISOR'S DECLARATION**

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science in Chemical Engineering.

---

(Supervisor's Signature)

Full Name : Dr. Mior Ahmad Khushairi B. Mohd Zahari

Position : Senior Lecturer

Date : 7 June 2018

---

(Co-supervisor's Signature)

Full Name : Dr. Abdul Aziz Bin Mohd Azoddein

Position : Senior Lecturer

Date : 7 June 2018



### **STUDENT'S DECLARATION**

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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(Student's Signature)

Full Name : Imla Syafiqah Binti Mohd Salleh

ID Number : MKC16019

Date : 7 June 2018

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Thesis submitted in fulfillment of the requirements  
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## LIST OF SYMBOLS

$C_o$	Initial concentration
$C_e$	Residual concentration
$K_C$	Equilibrium constant
$K_F$	Indication of the adsorption
$K_L$	Langmuir constant
$k_1$	Equilibrium rate constant of pseudo first-order adsorption
$k_2$	Rate constant of second-order adsorption
$m$	Mass
$1/n$	Adsorption intensity
$q_e$	Adsorption capacity at the time of equilibrium
$q_t$	Adsorption capacity at time
$q_m$	Maximum adsorption capacity
$R^2$	Correlation coefficient
$R_L$	Value of separation factor
$R$	Percent removal
$t$	Time
$T$	Temperature
$V$	Volume
$\Delta G^\circ$	Gibbs free energy
$\Delta H^\circ$	Enthalpy
$\Delta S^\circ$	Entropy

## **LIST OF ABBREVIATIONS**

ANOVA	Analysis of variance
AMD	Acid mine drainage
BET	Brunauer-Emmett-Teller
BOD	Biochemical oxygen demand
CCD	Central composite design
COD	Chemical oxygen demand
DOE	Design of experiment
EFB	Empty fruit bunches
FFD	Fractional factorial design
FTIR	Fourier transform infrared spectrometry
G-POFA	Ground-Palm oil fuel ash
OFAT	One factor at a time
OPC	Ordinary Portland cement
POS	Palm oil shell
POFA	Palm oil fuel ash
RSM	Response surface methodology
SEM	Scanning electron microscopy
TGA	Thermal gravimetric analysis
TSS	Total suspended solid
WHO	World Health Organization

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## ABSTRAK

Abu bahan api kelapa sawit (POFA) yang diubah suai telah digunakan sebagai penjerap untuk menyingkirkan merkuri (II) ion dari air sisa industri. Keputusan awal eksperimen menunjukkan bahawa POFA mempunyai keupayaan penjerapan yang baik untuk ion merkuri (II). Proses penjerapan ion merkuri (II) menggunakan POFA bukan sahaja menyumbang kepada penjerapan merkuri dari air sisa industri tetapi juga dapat mengurangkan pencemaran alam sekitar disebabkan oleh pengumpulan dan pertambahan sisa buangan tersebut. Oleh itu, POFA telah diperkenalkan sebagai bahan mentah dalam kajian ini untuk memaksimumkan penggunaan sisa buangan dari kelapa sawit. POFA telah diubahsuai secara pengaktifan kimia menggunakan asid sulfurik. Manakala, pengimbas mikroskop elektron (SEM), transformasi empatier spektrometri inframerah (FTIR), analisis gravimetrik haba (TGA), dan analisis kawasan permukaan Brunauer-Emmett-Teller (BET) telah digunakan untuk melihat struktur POFA sebelum dan selepas pengubahsuaian berlaku. Kajian saringan dianalisis menggunakan reka bentuk faktorial fraksional (FFD). Dalam kajian analisis faktorial, keadaan terbaik penyingiran ion merkuri (II) ialah pada  $98.03 \pm 0.06\%$  apabila dilakukan pada kelajuan 100 rpm, pH 2, 5 mg/L kepekatan awal merkuri (II) ion, 0.25 g dos penjerap dan masa selama 4 jam. Dari kajian skrining ini, dua faktor iaitu masa kenalan dan kelajuan agitasi dipilih untuk analisis lanjut dalam bahagian pengoptimuman. Pengoptimuman kecekapan penyingiran ion merkuri (II) telah dilakukan dengan menggunakan reka bentuk komposit pusat (CCD) dalam kaedah permukaan respon (RSM). Dalam reka bentuk komposit pusat (CCD), keadaan optimum bagi penyingiran ion merkuri (II) telah diperolehi pada 150 rpm selama 5 jam ialah  $98.93\% \pm 0.02\%$  ion merkuri (II). Data keseimbangan pada pelbagai kepekatan dianalisis dengan model isoterma Langmuir dan Freundlich. Dari kajian ini, nilai pekali korelasi ( $R^2$ ) diperolehi daripada isoterma Freundlich ialah 0.9899. Kajian kinetik telah dijalankan dengan perintah pseudo pertama dan pseudo kedua persamaan tindak balas. Hasil mendapati bahawa proses pengambilan ion merkuri (II) mengikuti pseudo kedua persamaan tindak balas. Parameter termodynamik iaitu tenaga bebas Gibbs ( $\Delta G^\circ$ ), entalpi ( $\Delta H^\circ$ ) dan entropi ( $\Delta S^\circ$ ) juga telah ditentukan. Hasil keputusan menunjukkan perubahan negatif pada tenaga bebas Gibbs (-764.32 kJ / mol) dan perubahan positif bagi entalpi (75531.86 kJ/mol). Ini menunjukkan bahawa penjerapan adalah proses spontan dan bersifat endotermik. POFA yang diubahsuai adalah penyerap yang baik untuk penghapusan ion merkuri (II) dengan penyingiran merkuri 91.18 % daripada air sisa industri. Keputusan ini adalah setanding dengan penyelidik lain dalam lingkungan 90 hingga 95% penyingiran ion merkuri (II). Secara keseluruhan, POFA mempunyai potensi untuk digunakan sebagai penjerap bagi menyingkirkan ion merkuri (II) dari air sisa industri disebabkan oleh prestasi yang tinggi dan ketersediaannya pada kos yang rendah.

## ABSTRACT

Activated palm oil fuel ashes (POFA) were used as the adsorbents for the removal of mercury (II) ion from industrial wastewater. From the preliminary experimental results, it shows that the POFA had good adsorption capability for mercury (II) ion. The adsorption process of mercury (II) ion using POFA not only contributes to the mercury adsorption from industrial wastewater but also removes the environmental pollution caused by accumulation and abundance of waste in nature. Therefore, POFA was introduced as a raw material in this study to maximize the utilization of oil palm waste. POFA was activated using chemical activation and sulphuric acid used as a solvent before proceeding with mercury (II) ion adsorption process. Scanning electron microscopy (SEM), fourier transform infrared spectrometry (FTIR), thermal gravimetric analysis (TGA), and Brunauer-Emmett-Teller (BET) surface area analysis had been applied to observe the effect of the POFA structure before and after activation. The screening study was analyzed using fractional factorial design (FFD). In factorial analysis study, the best mercury (II) ion removal condition removed  $98.03 \pm 0.06\%$  when performed at 100 rpm of agitation speed, pH 2, 5 mg/L of initial mercury (II) ion concentration, 0.25 g of adsorbent dosages and 4 h of contact time. From this screening study, two factors which are contact time and agitation speed were selected for further analysis in optimization part. The optimization of mercury (II) ion removal efficiency was done by using central composite design (CCD) in response surface methodology (RSM). In central composite design (CCD), the optimum condition for mercury (II) ion removal was obtained at 150 rpm for 5 h which removed  $98.93 \pm 0.02\%$  of mercury (II) ion. The equilibrium data at various concentrations were analysed by Langmuir and Freundlich isotherms models. From this present study, the values of correlation coefficient ( $R^2$ ) obtained from the Freundlich isotherm was 0.9899. A kinetic study was carried out with pseudo first order and pseudo second order reaction equations. It was found that the mercury (II) ion uptake process followed the pseudo second order rate expression. Thermodynamic parameters of the Gibbs free energy ( $\Delta G^\circ$ ), enthalpy ( $\Delta H^\circ$ ), and entropy ( $\Delta S^\circ$ ) were also determined. The negative Gibbs free energy change (-764.32 kJ/mol) and the positive enthalpy change (75531.86 kJ/mol) indicated that adsorption was spontaneous process and endothermic nature. It was found that activated POFA was a good adsorbent for mercury (II) ion removal with 91.18 % mercury removal from industrial wastewater. This result was comparable to other researcher in the range from 90 to 95 % mercury (II) ion removal. Overall, POFA has the potential to be used as an adsorbent for the removal of mercury (II) ion from industrial wastewater due to its high performance and availability at low cost.

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