

CATALYTIC OXIDATIVE HYDROTHERMAL  
TREATMENT OF PALM OIL MILL EFFLUENT  
OVER  $\text{CuO}/\text{Al}_2\text{O}_3$  and  $\text{Ce-CuO}/\text{Al}_2\text{O}_3$   
CATALYSTS

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I hereby declare that I 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 Doctor of Philosophy.

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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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## ABSTRAK

Tesis ini mengkaji kualiti rawatan kumbahan kilang minyak sawit (POME) dengan menggunakan kaedah hidro-haba beroksigen (OHT) dan tanpa oksigen (NOHT). Penyelidikan ini turut melibatkan penggunaan pemangkin dalam proses hidro-haba beroksigen (COHT). Proses hidro-haba sering digunakan dalam rawatan sisa biomas basah, tetapi ia tidak pernah digunakan dalam rawatan POME sebelum kajian ini dijalankan. Objektif kajian ini terdiri daripada kajian proses hidro-haba pada tahap suhu dan masa eksperimen yang berlainan, fungsi pemangkin dalam proses dan cadangan mekanisme proses serta kajian kinetik. 500 mL POME telah digunakan pada tahap suhu 493-533 K dan tempoh eksperimen 2-8 jam. Dalam eksperimen COHT pula, dua jenis pemangkin, iaitu  $\text{CuO}/\text{Al}_2\text{O}_3$  and  $\text{Ce-CuO}/\text{Al}_2\text{O}_3$  telah dikaji pada kapasiti muatan 0.2 mg/ml, 1.0 mg/ml dan 2.0 mg/ml pada tahap suhu 533 K selama 8 jam. Semakin tinggi suhu proses, semakin berkurang sebatian utama dalam POME mentah, iaitu asid heksadekanoik yang ditemui dalam produk cecair NOHT and OHT. Dalam proses NOHT, peningkatan tahap suhu eksperimen menyebabkan sebatian karboksil semakin berkurang manakala sebatian fenolik semakin bertambah. Produk gas yang dihasilkan daripada NOHT mengandungi  $\text{CO}_2$ ,  $\text{CO}$ ,  $\text{H}_2$ , and hidrokarbon  $\text{C}_3\text{-C}_6$ . Hanya sedikit gas  $\text{CH}_4$  yang ditemui dalam produk gas NOHT pada tahap suhu 533 K yang disebabkan proses gasifikasi hidro-haba pada suhu yang rendah. Pada suhu eksperimen 533 K selama 8 jam, penyahwarnaan POME yang dicapai melalui NOHT dan OHT adalah sebanyak 7.02% dan 54.10% masing-masing. Fenomena ini menandakan bahawa keefisienan penyahwarnaan OHT adalah lebih tinggi. Pada tahap suhu dan tempoh eksperimen yang sama, tahap pengurangan permintaan oksigen kimia (COD) dan permintaan oksigen biokimia lima hari ( $\text{BOD}_5$ ) yang dicapai melalui NOHT adalah setinggi 61.4% dan 68.0% masing-masing. Walau bagaimanapun, keefisienan OHT dalam mengurangkan COD and  $\text{BOD}_5$  adalah lebih tinggi, iaitu 81% dan 87% masing-masing, dengan peningkatan nilai pH produk cecair dari 3.5 ke 6.5. Kualiti rawatan POME melalui COHT adalah lebih tinggi berbanding dengan proses tanpa pemangkin. Dalam COHT, pemangkin  $\text{Ce-CuO}/\text{Al}_2\text{O}_3$  dengan kapasiti muatan sebanyak 2.0 mg/ml telah mengurangkan 86.1% COD dan 94.7%  $\text{BOD}_5$  pada suhu 533 K selama 8 jam. Kualiti rawatannya lebih baik daripada  $\text{CuO}/\text{Al}_2\text{O}_3$  yang mencapai pengurangan COD dan  $\text{BOD}_5$  sebanyak 79.4% dan 93.8% masing-masing. Sumbangan pemangkin dalam menurunkan COD dan  $\text{BOD}_5$  POME tidak lagi ketara dengan kapasiti muatan pemangkin melebihi 1.0 mg/ml, Jadi 1.0 mg/ml menjadi kapasiti muatan optimum dari segi teknikal dan ekonomi. Kajian kebolegunaan semula pemangkin  $\text{Ce-CuO}/\text{Al}_2\text{O}_3$  menunjukkan bahawa perlakuan larut lesap elemen Cu dan Al adalah tidak ketara. Tambahan pula, pengurangan COD (75.4%) dan  $\text{BOD}_5$  (85.2%) selepas tiga kali penggunaan semula menandakan tiada penyahaktifan pemangkin yang ketara. Tindak balas tertib kedua ialah hukum kadar kinetik COHT yang sesuai kerana pekali korelasi model ini dengan data eksperimen adalah melebihi 0.90 serta sisihan piawai yang kurang daripada 0.1%. Dalam aspek mekanisme kimia NOHT, POME mereput menjadi sebatian larut air lalu mengalami proses penyahoksigenan (dehidrasi dan dekarboksilasi) untuk menghasilkan hidrochar yang mengandungi oksigen yang rendah serta produk cecair yang mengandungi sebatian aromatik yang tinggi. Mekanisme kimia bagi COHT pula ialah proses radikal bebas. Hasil kajian ini membuktikan bahawa rawatan POME berpotensi menggantikan proses rawatan kumbahan yang sedia ada. Hasil kajian ini boleh dijadikan sebagai bahan asas bagi mengkomersialkan dan menaiktarafkan proses hidro-haba dalam rawatan POME pada masa akan datang.

## ABSTRACT

This study aspires to investigate the performance of novel non-oxidative and oxidative subcritical hydrothermal treatments (NOHT and OHT, respectively), followed by catalytic oxidative hydrothermal treatment (COHT) of palm oil mill effluent (POME). Hydrothermal reaction is commonly used in wet biomass treatment, but it had never been used in POME treatment before this research was conducted. The high reaction temperature and time of hydrothermal process and catalyst activity in an aqueous environment are problems to be addressed. Hence, the objectives of this research are to investigate the performance of hydrothermal reactions and catalytic reactions as well as to propose the reaction mechanisms. The experiments of OHT and NOHT were conducted using 500 mL POME of initial COD and BOD<sub>5</sub> at 52200 mg/L and 18020 mg/L, respectively, at different temperatures (493-533 K) and reaction times (2-8 h). Meanwhile, the experiments of COHT were performed using a similar working volume of POME over two types of catalysts, i.e., CuO/Al<sub>2</sub>O<sub>3</sub> and Ce-CuO/Al<sub>2</sub>O<sub>3</sub>, at loadings of 0.2, 1.0, and 2.0 mg/ml at 533 K for 8 h. The dominant component of fresh POME, i.e., n-hexadecanoic acid, gradually reduced in the liquid products of NOHT and OHT in reactions with elevated temperatures. Carboxyl compounds reduced while phenolic components increased as reaction temperature increased over NOHT. The gaseous products of NOHT contained carbon dioxide (CO<sub>2</sub>), carbon monoxide (CO), hydrogen gas (H<sub>2</sub>), and C<sub>3</sub>-C<sub>6</sub> hydrocarbons. Traces of methane gas (CH<sub>4</sub>) were only found at 533 K due to subcritical hydrothermal gasification at low temperatures. The decolourisations of POME in NOHT and OHT were 7.02% and 54.10% respectively at 533 K and 8 h, indicating the notable performance of OHT in decolourising POME. At 533 K and 8 h, NOHT achieved the highest reductions of chemical oxygen demand (COD) and five-day biochemical oxygen demand (BOD<sub>5</sub>) at 61.4% and 68.0%. However, the OHT reaction resulted in better removals of COD and BOD<sub>5</sub>, recording 81% and 87% respectively, with the pH of the liquid product approaching 6.5 from the initial value of 3.5 at the same reaction temperature and time. Notwithstanding, COHT of POME achieved more remarkable performance than the non-catalytic reactions. At a loading of 2.0 mg/ml catalyst in COHT, Ce-CuO/Al<sub>2</sub>O<sub>3</sub> recorded 86.1% COD and 94.7% BOD<sub>5</sub> removals at 533 K and 8 h, where its performance was slightly better than CuO/Al<sub>2</sub>O<sub>3</sub> (79.4% COD and 93.8% BOD<sub>5</sub> removals). The increase of catalysts loading after 1.0 mg/ml did not contribute any significant difference in degrading the pollutants of POME, suggesting the adequacy of using 1.0 mg/ml catalysts viewing from the technical and economic aspects. The reusability test of spent Ce-CuO/Al<sub>2</sub>O<sub>3</sub> catalysts showed insignificant leaching of Cu and Al elements, and the reductions of COD and BOD<sub>5</sub> were as high as 75.4% and 85.2% respectively, indicating no significant deactivation of catalysts up to three reaction cycles. In the derivation of kinetic rate law, a second-order kinetic conformed well to experimental data of COHT with correlation coefficients greater than 0.90 and an error of less than 5% between the estimated and experimental findings. For the mechanism studies, POME in NOHT decomposed into water-soluble compounds, followed by deoxygenation (dehydration and decarboxylation) in producing hydrochar with lower oxygen content and higher aromatic compounds in the liquid product. On the other hand, the reaction mechanism was dominated by the free radicals' reaction in COHT. The experimental findings have successfully revealed the potential of this novel POME treatment method in substituting the conventional treatment process. This novel study could act as a foundation for the scalability and commercialisation of hydrothermal processes in POME treatment in the future.

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