HYDROGEN-RICH SYNGAS PRODUCTION FROM STEAM REFORMING OF PALM OIL MILL EFFLUENT (POME) OVER $LaNiO_3$ & $LaCoO_3$ CATALYSTS

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SUPERVISOR’S DECLARATION

I hereby declare that I have checked this thesis and in my 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 citation 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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Thesis submitted in fulfilment of the requirements
for the award of the degree of
Doctor of Philosophy

Faculty of Chemical and Natural Resources Engineering
UNIVERSITI MALAYSIA PAHANG

JULY 2019

I want to dedicate my thesis to my beloved supervisor, friends, and family who supported me each step of the way.

ACKNOWLEDGEMENTS

First and foremost, I would like to express my most profound appreciation to my dear supervisor, Associate Professor Dr Cheng Chin Kui for his excellent guidance, valuable suggestions, and expert consultation. He had provided me with a comfortable yet flexible atmosphere throughout this research work. Special gratitude again goes to his guidance in all the time of research and writing of this thesis.

Million thanks to the postdoctoral researcher Dr Ng Kim Hoong for his pioneer guidance and insightful comments on laboratory skills. I would also like to thank all technical staffs of the Faculty of Chemical and Natural Resources Engineering laboratory for their assistance while completing the research.

Finally, I would like to express my heartiest appreciation to my beloved family for their boundless encouragement. My family’s love and support were crucial for me to overcome all the obstacles faced in the completion of this research. Thanks again to all who helped me.

ABSTRAK

Pembangunan pesat industri kelapa sawit tempatan membentuk sisa eluen kilang minyak sawit (POME) yang sangat tercemar dalam kuantiti yang banyak. Rawatan kolam terbuka yang kerap dipakai mempunyai beberapa masalah seperti keperluan tanah yang besar, perlahan, tidak cekap untuk menepati ambang pelepasan, dan pelepasan gas rumah hijau (CO_2 and CH_4). Kajian ini menyiasat kemungkinan penukaran bahan pencemar dalam POME ke gas sintesis yang kaya dengan hidrogen melalui reformasi wap dengan pemangkin ($LaNiO_3$ dan $LaCoO_3$). Bahan mentah POME ialah air kumbahan yang berkecoklatan (A = ~1.93), berasid (pH 5), dan sangat tercemar (COD = ~70000 mg/L, BOD_5 = ~11000 mg/L, dan TSS = ~7700 mg/L). POME terdiri daripada 99.73 mol% air dan 0.27 mol% organik (kebanyakkan asid karboksilat, fenol, dan alcohol). Dengan kaedah pengurangan tenaga bebas Gibbs, simulasi termodinamik dari 573 – 1173 K mengesahkan pengeluaran gas sintesis melalui reformasi wap POME dan meramal kemungkinan tindak balas sampingan. Selepas itu, $LaNiO_3$ dan $LaCoO_3$ dihasilkan melalui kaedah sol-gel terubah suai yang memakai asid sitrik. CO_2-TPD dan NH_3-TPD membuktikan keasidan bersih $LaNiO_3$ dan kealkalian bersih $LaCoO_3$. Sebelum reformasi wap POME, pemangkin dikurang oleh hidrogen untuk membentuk logam aktif (Ni atau Co) yang tersebar atas sokongan La_2O_3. Khususnya, logam aktif memangkin tindak balas sementara sokongan La_2O_3 mengurang pemendapan karbon. Bagi kedua-dua reformasi wap POME dengan pemangkin, hasil gas sintesis dan kecekapan degradasi yang optimum ditentukan dengan menala suhu (T), kadar aliran POME ($\dot{V}_{POME}$), kuantiti pemangkin (W_{cat}), dan saiz zarah (d_{cat}). Hasil gas sintesis dan kecekapan degradasi bertambah dengan T yang lebih tinggi sehingga 873 K, $\dot{V}_{POME}$ yang lebih cepat sehingga 0.09 mL/min, W_{cat} yang lebih banyak sehingga 0.3 g, dan d_{cat} yang lebih kecil sehingga 74 µm. Apabila $T \geq 973$ K, pemangkin mengalami pemendapan karbon dan sintering yang ketara. Jika $\dot{V}_{POME} > 0.09$ mL/min, pemendapan karbon pada pemangkin adalah ketara. Kalau $W_{cat} > 0.3$ g, permukaan pemangkin berkurang akibat pengumpulan pemangkin ke struktur plat. Ketika $d_{cat} < 74$ µm, lubang pemangkin yang tersumbat menyebabkan aktiviti pemangkin yang rendah. Justeru, keadaan optimum bagi kedua-dua reformasi wap POME dengan pemangkin adalah T = 873 K, $\dot{V}_{POME}$ = 0.09 mL/min, W_{cat} = 0.3 g, dan d_{cat} = 74 – 105 µm. Walau bagaimanapun, $LaNiO_3$ yang berasid bersih menghasilkan lebih banyak gas sintesis (F_{Syngas} = 132.47 µmol/min, , y_{Syngas} = 72.60%, dan HHV = 220.31 kJ/mol) yang kaya dengan hidrogen berbanding $LaCoO_3$ yang beralkali bersih (F_{Syngas} = 86.60 µmol/min, y_{Syngas} = 70.71%, dan HHV = 231.14 kJ/mol). Selain itu, rawatan optimum dengan pemangkin $LaNiO_3$ membentuk cecair kondensat yang kurang tercemar (COD = 326 mg/L dan BOD_5 = 27 mg/L) daripada $LaCoO_3$ (COD = 435 mg/L dan BOD_5 = 62 mg/L). Keasidan bersih menggalak perengkahan organik POME sebelum reformasi wap manakala kealkalian bersih mempromosi tindak balas "reverse Boudouard" yang menggunakan karbon dengan menggalak penjerapan CO_2. Kesimpulannya, reformsi wap POME melalui $LaNiO_3$ atau $LaCoO_3$ adalah menarik kerana ia memanfaatkan gas sintesis ketika mendegradasi POME.

ABSTRACT

The flourishing development of local oil palm industry inflicts concomitant generation of enormous, highly polluted palm oil mill effluent (POME). The prevalent open ponding treatment was land-intensive, sluggish, and incompetent to degrade POME to below discharge threshold yet being accused for greenhouse gases (CO_2 and CH_4) emission. This study investigated the potentiality of novel catalytic POME steam reforming over $LaNiO_3$ and $LaCoO_3$ to valorise pollutant-laden POME into valuable H_2-rich syngas. The POME feedstock was a brownish (A = ~1.93), acidic (pH of 5), and highly polluted (COD = ~70000 mg/L, BOD_5 = ~11000 mg/L, and TSS = ~7700 mg/L) wastewater. POME was composed of 99.73 mol% water and 0.27 mol% organics (mainly carboxylic acids, phenol, and alcohols). Through minimisation of total Gibbs free energy, thermodynamic simulation from 573 – 1173 K confirmed syngas production from POME steam reforming and predicted the likelihood of side reactions. Subsequently, $LaNiO_3$ and $LaCoO_3$ were synthesised using modified citrate sol-gel route. Combination of CO_2-TPD and NH_3-TPD asserted the net-acidity of $LaNiO_3$ and the net-basicity of $LaCoO_3$. Before POME steam reforming, the catalysts were reduced by H_2 to form well dispersed active metal (Ni or Co) on La_2O_3 support. Specifically, the active metal catalysed the reaction while the La_2O_3 support suppressed the coking deactivation. For both catalytic POME steam reforming, the optimum syngas yield and degradation efficiencies were determined by tuning temperature (T), POME flow rate ($\dot{V}_{POME}$), catalyst weight (W_{cat}), and particle size (d_{cat}). The syngas yield and degradation efficiencies increased with greater T up to 873 K, higher $\dot{V}_{POME}$ up to 0.09 mL/min, greater W_{cat} up to 0.3 g, and smaller d_{cat} down to 74 µm. When $T \geq 973$ K, the catalysts experienced significant coking and sintering deactivation. If $\dot{V}_{POME} > 0.09$ mL/min, coking deactivation of catalysts was conspicuous. For $W_{cat} > 0.3$ g, the catalysts certainly agglomerated into a plate-like structure with reduced catalytic surface. When $d_{cat} < 74$ µm, pore occlusion of catalysts responsible for appreciably declined catalytic activity. Thus, the optimum conditions of both catalytic POME steam reforming were T = 873 K, $\dot{V}_{POME}$ = 0.09 mL/min, W_{cat} = 0.3 g, and d_{cat} = 74 – 105 µm. However, the net-acidic $LaNiO_3$ granted higher amount of H_2-rich syngas (F_{Syngas} = 132.47 µmol/min, y_{Syngas} = 72.60%, and HHV = 220.31 kJ/mol) than the net-basic $LaCoO_3$ (F_{Syngas} = 86.60 µmol/min, y_{Syngas} = 70.71%, and HHV = 231.14 kJ/mol). In addition, the optimal catalytic treatment over $LaNiO_3$ generated a less polluted liquid condensate (COD = 326 mg/L and BOD_5 = 27 mg/L) than $LaCoO_3$ (COD = 435 mg/L and BOD_5 = 62 mg/L). The net-acidity favoured the cracking of POME's organics before steam reforming while net-basicity promoted the carbon-consuming reverse Boudouard reaction by facilitating CO_2 adsorption. Conclusively, the novel catalytic POME steam reforming over $LaNiO_3$ or $LaCoO_3$ is alluring as it harnesses syngas while degrading the POME wastewater.

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$\dot{V}_{POME}$	POME flow rate
F_i	Molar flow rate of gas species i
K_{eq}	Equilibrium constant
P_{POME}	Partial pressure of POME vapour
W_{cat}	Catalyst weight
X_P	Degradation efficiencies of wastewater parameters P
X_i	Concentration of gas species i
d_{cat}	Particle size
y_i	Composition of gas species i
A	Colour intensity
T	Temperature

LIST OF ABBREVIATIONS

BOD	Biochemical oxygen demand
CB	Conduction band
COD	Chemical oxygen demand
DOE	Department of Environment
EDX	Energy dispersive X-ray analysis
EFB	Empty fruit bunch
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transform infrared spectroscopy
GC-MS	Gas chromatography-mass spectroscopy
HHV	Higher heating value
OEA	Organic elemental analysis
OFAT	One-factor-at-a-time approach
OPF	Oil palm frond
PKS	Palm kernel shell
POFA	Palm oil fuel ash
POME	Palm oil mill effluent
SEM	Scanning electron microscopy
SR	Steam reforming
TEM	Transmission electron microscopy
TGA	Thermogravimetric analysis
TPD	Temperature programmed desorption
TPR	Temperature programmed reduction
TSS	Total suspended solids
UV	Ultraviolet
VB	Valence band
WGS	Water gas shift
WHSV	Weight hourly space velocity
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction spectroscopy

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